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Impact of Different Curing Techniques on the Physical, Microstructural, and Hydration Characteristics of Cement Mortar Contaminated with Heavy Crude Oil

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Abstract: *Crude oil contamination is a serious environmental issue, particularly in regions where it affects soil and groundwater. Repurposing oil-contaminated materials in construction offers a potentially cost-effective solution for recycling waste and reducing pollution. This study examines the impact of curing methods and crude oil contamination on the mechanical properties of cement mortar. Mortar samples with varying oil concentrations were subjected to three curing techniques: air, water, and sealed plastic. Results indicate that water curing produced the highest compressive strength across all contamination levels, owing to sustained moisture availability. In contrast, air-cured samples exhibited the lowest strength, particularly in uncontaminated specimens and those containing 2% oil content. At a contamination level of 10%, the curing method had minimal influence on strength. Heavy crude oil was found to have a more pronounced negative impact than light crude oil, forming a dense layer around cement particles that restricts hydration, increases porosity, and weakens the cement matrix. While low levels of crude oil contamination did not show a significant effect, suggesting potential for specific civil applications, higher concentrations of heavy crude oil substantially compromised the structural integrity of the mortar.*

Keywords: *Heavy crude oil, contamination, microstructure, remediation, mechanical properties*

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I. Introduction

Sand contaminated with crude oil and other hydrocarbons has emerged as a global environmental challenge. Such contamination risks human health, disrupts ecosystems, and alters the physical and chemical properties of the affected sand [1, 2]. Traditional remediation methods for oil-contaminated sand are often costly and complex, necessitating more innovative and cost-effective solutions [3-5]. One promising approach is to repurpose contaminated sand by mixing it with cement and using this mixture as an alternative construction material. This method addresses disposal issues and mitigates environmental impact [6-8]. Recent research in Australia has highlighted that light crude oil impacts fine sand's physical and mechanical properties and the quality of mortar and concrete produced with contaminated sand. Studies indicate that higher oil contamination levels decrease water absorption, permeability, contact angle, frictional angle, and cohesion. Notably, a 1% oil contamination level resulted in the highest cohesion (10.76 kPa) and a 10% enhancement in shear strength, demonstrating the potential of using such waste material in construction [9]. Further analysis revealed that cement mortars incorporating oil-contaminated sand achieved up to 19% higher compressive strength when cement and water were mixed prior to adding the sand due to improved cement hydration. Mortars cured in a fog room exhibited up to 45.6% higher compressive strength than those cured under other conditions, such as water, air, or plastic bags. Scanning electron microscopy showed fog room-cured mortars had lower total porosity, smaller capillary pores, and denser calcium silicate hydrate formations [10].

An investigation into concrete containing oil-contaminated sand revealed that density decreased with increasing crude oil content due to higher surface voids and total porosity. Concrete with 1% light crude oil contamination achieved the highest compressive and splitting tensile strengths due to optimal sand cohesion. However, strength properties declined with contamination levels above 1% as the bond between cement paste and aggregates deteriorated. Concrete beams with 6% oil contamination only showed a 20% reduction in moment capacity compared to beams made with uncontaminated concrete [11]. In contrast to light crude oil, heavy crude oil has a higher density and specific gravity, potentially enhancing the cohesion between sand particles and reducing total porosity [12]. This improvement may positively affect the properties of concrete properties and decrease water absorption during mixing, thereby enhancing the cement hydration process [13]. Given that Petroleum Development Oman (PDO) generates approximately 53,000 tons/year of petroleum-contaminated soil (PCS), which poses disposal challenges due to inadequate waste management facilities, repurposing heavy oil-contaminated sand could offer a sustainable alternative [14]. This study explores the impact of various concentrations of heavy crude oil on the properties of cement mortars, comparing these effects with those observed with light crude oil. It examines how different curing methods—ambient, plastic bags, and water—affect mortars with oil contamination levels of 0%, 2%, and 10%, focusing on changes in the concrete's physical and mechanical properties. This research further examines heavy crude oil's impact on cement mortar's microstructural characteristics, focusing specifically on alterations in porosity and the hydration process. The findings reveal how crude oil presence disrupts typical microstructural formation, affecting the density and durability of the mortar. Based on these insights, the study suggests possible applications for oil-contaminated sand within the construction industry, emphasizing its potential as a sustainable alternative building material. Repurposing contaminated sand could mitigate environmental damage while offering a resource-efficient solution for construction.

II. Materials And Methods

2.1 Sample preparation

Air-dried fine sand was utilized in this study, with its Particle Size Distribution (PSD) analyzed according to AS-1141-2011 [22], as shown in Figure 1. Results indicated a maximum particle size of less than 10 mm.

