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Sustainability of Well Wall Concrete Exposed to Salts Using Varying Proportions of Metakaolin in Cement Mortar

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ABSTRACT

This research investigated the effect of replacing salt-resistant cement in oil well casings with varying proportions of Metakaolin, focusing on compressive strength, flexural strength, and sample density. Ultrasonic testing was also performed on all samples. Various proportions of cement, sand, and Metakaolin were prepared, and molds were cast for testing. Five mixtures were used: the first mixture consisted of salt-resistant cement with sand only; the second: cement with 5% Metakaolin; the third: cement with 10% Metakaolin; the fourth: cement with 15% Metakaolin; and the final mixture of cement with 20% Metakaolin. The samples were incubated for 7days, 28days, and 150 days in water only, and a further 150-day period (the first 28days in water followed by 120days in sulfate chloride solution). The results showed changes in compressive strength, bulk density, UPV, and flexural stress. It can be concluded that the optimal Metakaolin content (10–15%) achieves the best flexural strength performance under all processing conditions, without any adverse effects from salt exposure. The study's findings demonstrate the feasibility of using metakaolin in oil well walls at approximately 10–15% as a partial replacement for cement, provided that a highly efficient kaolin burning process is employed to prevent premature loss of concrete strength. Adequate water curing is essential before the concrete is exposed to saline environments. This partial replacement helps reduce the environmental impact of the cement used, while maintaining mechanical properties and durability, thus achieving a suitable balance between structural performance and sustainability.

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استدامة خرسانة جدران الابار المعرضة للأملاح عن طريق إضافة نسب متفاوتة من الميتاكاؤولين إلى مونة الإسمنت

سرى محمد علي

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الخلاصة

بحثت هذه الدراسة تأثير الاستبدال الجزئي للإسمنت المقاوم للأملاح المستخدم في جدران الآبار النفطية بنِسَب متفاوتة من الميتاكاولين، مع التركيز على فحص مقاومة الانضغاط، ومقاومة الانحناء، والكثافة الظاهرية، إضافةً إلى فحص جميع العينات باستخدام الموجات فوق الصوتية للخلطات المختلفة. حيث تم تحضير خلطات مختلفة من الإسمنت والميتاكاولين وإضافتها إلى الرمل والماء بنِسَب محددة، ثم صُبّت النماذج في القوالب القياسية المخصصة لإجراء كل فحص من الفحصوصات المختبرية. تكوّنت الخلطة الأولى من أسمنت مقاوم للأملاح مع الرمل فقط، في حين احتوت الخلطة الثانية: أسمنت مع 5% ميتاكاولين؛ والثالثة: أسمنت مع 10% ميتاكاولين؛ والرابعة: أسمنت مع 15% ميتاكاولين؛ والخامسة: أسمنت مع 20% ميتاكاولين. وتم حفظ النماذج لمدة 7 أيام، و28 يومًا، و150 يومًا في الماء فقط، وتم حفظ نماذج اخرى لمدة 150 يومًا (28 يومًا في الماء تليها 120 يومًا في محلول كبريتات الكلوريد). حُفظت نماذج لمدة 7 أيام و28 يومًا و150 يومًا في الماء، كما حُفظت مجموعة أخرى من النماذج لمدة 150 يومًا، شملت 28 يومًا من المعالجة بالماءثم تحويلها لمحلول كبريتات الكلوريد مدة 120 يومًا. حيث أظهرت النتائج وجود تحسينات ملحوظة في مقاومة الانضغاط، والكثافة الظاهرية، وسرعة الموجات فوق الصوتية، ومقاومة الانحناء وذلك تبعًا لنِسَب الاستبدال وظروف المعالجة. ويمكن استنتاج أن المحتوى الأمثل من الميتاكاولين، 10–15%، حقق أفضل أداء لمقاومة الانحناء في جميع ظروف المعالجة، دون ملاحظة اي تأثيرات سلبية ناتجة عن تعرض النماذج للأملاح. ومن نتائج الدراسة تثبت إمكانية استخدام الميتاكاولين في جدران الآبار النفطية بنِسَب تتراوح تقريبًا بين 10–15% كبديل جزئي للإسمنت، بشرط اعتماد عملية حرق عالية الكفاءة للكاؤولين لتجنب انخفاض المقاومة المبكرة للخرسانة، مع ضرورة استمرار المعالجة الكافية بالماء قبل تعرض الخرسانة للبيئات المالحة. ويُسهم هذا الاستبدال الجزئي في تقليل الأثر البيئي للإسمنت المستخدم، مع التركيز على الحفاظ على الخصائص الميكانيكية والمتانة، وذلك لتحقيق توازنًا مناسبًا بين الأداء الإنشائي والاستدامة.

