

## Evaluating the effect of mixing method on the performance of mortar containing oil

Magdi H. Almabrok<sup>1</sup>, Robert G. McLaughlan<sup>2</sup>, Kirk Vessalas<sup>3</sup>

<sup>1, 2, 3</sup> School of Civil and Environmental Engineering, University of Technology, Sydney, Australia

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**ABSTRACT:** *There is a vital need for managing oily materials resulting from the petroleum industry as their toxic and persistent nature threatens the environment. In view of oil waste remediation, current treatment technologies are either cost prohibitive and/or the treated products have to be sent to landfill without any potential end-use. Cement-based stabilisation/solidification of oil contaminated materials is an emerging method however there is limited knowledge in terms of the effect of the mixing method on the properties of the resultant cementitious mix. For this purpose, the water wet (WW) and oil wet (OW) protocol was devised to see if the observed behaviour of the mortar was a function of the mixing method rather than the ingredients. A cement-based mortar incorporating a mineral oil addition of up to 10% of the aggregates mass was used. The results indicated that the mixing method has only a small effect on the fresh and hardened properties. Increased oil content in the cement mortar was found to increase the flow and setting time whereas there was decreased wet density and air content irrespective of the type of the mixing method used. The compressive strength decreased by 75% and 77% for water wet and oil wet respectively compared to the control at 28 days of age. The mixing method has a relatively small impact overall on the hydration process. The calorimetry results showed that both mixing methods followed the same trend whereby the hydration is inhibited due to oil incorporation.*

**KEYWORDS:** *Oil, Mortar, Hydration, Mixing method, Stabilisation/solidification*

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### I. INTRODUCTION

Significant quantities of oily materials are produced annually around the world through the processes of producing oil and gas, and through associated activities. These materials can impact the environment and health; as a result, effective ways to manage these materials are needed. Cement-based stabilisation / solidification is an emerging environmental technology for this type of contamination through the encapsulation of pollutants in a cement-based matrix. Performance of monolithic solidified/stabilised material is often judged by measuring its ability to resist mechanical stress through the form of a compressive strength test. Compressive strength is linked to the progress of the hydration reaction in the mortar, and the durability of a monolithic S/S material, and is therefore a key variable [1]. It can be used as an indirect method to determine the extent to which the waste has been chemically transformed into a monolith. The overall hydration performance of cementitious mixtures can be tracked and evaluated by monitoring the evolving heat and emphasising the timing and size of the main hydration exothermic reaction which strongly affects the cementitious mixture's setting and early strength development [2].

Although there are scattered studies into the effect of organic compounds on the properties and behaviour of the stabilised and solidified products [3-6], there is limited knowledge of the effect of the mixing method on the properties of the resultant cementitious mix. The properties of cement-based mortar whether in a fresh or hardened state depend on several factors (e.g. constituents, mixing, placing, compacting and curing etc). The mixing method comprises the specified order, in which ingredients of mortar are introduced in the mixer along with the stages of addition and time of agitation at each stage [7].

The rate of stiffening mortar from plastic to solid, and the subsequent increase in strength, depends on the rate of the chemical reactions during cement hydration. The mechanistic details of cement hydration inhibition are still a subject of consideration despite the fact that it has been extensively studied. In the presence of organics, it will adsorb directly onto the surface of cement and then will form a protective layer which blocks future reactions with water; as a result, hydration will slow down [8]. Consequently, improper adhesive between the cement particles and sand will result and then the compressive strength will be reduced.

It is apparent from the literature review that the relationship between oily materials, the mixing method and the properties of cementitious materials has not, to date, been thoroughly investigated and published. Accordingly, understanding this affiliation is necessary for the development of construction materials with suitable characteristics for re-use rather than disposal.

The objective of the work reported in this article is to investigate the effect of the mixing method on the properties of fresh, hardened properties and the hydration process of the mortar containing mineral oil.

## II. EXPERIMENTAL PROGRAM

### 2.1. Materials

General Purpose Cement (Cement Australia) was used in this study which complies with the type GP requirements specified in AS 3972 [9]. The chemical properties of this cement are given in Table 1. The fine aggregate used was Calga double washed sand (Rocla Quarry Products Pty Ltd) with an absorption 0.65% and specific gravity 2.57. The particle size distribution of Calga sand by sieving method (AS 1411.11.1) [10] is illustrated in Figure 1. The water was of drinking water standard (pH 7.4; 2.29  $\mu\text{S}/\text{cm}$ ). Glenium, a polycarboxylate ether polymer based high-range water reducing admixture (HWR) (BASF Construction Chemicals Pty Ltd). The mineral oil (Castrol Motorcycle Fork Oil – SAE 10) used had a viscosity similar to medium crude oil ( $\sim 35 \text{ mm}^2/\text{sec}$  @  $40^\circ\text{C}$ ).