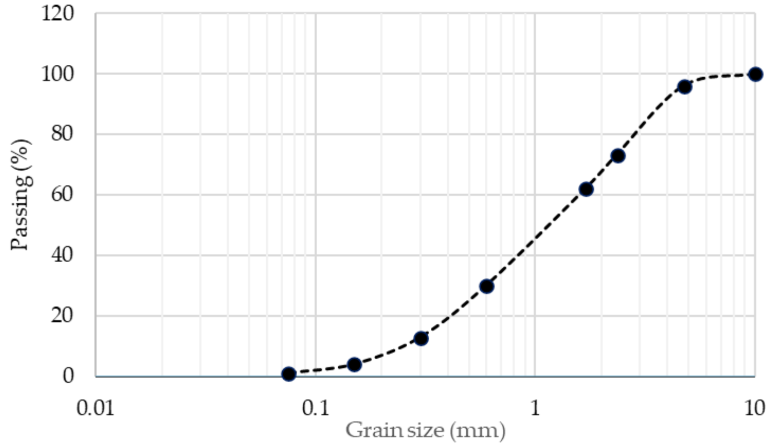

Figure 1: Particle size distribution curve of the sand

To prepare samples, dry sand was manually mixed with heavy crude oil at concentrations of 2% and 10% by weight, with uncontaminated sand (0% oil) as the control. The sand-oil mixtures were stored in plastic containers for 72 hours to ensure thorough homogenization.

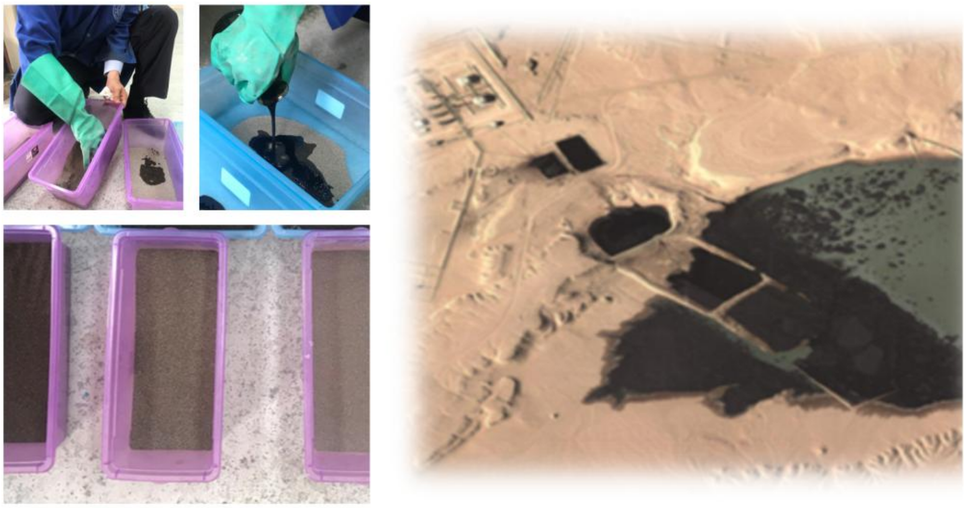

Figure 2 Preparation of the samples

Oman Petroleum Development (OPD), through Sultan Qaboos University, supplied the heavy crude oil used as the contaminant in this experiment. The crude oil's detailed specifications are provided in Table 1 below.

Table 1 Specification of heavy crude oil used in this experiment [15]

Density at 15°C, kg/m3	868.7
°API	31.3
Bbl/mt	7.253
Acidity, mg KOH/g	0.64
Sulphur, wt%	1.410
Hydrogen Sulphide, mg/kg	<1
Mercaptan Sulphur, mg/kg	160
Viscosity, cSt at 10 °C	51
Viscosity, cSt at 50 °C	10
Pour Point °C	-36
Total Nitrogen, wt%	0.121
Wax, wt%	-
Wax Appearance Temperature	-
RVP at 37.8 °C, kPa	16
Water, vol%	0.1
NaCl, mg/kg	59.0
Nickel, mg/kg	10.1
Vanadium, mg/kg	10.1
Iron, mg/kg	4.0
Mercury, µg/kg	2.0

2.2 Preparing and Casting of Mortar

The mortar mix was made following AS 2350.12 (2012) with a ratio of 3:1:0.5. The moulds were filled in three uniform layers, each compacted using a 1.8 kg steel rod. Laboratory procedures were performed at lab ambient temperature. Following 24 hours, the specimens were removed from their moulds and exposed to various curing conditions for up to 28 days, which included water immersion, air exposure, and encapsulation in plastic bags. Figure 3 presents a summary of the mortar preparation and casting procedure.

Figure 3 Preparation of the samples

This study details three curing methods for concrete specimens: air curing (A), water curing (W), and plastic bag curing (PB), as depicted in Figure 3. In air curing, specimens are exposed to ambient conditions for 28 days. Water curing involves storing specimens within plastic bags filled with tap water to minimize oil leaching. Plastic bag curing uses 125 µm thick plastic bags placed inside UV-stabilized polypropylene bags and cured in air. Each method aims to prevent oil leaching, ensuring consistent oil content throughout the curing process.

2.3 Compressive Strength Testing

Compressive strength tests were conducted on all specimens after a 28-day curing period. Each specimen was tested for failure using a 2-channel Automatic Cube and Cylinder Compression Machine (CT340-CT440) at a 1.5 kN/min loading rate, as shown in Figure 4(b). To ensure even load distribution, the ends of the cylindrical specimens were ground smooth, and rubber capping was applied to provide a uniform surface during testing, as depicted in Figure 4(a).