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Introduction

Concrete is a primary and widely used building material in most infrastructure projects worldwide due to its high compressive strength, resistance to various environmental conditions, and cost-effectiveness compared to other materials. However, conventional concrete, containing ordinary Portland cement, faces increasing environmental and technical challenges, most notably the rising carbon dioxide emissions resulting from cement production. Some

studies indicate that producing one ton of cement releases approximately one ton of carbon dioxide into the atmosphere (Al-Hashem et al., 2022).

Contemporary studies indicate that replacing cement with kaolin at a rate of 5% to 15% has led to increased compressive, tensile, and flexural strength, as well as the construction of a less porous concrete structure with improved resistance to chloride ion permeability, resulting in concrete that is more resistant to adverse environmental factors (Shende et al., 2024), (Almattar, et al., 2009), (Poon et al., 2001). Furthermore, microscopic examination of concrete has shown that kaolin produces microscopic reactions and forms hydrated calcium silicate (C-S-H) phases, thereby increasing the long-term durability of the concrete. (Chorzepa et al., 2017).

The direct impact of saline groundwater on well wall concrete, as well as temperature fluctuations and hydraulic pressure from pumping and extraction operations, necessitates that the concrete used for well wall cladding be low-permeability and high-strength to ensure the continued stability and integrity of the well (Fan et al., 2024).

In the field of sustainability, recent research has focused on finding alternative cementation materials that can partially replace cement in specific proportions to reduce emissions and improve the properties of concrete to withstand harsh conditions. These materials include fly ash, blast furnace slag, silica fume, and metakaolin. Most research has shown that adding these materials increases compressive strength, reduces porosity and permeability, which leads to increased density and enhanced concrete durability (N. Shafiq, 2019), (Siddique &Khatib,2009).

A comparison of durability when using cement mortar with metakaolin, conducted by Corard (2003), demonstrates this. He prepared mixtures with varying proportions of metakaolin and then compared the concrete's behavior against sulfate and chloride ion diffusion. The results showed that adding metakaolin to cement reduced porosity, which in turn reduced sodium chloride diffusion in the samples, and increased resistance to shrinkage and swelling caused by sulfates compared to standard mortar. A study by Al-Akhras (2006) demonstrated the effect of replacing 5%, 10%, and 15% of cement in mortar with metakaolin on concrete's sulfate resistance. The results showed that the 10% - 15% yielded the best resistance to sulfate corrosion, while reducing expansion and swelling resistance, which in turn improved the mechanical properties of the concrete in both early and late ages. The published study by Ferreira (2016) examined the chloride permeability properties of concrete mixes containing varying proportions of kaolin mineral using diffusion and natural migration tests. The results indicated that the addition of kaolin mineral, replacing cement, significantly improved the concrete's resistance to chloride ion penetration by reducing large pores and improving their distribution within the matrix, i.e., by restructuring the concrete. (Khater ,2011) He conducted experiments on cement mortar and concrete with high replacement ratios of mineral kaolin, and calculated the resistance of these samples to magnesium and sulfate solutions. The mixtures containing mineral kaolin showed greater resistance to the effects of the magnesium salt solution compared to the control mixture, thanks to the lower permeability content and improved internal structure.