Table 1: Chemical properties of General Purpose Cement

Chemical entity	Proportion
Portland cement clinker	< 97 %
Gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ )	2 - 5 %
Limestone ( $\text{CaCO}_3$ )	0 - 7.5 %
Calcium Oxide	0 - 3 %
Hexavalent Chrome (Cr VI)	< 20 ppm
Crystalline Silica (Quartz)	< 0.04 - 0.5 %
Specific gravity	3 - 3.2

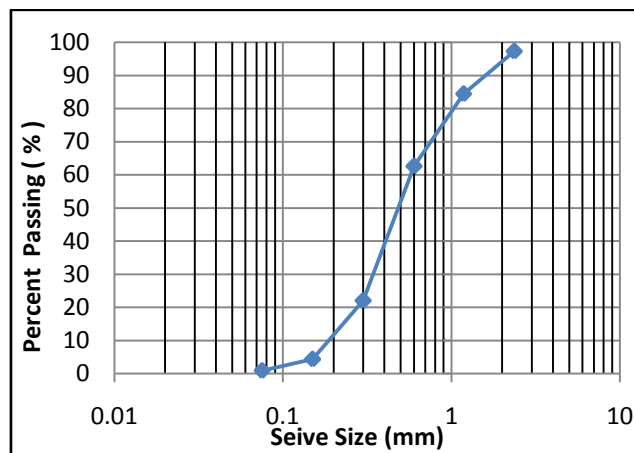


Figure 1: Particle size distribution (sieving method) of Calga double washed sand

### 2.2. Mix proportions

The composition of the mortar was in accordance with AS 2350.12 [11] with the mix proportions being 1 part of cement and 3 parts of sand (by mass) at a fixed water/cement ratio (w/c) of 0.50. Each mortar batch comprised cement (225 g), fine aggregate (675g), water (112.4g), HWR (0.2ml) with between 0 to 10% of added oil (by sand mass) which has been reported as 0 to 67.5g.

### 2.3. Preparing, casting and curing of test specimens

The batches were prepared using the Hobart mixer (model N-50 G) mixer following the procedure outlined in AS 2350.12 [11] except for the oil addition. Two different mixing protocols (water-wet; oil-wet) were used. In the method of water wet, oil was weighed (by sand mass) and added to sand then placed in plastic bowl where a spatula was used for 5 to 7 minutes to ensure thorough mixing before being added to the other ingredients. For the oil wet method, the same mixing procedure as specified for water wet was followed unless the oil was mixed with cement instead of sand. All laboratory work was conducted at  $22 \pm 2^\circ\text{C}$ . HWR (added directly to water before commencing the mixing) was used with all mixes to give reproducible flow ( $60 \pm 10\%$ ) and this provides to be most suitable for proper consolidation of the specimens by hand.

The protocol for moulding the mortar (ASTM C109) [12] was adapted and modified to minimise any impact of the protocol on any subsequent leaching tests. No mould release agents were used; instead the moulds were lined with non-stick tape. The cube moulds were sealed using zip lock plastic bags to prevent water from evaporating and were then stored in a large plastic box and kept in a moist atmosphere for 24 hours. Demoulding took place after that and specimens possessing 50 x 50 x 50 mm dimensions were sealed in zip lock plastic bags and thereafter placed in a curing tank filled with water for up to 28 days at a temperature of  $22 \pm 0.5^\circ\text{C}$ .

#### 2.4. Testing procedures

The fresh mortars were tested for flow (ASTM C 1437) [13], wet density (ASTM C 138) [14], and setting times (H-3085 Humboldt Vicat Tester, ASTM C 807) [15]. The compressive strength of hardened 50 mm mortar cube specimens was determined using an Avery Compression Testing Machine (ACTM) with a maximum capacity of 1993 kN following the listed procedures of the test method ASTM C 109 [12]. Vertical load at a rate of 1.5 kN/sec was exerted on the specimens and records were taken of the maximum load indicated by the testing machine (load at failure). F-Cal 4000 semi-adiabatic calorimeter (Calmetrix Incorporation, USA) was used for monitoring the temperature evolution of mortar mixes while the hydration reaction was taking place. F-Cal 4000 is designed for up to four standard plastic cylinders with lids as sample vials to carry the 2 kg large sample size of mortars (which is approximately half the volume of a 100 mm x 200 mm cylinder).