Figure 4 Compressive strength machine

2.4 Porosity

The internal structure of the specimen sections was analysed under a microscope at 65x magnification, as seen in Figure 5. The TBitmap programme was used to examine resin colour, facilitating the distinction of pixels associated with pores in the pictures. The porosity examination was conducted immediately after the compression testing, after a 28-day curing period.

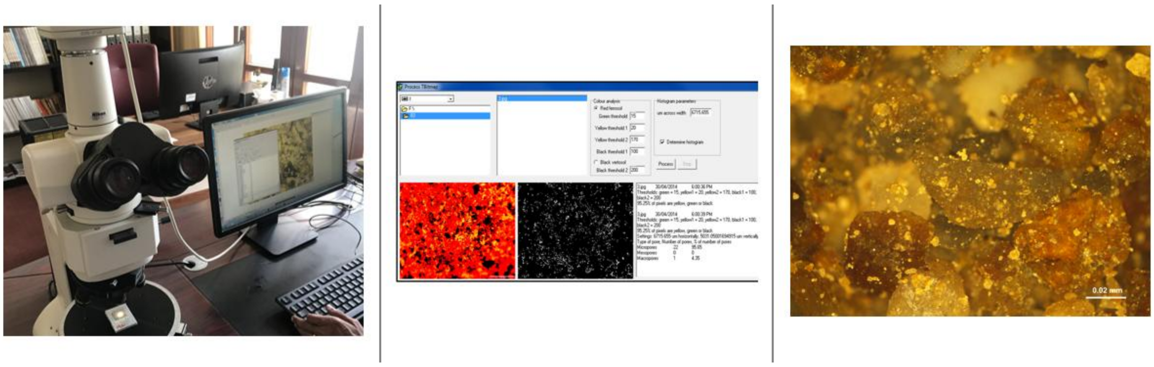

Figure 5 shows the surface of specimens with pores.

III. Results and discussions

3.1 Failure behavior

Figure 6 illustrates the characteristic failure mechanisms of mortar with heavy crude oil during compression testing. Two distinct failure mechanisms were identified: axial splitting and shear failure. A transitory alteration occurred between the failure modes when multiple failure modes were evident; for example, a specimen demonstrating axial splitting sometimes exhibited shear failure characteristics despite axial splitting being the predominant failure mode. This occurred with specimens containing 0% and 2% crude oil contamination and cured in water and plastic cover. In contrast, axial splitting was the dominant failure mode for the specimens when 10% of heavy crude oil was used under all curing methods. This may be correlated to the percentage of the porosity in the specimens, as shown in the outside sample surface. It can be observed that the surface porosity increases as the level of oil contamination increases. Hudyma, et al. [16] have indicated that the porosity of the specimens affects the failure modes. Shear failure in mortar is a more robust and enduring failure mechanism than brittle splitting failure, particularly with increasing porosity [17]. The structure is less susceptible to pore-induced flaws under shear pressures than tensile or axial forces. The mortar matrix distributes the stress, enabling the material to depend on its compressive strength, which is often more resistant to voids and microcracks [18]. Porosity, albeit diminishing overall strength, does not significantly impede load transmission in shear compared to tension. Conversely, when mortar undergoes tensile loads, pores function as stress concentrators, initiating fracture and propagation; increased porosity in mortar renders it more susceptible to brittle splitting or tensile failure, with even little porosity increments leading to a significant decline in tensile strength [19].