Recently, Yuan (2022) studied the combined effects of carbonates, sulfates, and chlorides on the performance of cement mortar cured with metakaolin. The results showed that the addition of metakaolin reduced water absorption and ion translocation in the concrete. Comprehensive review by Homayoonmehr (2022) investigated the mechanisms by which metakaolin binds to chloride ions and its impact on chloride binding within the concrete structure. (Cheng et al.,2024) conducted a recent study on Metakaolin-based geopolymer mortar and tested the resistance of the MK-based geopolymer mixture to chloride-sulfate mixed

environments (completely/semi-immersion). The results show that appropriate additions of GBFS with Metakaolin improved abrasion resistance under partial/full immersion conditions, with a clear improvement in mechanical properties and mass retention, supported by XRD and SEM analysis.

A local study (Shihab, 2016) on fiber-reinforced concrete showed that the introduction of highly reactive Metakaolin reduced chloride ion penetration and increased resistance over both short and long time periods. This study is useful as a practical model for local applications, as it tested readily available Metakaolin types and actual aqueous environments. (Zhong et al. ,2024) conducted a recent study involving recycled concretes with added MK. They analyzed the resistance of the compounds to sulfate attack and the effect of MK in improving the performance of recycled mixes. They found that MK contributes to enhanced durability and reduced structural failure under sulfate conditions.

Long-term studies on HRM (High-Reactivity Metakaolin) and a series of long-term experiments have shown that concretes containing high-reactivity MK exhibit prolonged resistance to chloride penetration over extended periods. However, the time-based improvements depend on the cement replacement ratio and the curing method. The long-term papers available on ResearchGate show follow-up results even years later. (S. Mousavinezhad ,2024) presented a practical approach to evaluating sulfate resistance in mortar and concrete, reviewing various testing methods and discussing the effect of additives such as Metakaolin on improving sulfate resistance results, while also providing recommendations for designing rapid and reliable tests.

Metakaolin

Metakaolin is a pozzolanic material produced by the thermal treatment of kaolin mineral, which is found in the western desert region of Iraq, at temperatures ranging between 600 and 800 degrees Celsius. When cement is mixed with metakaolin, the mixture transforms into an amorphous phase with a chemically active reaction, capable of reacting with calcium hydroxide produced during the cement hydration process, to form additional compounds of calcium silicates and aluminates (Rashad, 2013).

Casing

Well casing and cementing are essential elements of well construction. They are typically used to stabilize the wellbore, prevent contamination between formations, and protect the well from external pressures. This process involves installing steel pipes in the drilled well using cement to form a strong and resistant wall, ensuring the well's safety from collapse and enabling the extraction of resources with the highest efficiency (Rabia, H., 2001).

Cementing

Well wall stabilization is the process of injecting cement slurry into the annular space between the well wall and the casing after the casing has been driven to the required depth. This process aims to stabilize the casing and prevent fluid leakage from the well into the surrounding soil layers. The goal is to secure the casing in the well and prevent its movement or collapse. Additionally, it isolates and protects groundwater layers from contamination by fluid or gas leaks and safeguards the casing from corrosion and chemical degradation. For this reason, the cement mix must be designed with high precision and careful consideration to obtain concrete that can withstand high pressures and resist the effects of high temperatures in deep wells. In addition, appropriate casting techniques must be used for each well to eliminate several drawbacks, such as fluid leakage, well collapse, and contamination of nearby freshwater sources. (Halliburton, 2010).

Sulfate-Resistant Cement

Sulfate-resistant cement is essentially ordinary Portland cement, but it contains less than 5% tricalcium aluminate (C3A) and less than 25% (2C3A + C4AF), which leads to resistance to sulfate formation and thus reduces the percentage of sulfates that attack the concrete and reduce its resistance to harsh conditions (H. A. Abdel-Gawad et al., 2017).

The aim of this study was:

The study aimed to replace salt-resistant cement with four different ratios of metakaolin to form cement mortar cast in molds designated for each test, and then preserve the samples in water and others in a sulfate chloride solution. Then the preserved samples were examined in the short term and the long term, and the effect on compressive strength, density, flexural strength, and ultrasonic properties was observed. The results of this study were then used to design optimal mixes for repair mortars used in preparing the composite concrete component for oil well casing.