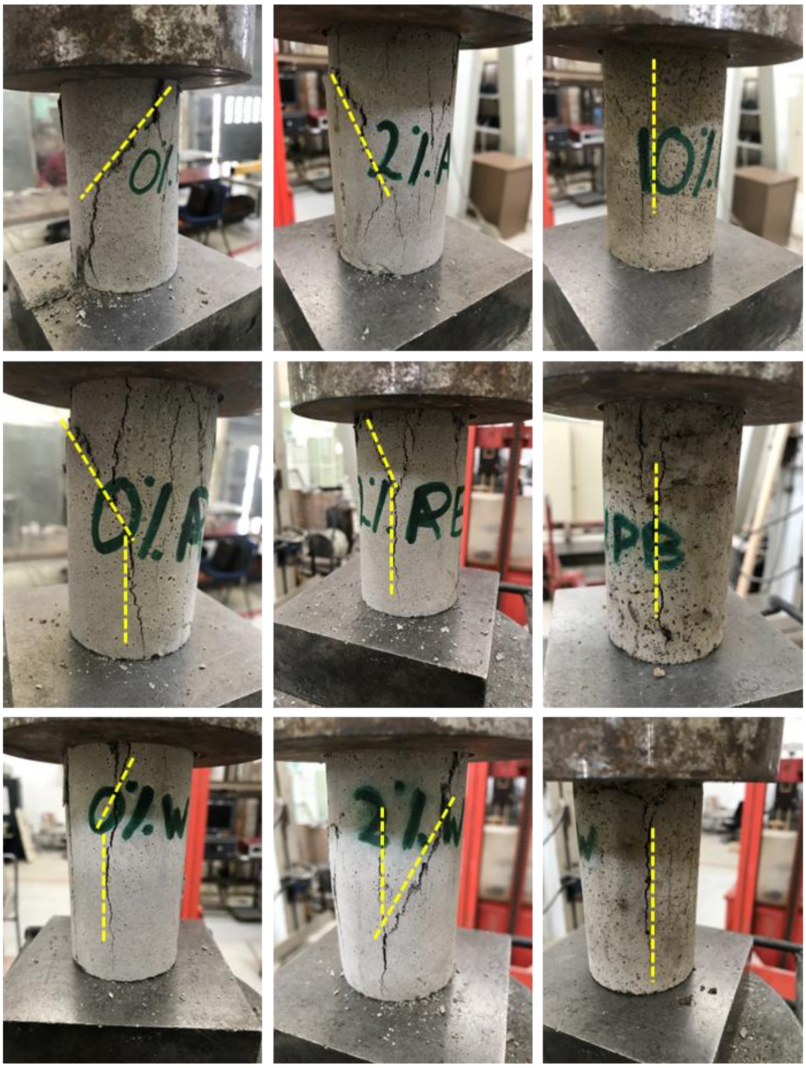

Figure 6 Failure modes of cylinder mortar in compression under different curing methods

3.1 Effect of heavy crude oil content on the mechanical properties

Figure 7 presents the average compressive strength of mortar with varying crude oil content under different curing conditions. For uncontaminated samples (0%), water curing yielded the highest compressive strength at 19 MPa. The compressive strength decreased by 9.7% under plastic bag curing and by 22% under ambient conditions. In samples with 2% heavy crude oil contamination, the compressive strength was nearly the same for ambient and plastic bag curing conditions, achieving 12.4 and 12.3 MPa, respectively. At this contamination level, water curing again resulted in the highest strength, with an average of 17 MPa. However, when crude oil contamination increased to 10%, the influence of curing methods on compressive strength became negligible. The strength average of the ambient, plastic bags and water curing when 10% of crude oil contamination was used was 8.8, 9, and 8.9 MPa, respectively.

The development of higher compressive strength in mortar cured in water compared to the other curing methods, ambient and plastic bags, is attributed to sufficient moisture during the curing process, which allows the hydration process to complete. This result indicates that it is essential to keep the specimens in high and constant humidity during curing to maintain the right level of moisture needed to complete the hydration process. This finding is supported by Taylor [20], who indicated that continued curing at a relative humidity of 90% better facilitates the hydration in cement and leads to red microstructural development. Furthermore, Wang and Park [21] stated that high humidity can enhance hydration even under the same temperature. Conversely, the lower compressive strength observed in mortar cured at elevated temperatures is attributed to the rapid formation of hydrates, which increases porosity and reduces ultimate strength [22]. Moreover, it’s been reported by Ezziane, et al. [22], that the compressive strength of concrete cubes cured in water at 7 and 28 days is higher than that of those cured in air. This increase in the compressive strength and other mechanical properties of concrete induced by water curing is due to the improved gel/space ratio inside the concrete [23].

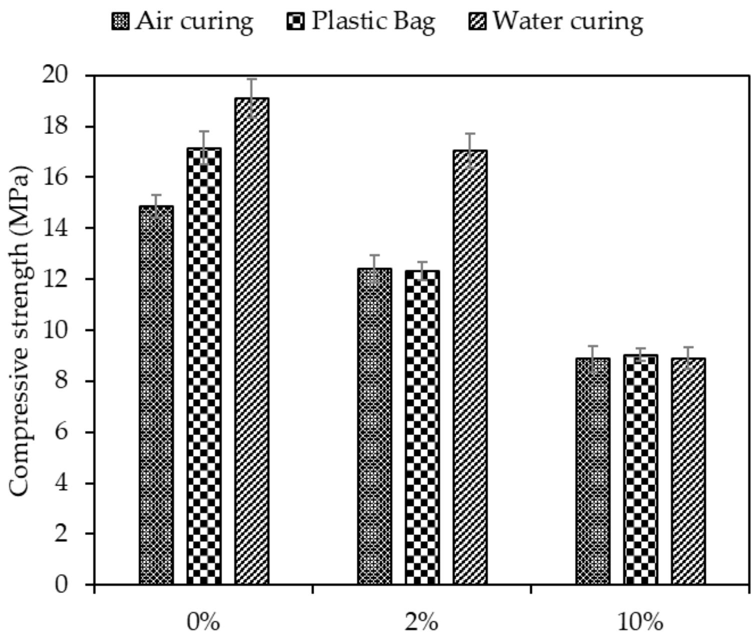

Figure 7 Effect of curing method on the mechanical properties of mortar with different heavy crude oil content %.