Material Used and Experimental Procedures

Overview of the comprehensive materials used in this study, the mix design (casting) procedures, and the experimental tests conducted to evaluate the physical and mechanical properties of the mortar. The study focuses on key properties such as water absorption, compressive strength, and direct tensile strength, following ASTM standards. In addition, the mortar is cast with different mix proportions, both a control mix (cement and sand only) and various Metakaolin-modified mixes, to assess their performance under normal and sulfate-rich curing conditions.

Materials Used

The materials used in this study include**:**

1. Sand

River sand conforming to specifications BS 882 (BS 882, 2002) was used**.**

2. Sulfate-Resistant Cement (SRC)

Sulfate-resistant cement (SRC) used satisfies Iraqi Standard Specification (IQS: 5/1984) in chemical requirements.

3. Metakaolin

Metakaolin is a highly effective pozzolanic material used in concrete. It is produced by heating kaolin mineral to 750°C. The natural kaolin was sourced from Al-Qaim in Iraq's Anbar province and heated using a programmed laboratory kiln during the calcination process

4. Sulfate-Chloride Solution

The solution used consists of sodium chloride and sodium sulfate dissolved in water to study the mechanism of cement material degradation.

Mortar Mix Proportions, Casting, and Curing

1. Mix Proportions

The overall mix design follows a scale of 1:2.25:0.5, where one part represents cement and 2.25 parts represent sand, with water amounting to half the weight of cement. Two categories of mixes were prepared:

- Reference Mix (Cement + Sand Only)

- Metakaolin-Modified Mixes (Cement + Metakaolin + Sand): five different percentages of mix proportions were prepared to study the effect of Metakaolin in the mixes, as shown in the Table1:

Table 1 shows the percentage of cement and metakaolin in the mixes

Mix code	cement%	Metakaolin%
Reference	100%	0%
MK_1	95%	5%
MK_2	90%	10%
MK_3	85%	15%
MK_4	80%	20%

2. Curing Regime

After casting, the specimens were left in the molds for 48 hours. All specimens were initially immersed in tap water. The curing procedure is as follows:

For the control mix and for some Metakaolin-modified specimens, samples remained in tap water for the entire curing period. For a subset of specimens, after 28 days of curing in tap water, they were transferred to a sulfate chloride solution for the remaining 120 days.

3. Experimental Tests

a. Compressive Strength Test

The compressive strength was measured in accordance with ASTM C109 (ASTM C109, 2016) and was tested at 7days, 28 days, and 150 days.

b. Flexural Strength Test

This test is performed on prism specimens (40mm*40mm*160mm) according to ASTM C348 (ASTM C348, 2014).

c. Ultrasonic Pulse Velocity Test

The ASTM C597 (ASTM C597, 2006) was followed in conducting this non-destructive test.

d. Bulk Dry Density Test

In accordance with the ASTM C642 (ASTM C642, 2013).

Results and Discussion

Will discuss the effect of percent NS, SF, the replacement SRC at mortar, and study the Change of Compressive Strength, flexural, UPV tests, and bulk density.

1. Compressive Strength

Fig. 1 shows that the compressive strength at 7 days gradually increases with increasing Metakaolin content, reaching approximately 33.2 MPa at 15% MK compared to 31.25 MPa for the reference mix. However, it decreases with increasing substitution to 20%. This change aligns with Poon et al. (2001) observation that highly reactive Metakaolin accelerates pozzolanic reactions in the early stages due to an increased micro reaction area, except at high percentages, which may reduce the available lime content and delay C–S–H structural development. Al-Akhras (2006) also indicated that the optimal Metakaolin content in most mixes' ranges from 10–15% to achieve the best balance between pozzolanic reaction and cement content.

Meanwhile, the compressive strength at 28 days shows a significant improvement in all mixes containing Metakaolin, reaching a maximum strength of approximately 48.6 MPa at 15% MK compared to 46.6 MPa for the reference sample. This increase is attributed to the continuous reaction of Metakaolin, which reduces porosity and improves the distribution of microspores within the cement paste (Courard, 2003).