3.2 Effect of Curing Method on Heavy Crude Oil-Contaminated Concrete Properties

The mortar cured at ambient temperature exhibited the lowest compressive strength for the uncontaminated samples. Ambient curing, particularly in dry or fluctuating environments, produces less moisture for cement hydration, a crucial process that forms bonds with cement particles [20, 24]. Without enough moisture, hydration is incomplete, resulting in a weaker microstructure and lower compressive strength. Temperature variability also affects the hydration process, especially if temperatures fall below ideal conditions, leading to differential shrinkage and cracking, further weakening the mortar [25]. However, introducing heavy crude oil to the mix design has affected the compressive strength under all curing methods (ambient, plastic cover, and water curing). At 2%, the compressive strength decreased by 10%, 28%, and 16%, respectively. One interesting finding from this study regarding the effect of curing methods on the compressive strength of mortar with 0% and 10% crude oil contamination reinforces the results of previous studies [26-28], which have all observed that the curing method affected the compressive strength. Notwithstanding this result, the study also revealed that 2% of crude oil contamination, even under different curing conditions, had no significant effect on the compressive strength. Introducing heavy crude oil into mortar mix designs typically decreases compressive strength because the oil disrupts the cement's hydration process, which is essential for strength development. The oil can create a barrier on cement particles, limiting water access and preventing complete hydration. Additionally, oil droplets may weaken the bond between cement paste and sand particles, reducing overall cohesion. This effect varies by curing method: ambient curing might be more vulnerable to these disruptions, while water curing could mitigate them slightly but not eliminate the negative impact [29]. Conversely, a previous study on light crude oil contamination in concrete showed that crude oil levels up to 2% had no significant effect on the mechanical properties of the concrete. It was concluded that 2% crude oil contamination did not significantly impact compressive strength. This outcome was attributed to the degree of wettability achieved by adding crude oil to dry sand, which enhanced the hydration process. The amount of water absorbed by the sand was reduced by increasing the wettability of dry sand with light crude oil.

Figure 8 illustrates the impact of curing methods on the compressive strength of mortar containing varying percentages of heavy crude oil compared to light crude oil. Specifically, Figure 8a depicts the samples with heavy crude oil (HCO), while Figure 8b displays those with light crude oil. Overall, the results indicate that the samples with heavy crude oil exhibited lower strength than the light crude oil samples and the control sample. This general reduction in strength is attributed to the elevated temperature during casting, which was approximately 40°C for the heavy crude oil samples prepared at SQU, Oman, during the summer. In contrast, the light crude oil samples were prepared in Australia at room temperature of 22°C ±2. Elevated casting temperatures accelerate hydration, leading to rapid setting and strength gain in the early stages. However, this accelerated reaction can reduce long-term compressive strength, primarily if adequate curing methods are not implemented to retain moisture and control temperature [30, 31]. Several studies have shown that casting concrete at high temperatures can increase porosity and micro-cracking, weakening the material's compressive strength over time. This effect is particularly noticeable in climates where day-to-night temperature fluctuations contribute to further drying and shrinkage, ultimately compromising structural integrity [32].

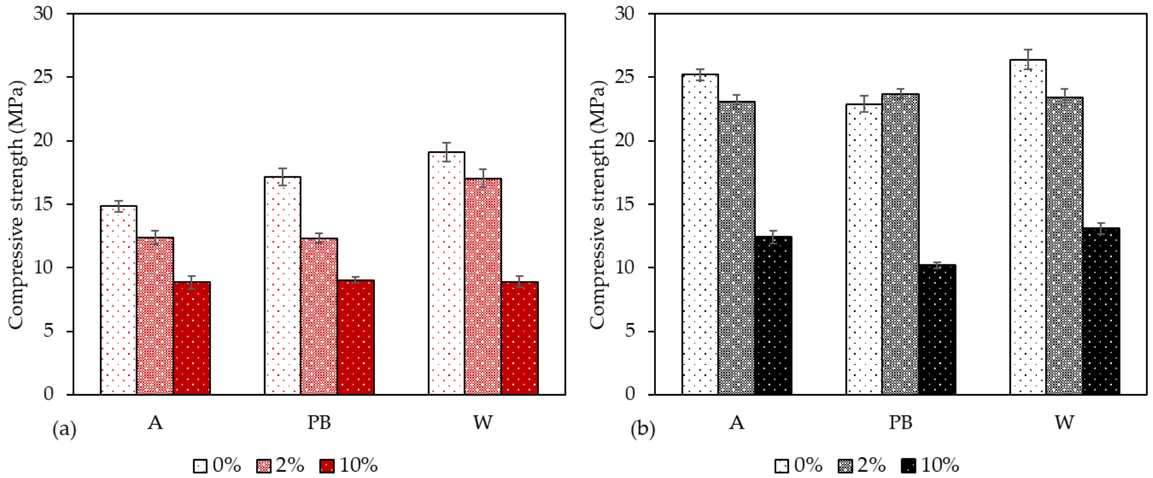

Figure 8 Effect of Curing Method a) Heavy crude oil b) light crude oil

Heavy crude oil has a more significant proportion of asphaltenes and heavy hydrocarbons, which are substantial molecules that exhibit low volatility. These elements enhance its elevated viscosity and density [33, 34]. When heavy crude oil infiltrates or encounters concrete, its chemical composition reacts with the concrete matrix. This contact may lead to modifications in the concrete's characteristics. Heavy crude oil and concrete interaction entails penetration and possible chemical reactions. In concrete buildings, oil penetration may result in heightened porosity and the development of oil-saturated regions inside the concrete [35, 36]. The dense hydrocarbons may occupy the spaces within the concrete matrix, resulting in alterations to its mass and overall structural integrity. A primary consequence of heavy crude oil on concrete is plastic shrinkage cracking. The oil infiltrates the concrete surface, potentially hindering the hydration process of cement, which is crucial for strength development. Concrete polluted with heavy crude has heightened plastic shrinkage, diminishing its durability [37]. Moreover, heavy oils may modify the water-to-cement ratios in concrete, reducing material compressive strength. The oil's absorption may displace water essential for the hydration of cement particles [38]. This displacement leads to insufficient adhesion between the aggregates and cement paste, eventually undermining the mechanical strength of the concrete [39, 40]. In contrast, light crude oil has a more significant percentage of lighter hydrocarbons. Its reduced viscosity facilitates more effective evaporation and migration through materials, resulting in diminished long-term interactions with concrete. Heavy crude oil presents higher contamination hazards to concrete during oil spills than light crude oil [41, 42]. The dense hydrocarbons from the oil may infiltrate more profoundly into the concrete, resulting in more severe and perhaps irreparable damage. In contrast, light crude oil has a lesser adverse impact on the mechanical characteristics of concrete. Due to its reduced viscosity, light crude oil exhibits more volatility, leading to an increased evaporation rate. Light crude oil, despite causing structural damage, has less disruption to concrete's hydration process and absorption levels compared to heavy crude oil, resulting in less severe impacts [38, 42].

3.3 Effect of Total Porosity on Mortar with Heavy Crude Oil

Figure 8 depicts the surface porosity of mortar samples with and without heavy crude oil contamination. The figure indicates that bigger air holes exist in a mortar with sand polluted by 10% light crude oil, in contrast to samples with 0% and 2% contamination. Furthermore, while loading, specimens contaminated with 10% crude oil did not produce any cracking noises, even until failure occurred. This may be ascribed to the moisture retained inside these specimens post-failure. Samples with 10% crude oil contamination retained more residual oil than those with 2% contamination or control samples.

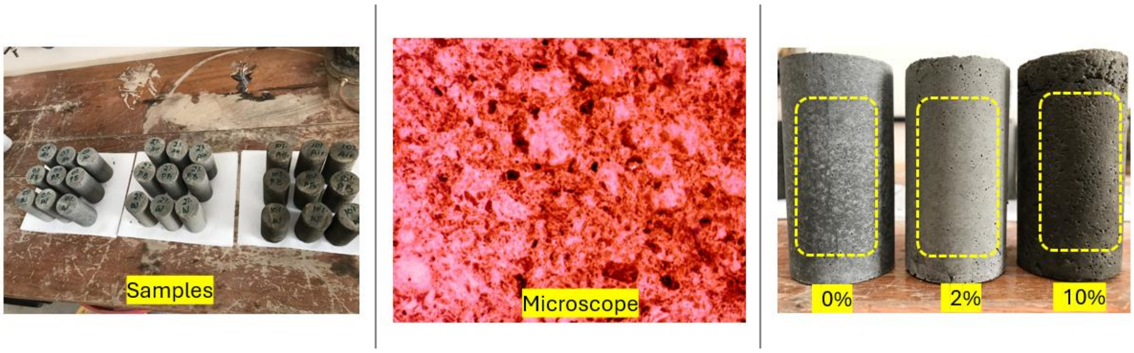

Figure 8 Surface Porosity Observations: Sample, Microscope Image for Software Analysis, and Outer Surface

Figure 9 shows the specimen's average internal total porosity with three oil contamination levels (0%, 2%, and 10%) as a function of four different curing methods (A, FR, W). Higher porosity was observed when the specimens were cured with A and plastic cover compared to W curing methods for uncontaminated samples and with 2%. In contrast, the lowest percentage of total porosity at the same percentage (0% and 2%) was observed with specimens cured in water. However, when 10% of heavy crude oil was used, the porosity was the highest and quite similar for the three curing methods. The saturation state of the sample, with 10%, significantly affected the property, and as a consequence, the strength decreased regardless of the curing method used.