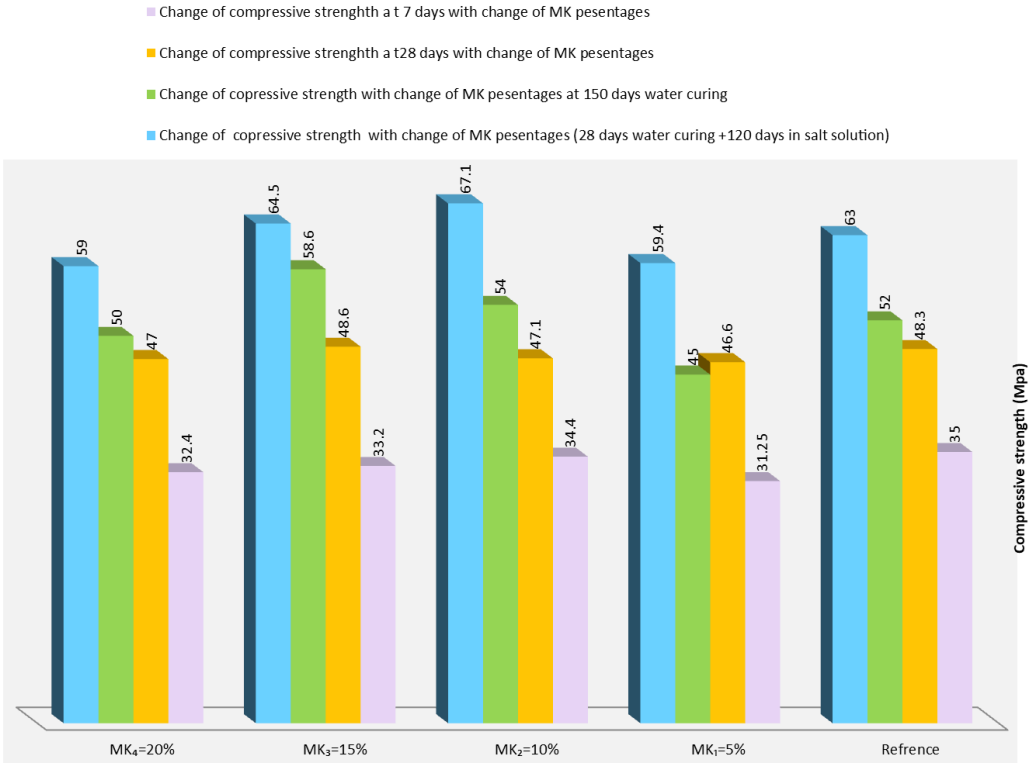


Fig. 1 Change of compressive strength with change of MK parentages and different curing times

The compressive strength after 150 days of aqueous curing reached its highest values at 10–15% Metakaolin (58.6–54 MPa), while it decreased relatively at 20%. This is consistent with the findings of (Khater, 2011) (Yuan et al., 2022), who observed that moderate additions of MK prolong pozzolanic activity and produce denser C–S–H layers that are more resistant to hydrolysis, while excessive additions reduce the free $Ca(OH)_2$ content necessary for the continuation of the reaction. Following further treatment in chloride sulfate solution, a slight decrease in all values was observed, but mixtures containing 10–15% Metakaolin retained the highest resistance (64.5–67.1 MPa) compared to the reference sample (63 MPa). This suggests that Metakaolin improved the cement mortar's resistance to chloride ion penetration and salt-induced hydration.

2. Flexural Strength Test

The Fig 2 shows the relationship between the percentage of Metakaolin (MK) in the concrete mix and its flexural strength at different curing times (-7 days, 28 days, 150 days, and 28 days in water + 120 days in sulfate-chloride solution). At all percentages of added MK, as well as in the reference sample, we observe that flexural strength increases from 7 days to 28 days, and then with longer curing times. This is because the pozzolanic reactions between MK and the cementitious reactants continue over time, promoting gel formation (C-S-H) and resulting in a denser structure with improved tensile and flexural strength.