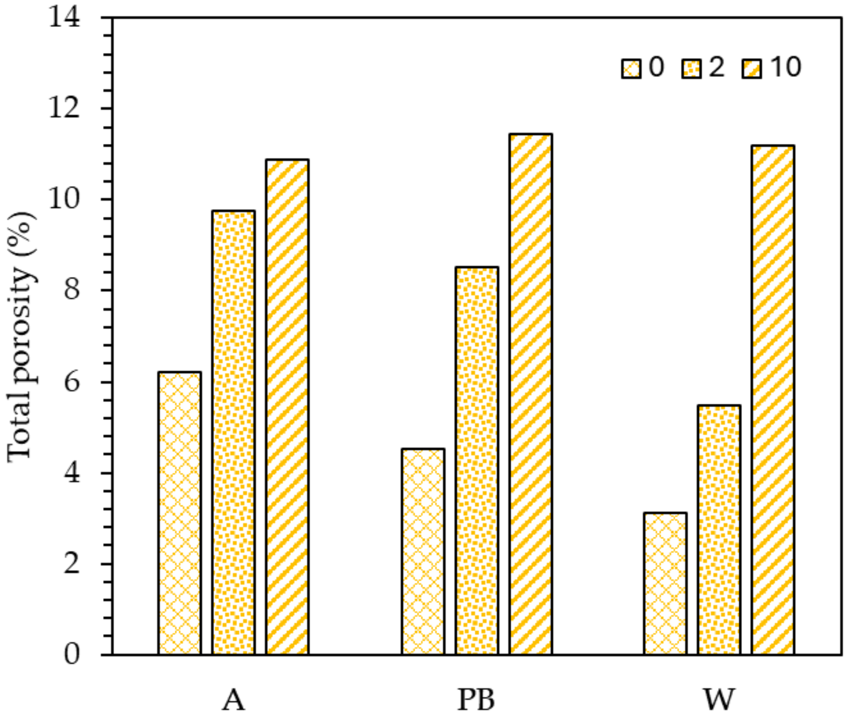

Figure 9 Total Porosity of Mortar with Varying Heavy Crude Oil Contents and Curing Methods

The plastic cover curing method resulted in lower porosity and higher strength than ambient curing. These findings contrast with previous studies on the effect of light crude oil on concrete's mechanical properties [11, 43]. The primary difference can be attributed to temperature conditions; in this study, testing occurred at Sultan Qaboos University in Oman during summer, where average temperatures reached approximately 40°C. Such high temperatures likely accelerated sample drying, slightly impacting hydration, increasing porosity, and, consequently, influencing the material properties. In addition, among all the different curing methods, the specimens treated in water (W) had the best compressive strength and the lowest total porosity. The constant availability of moisture during the curing time, which facilitates complete hydration of the cement particles, is believed to be responsible for the lower porosity found in specimens that were cured using water. As a result of this continuous hydration, the creation of pores within the matrix is reduced, resulting in a denser and more cohesive structure. Compared to other curing techniques, the compressive strength of water-cured specimens is much higher than that of other specimens. This is because the matrix is well-hydrated and has a low porosity. On the other hand, the porosity dramatically increased across all curing techniques when the heavy crude oil concentration increased to 10%. This was most likely caused by interference with cement-water processes, which either delayed or prevented the complete hydration of cement particles [44, 45]. The presence of heavy crude oil contributed to the increase in porosity and the decrease in compressive strength. Et al. [4] observed that extensive oil contamination in concrete significantly affects compressive strength. They attributed this impact to crude oil coating cement particles interrupting hydration.

3.4 Effect of heavy crude oil on the hydration process

Figure 10(a) displays the heat flow (mW/g), while Figure 10(b) depicts the accumulated heat (J/g) for samples contaminated with heavy crude oil at levels of 2% and 10%, alongside an uncontaminated control sample. Table 2 succinctly delineates the heat produced after 1 hour, 24 hours, and 72 hours for the uncontaminated sample compared to those containing 2% and 10% crude oil, respectively. A minor decrease in heat output was seen in samples with 2% and 10% crude oil during the first hour. After 24 hours, heat output decreased by 2.7% for samples containing 2% crude oil and 9.7% for those with 10% crude oil. This trend persisted at 72 hours, with decreases of 2.8% and 8.6% for the 2% and 10% contamination levels, respectively. The data indicate that elevated crude oil concentration progressively hinders heat production, which is detrimental to cement hydration. Despite the heat flow shown in Figure 10(a) demonstrating just a marginal decrease between 0% and 2%, it is significant that the 2% sample had the greatest peak value among the contaminated samples. The most significant peak values for 2% and 10% crude oil were 0.016729 W and 0.015535 W, respectively, in contrast to 0.017347 W for the control sample. The results shown in Figure 10(a-b) and Table 2 indicate that heavy crude oil pollution significantly affects the hydration process by postponing the hydration processes of both C_3S and C_3A [46].

Table 2 Effect of heavy crude oil on the heat flow and maximum peak

Sample	0%	2%	10%
Heat after 1 hour (J)	0.0084454	0.0079278	0.0070882
Heat after 1 day (J)	816.72	794.65	736.89
Heat after 3 days (J)	1268.2	1232.3	1159.1
Peak max (W)	0.017347	0.016729	0.015535

Conversely, increased crude oil content from 2% to 10% resulted in a notable decrease in cumulative heat and heat flow during hydration. For example, the cement pastes with 2% heavy crude oil contamination exhibited a maximum exothermic peak of 2.8 mW/g. The cement paste with 10% heavy crude oil contamination showed a slightly lower peak of 2.6 mW/g. In comparison, the control samples, which had no crude oil contamination, displayed a peak of 2.9 mW/g. This decrease in heat flow with higher crude oil content indicates a diminishing reactivity and heat release efficiency, which may be attributed to the excessive crude oil interfering with the chemical reaction between the cement particles. The results suggest that while low crude oil levels have no significant effect on the hydration process, raising the heavy crude oil to 10% has shown a significant effect. Crude oil consists of a wide range of organic compounds, including aliphatic, aromatic, and heteroatomic hydrocarbons, whose presence during the hydration process can introduce multiple variables. This agreed with a previous study conducted by Almabrok, et al. [29] who revealed that high content of unsaturated oils in canola oil led to a notable reduction in strength with even small additions, suggesting an interaction between the oil and the hydrating cement phases. Conversely, oils with higher levels of saturated aliphatic compounds exhibited a lesser impact on the hydration process.