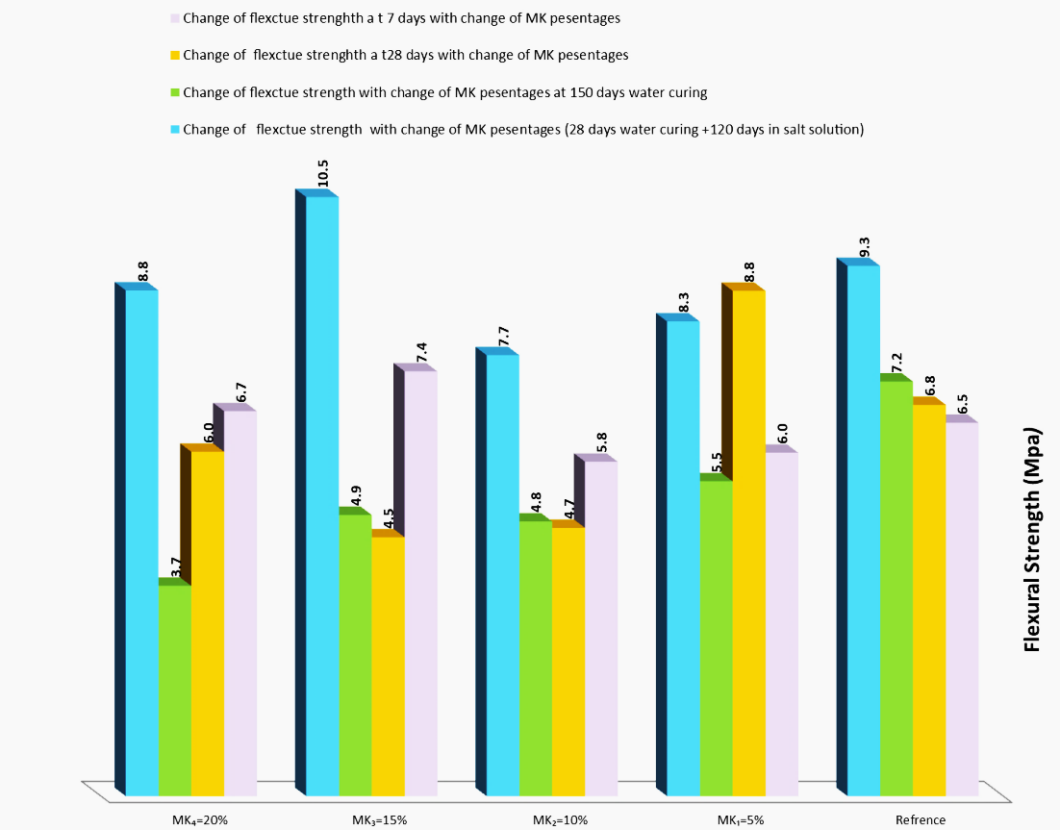


Fig. 2. Change of flexural strength with change of MK parentages and different curing times

The Fig. 2 also reveals non-linear behavior: as the MK percentage increases from 5% to 15%, and then to 20%, the flexural strength changes in a non-consistent manner. At certain times (especially at 150 days, or in brine curing), when MK = 15% is added, the flexural strength values are very high, particularly in long brine curing (28 days water + 120 days brine), compared to lower or higher percentages. This suggests that 15% may be approximately the optimal percentage for this formulation to achieve flexural strengthening. This could be explained by the fact that brine curing might induce additional reactions in the matrix, or that the matrix density changes in a way that reduces corrosion, leading to localized strengthening-however, this requires careful interpretation and an experimental approach.

When MK = 20% is added, the flexural strength values sometimes (especially in very long curing) do not increase at the same rate, or may be lower than the peak performance at 15%. This suggests that very high substitution can lead to performance degradation due to various factors, such as clinker dilution, or that a deficiency in the effective pozzolanic reaction may occur beyond a certain point.

3. Ultrasonic Pulse Velocity Test (UPV)

The UPV value reflects the cohesion of the concrete matrix, as well as the presence or absence of voids and micro-cracks. A higher UPV value typically indicates higher density, lower porosity, and better internal bonding between the sample components. Therefore, UPV is used as a non-destructive test to assess sample quality and correlate it with mechanical properties (such as compressive strength) (Poon et al., 2001; Homayoonmehr, 2022).

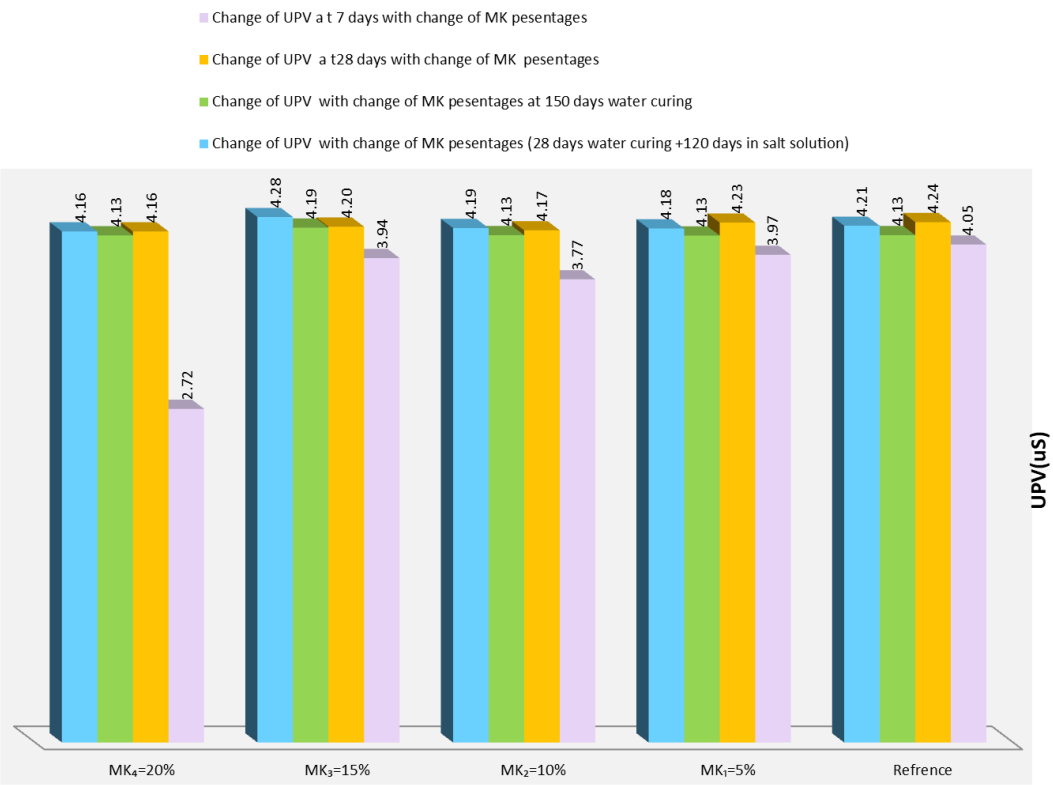


Fig. 3. Change of UPV strength with change of MK parentages and different curing times

The results in Fig. 3 show that the values recorded after long curing (28 days water + 120 days saline) yield the highest or near-highest UPV values in most mixes, while short-time (7 days) measurements are lower, especially in mixes with high MK content. This suggests that the positive effect of Metakaolin on matrix homogeneity develops over time (pozzolanic reactions gradually integrate and fill micro-cracks over time), so the UPV improvement becomes more pronounced at later curing times. (Ferreira et al., 2016; Yuan et al., 2022).

A significant decrease in UPV values was observed in some mixes (particularly MK20 and MK15 at 7 days) compared to the reference mix. This is because the high Metakaolin substitution may reduce the available active cement content for the early hydration stage. If appropriate additives are not provided or the water content is not adjusted, the early paste may be less cohesive or contain temporary water voids, thus reducing the pulse velocity in the first few days (Poon et al., 2001; Khatib & Wild, 1996).