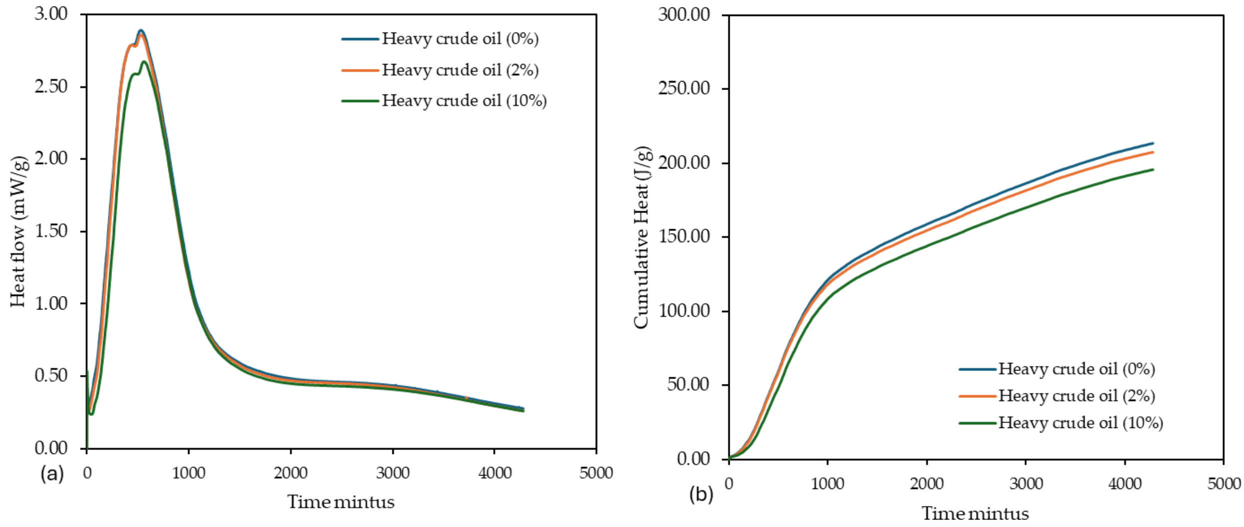

Figure 10 Effect of heavy crude oil on the hydration process (a) heat flow (mW/g) and (b) cumulative heat (J/g)

IV. Conclusion

Among the curing methods tested—air curing, water curing, and sealed plastic curing—water curing consistently resulted in the highest compressive strength for uncontaminated samples and those with 2% heavy crude oil contamination. This increase in strength is attributed to the enhanced hydration supported by the ample moisture provided during water curing. In contrast, air-cured specimens demonstrated the lowest compressive strength, particularly in uncontaminated samples and those with 2% oil contamination. This reduced strength likely results from the high ambient temperatures at Sultan Qaboos University in Oman, where summer temperatures average around 40°C. Such heat accelerates drying, reduces hydration efficiency, and increases porosity, weakening the mortar's mechanical properties. However, at 10% heavy crude oil contamination, the choice of curing method had minimal effect on compressive strength, likely because the high oil content saturated the samples, hindering hydration regardless of curing conditions.

Increasing heavy crude oil content to 10% led to a substantial increase in porosity, significantly weakening the mortar’s compressive strength. This reduction in strength is due to crude oil interfering with the cement matrix, creating an oily coating around cement particles that prevents proper bonding with water and impedes the hydration process. The result is a porous, less cohesive structure as water cannot adequately reach the cement particles. This increased porosity and compromised hydration process degrade the mortar’s structural integrity, highlighting the detrimental impact of heavy crude oil contamination levels.

Moreover, at comparable oil concentrations, heavy crude oil exhibited a more pronounced negative effect on the mechanical properties of mortar than light crude oil. This difference is attributed to heavy crude oil's unique physical and chemical properties, such as higher viscosity and a more complex molecular structure. Heavy crude oil forms a thicker layer around cement particles, more effectively blocking cement-water interactions than light crude oil, thereby limiting hydration efficiency, increasing porosity, and weakening the bonds within the cement matrix. Additionally, the greater density of heavy crude oil may further inhibit the formation of a cohesive structure, leading to a more substantial reduction in compressive strength than light crude oil. Finally, while 2% heavy crude oil content minimizes the hydration of cement pastes, increasing the content to 10% significantly hinders hydration. This inhibition is evidenced by decreased cumulative heat and heat flow, directly impacting the reactivity and heat release needed for optimal cement strength development.

Acknowledgments

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