4. Bulk density

Fig. 4 shows that the concrete density changes at different percentages of Metakaolin (MK) addition and for different curing times (7 days, 28 days, 150 days in water, and 28 days in water + 120 days in brine).

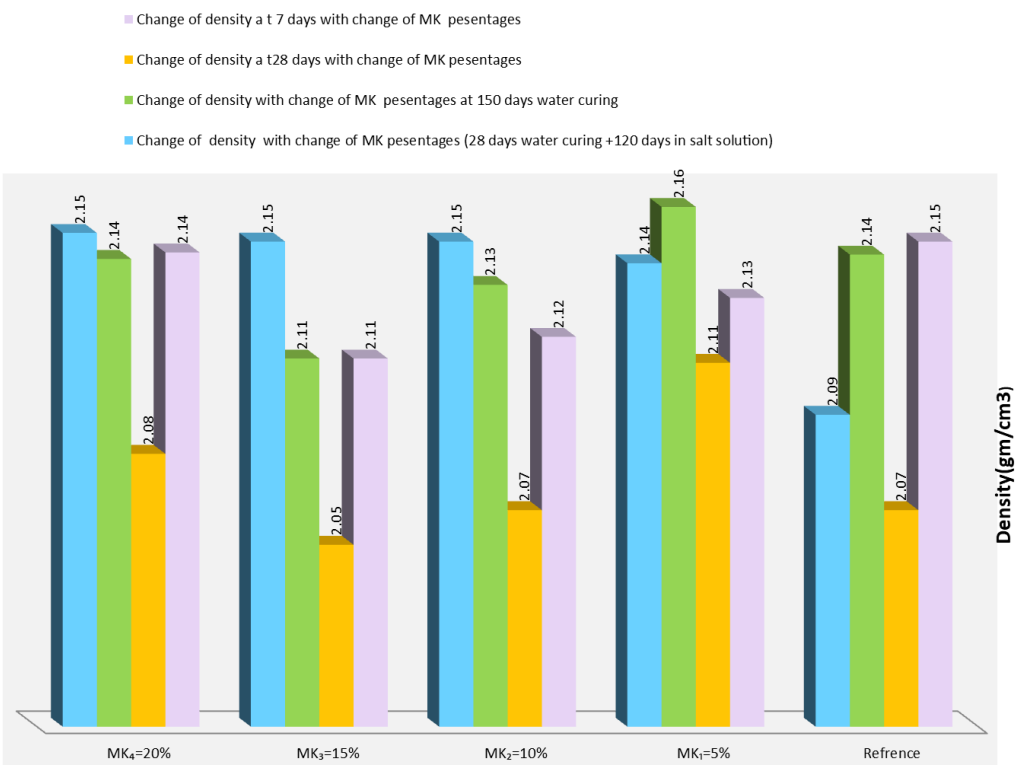


Fig. 4. Change of Bulk density with change of MK parentages and different curing times

At 7 days of water curing, the density is slightly lower than or close to the reference density in some percentages. At 28 days of water curing, the density drops significantly in some cases (MK = 20% → very low density), indicating that high substitution can lead to a relatively early decrease in density.

While the long-term water curing time of 150 days resulted in increased density at some percentages of Metakaolin addition, such as MK = 5%, it yielded a density higher than the reference.

In the case of brine curing (28 days in water + 120 days in brine), the densities are often higher than or equal to the highest values of the water curing in some percentages.

Conclusion

- The use of Metakaolin in well walls, ratios MK ≈ 10–15% achieves the best flexural strength performance in all curing conditions, with no adverse effects from salty exposure.
- The addition of metakaolin can reduce the reliance of traditional concrete on a long water curing period, and the addition must be in optimal proportions. It provides good mechanical performance, high durability, and high density.
- Metakaolin improves the resistance of cement mortar to chloride ion penetration and hydration caused by salts.
- When samples are cured for (28 days in water + 120 days in saline solution), the density is often greater than, or in some cases equal to, the highest values obtained through water curing.
- Since metakaolin is a partial cement substitute, its optimal use reduces environmental impact. However, it is essential to ensure that its strength and durability are not negatively affected, which would compromise its structural performance.

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