### III. EXPERIMENTAL RESULTS AND DISCUSSION

#### 3.1. Flow, wet density and air content

Regardless of the type of mixing method, the addition of mineral oil was generally found to increase flow but not proportional to the amount of oil (Figure 2). However, the results show that there is only a small difference in the mortar flow when water wet or oil wet are used as the mixing method thereby the mixing method has no significant effect on the flow.

Wet densities were found to decrease with increasing oil addition levels irrespective of the type of mixing method (Figure 3). The reduction in the wet density ranged from approximately 2% to 9% for both methods compared to the control mix. This reduction can be attributed to the oil free mortar ( $2270 \text{ kg/m}^3$ ) being replaced by lower density oils ( $866 \text{ kg/m}^3$ ) when it is placed in a mould of a fixed volume instead of being related to any effects of the mixing method.

As a general trend, the percentage of air content decreases with increased oil content in the mortar mixes. However, it is noted that the air content carries no significant effects since it uses different mixing methods (water wet and oil wet) where the air content in both methods varies from 3 – 7% (Figure 4).

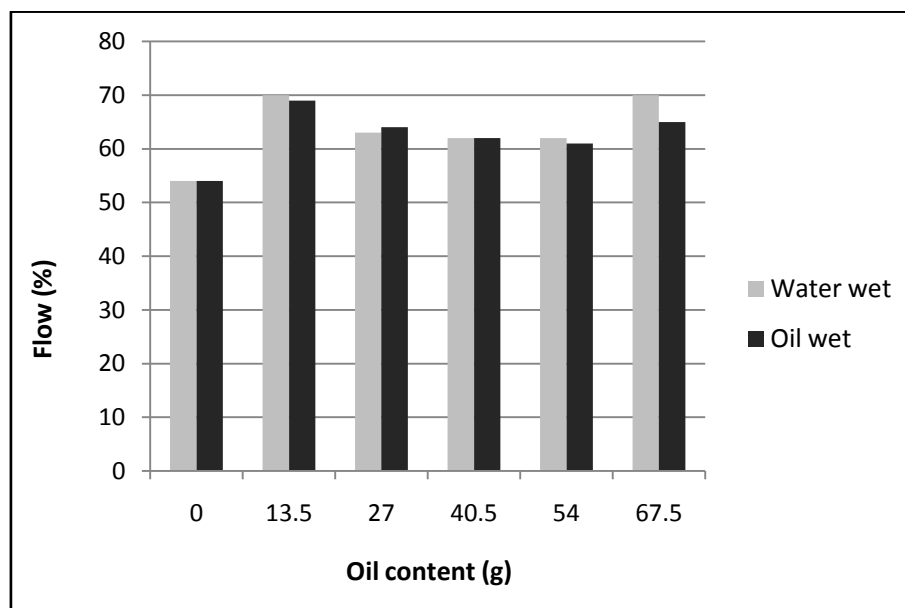


Figure 2: Effect of mixing method on the flow

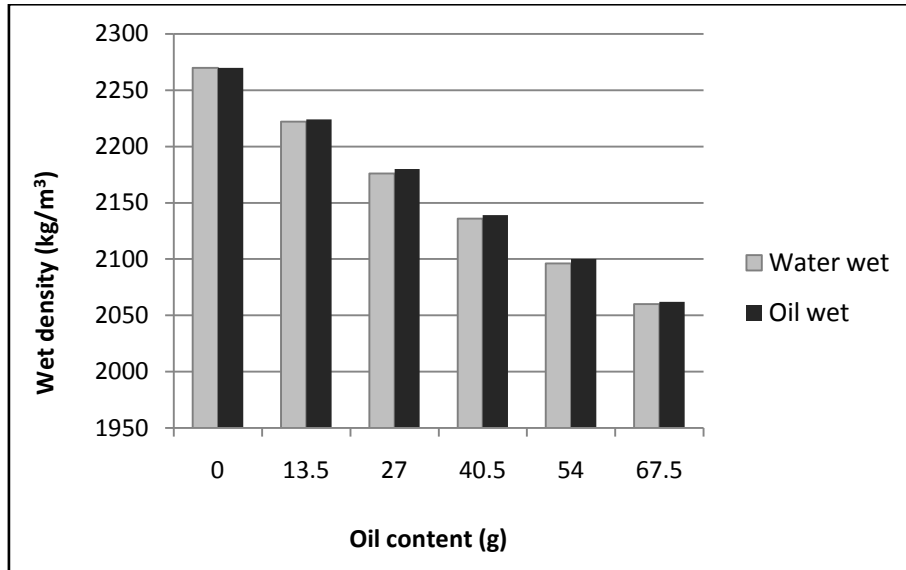


Figure 3: Effect of mixing method on the wet density

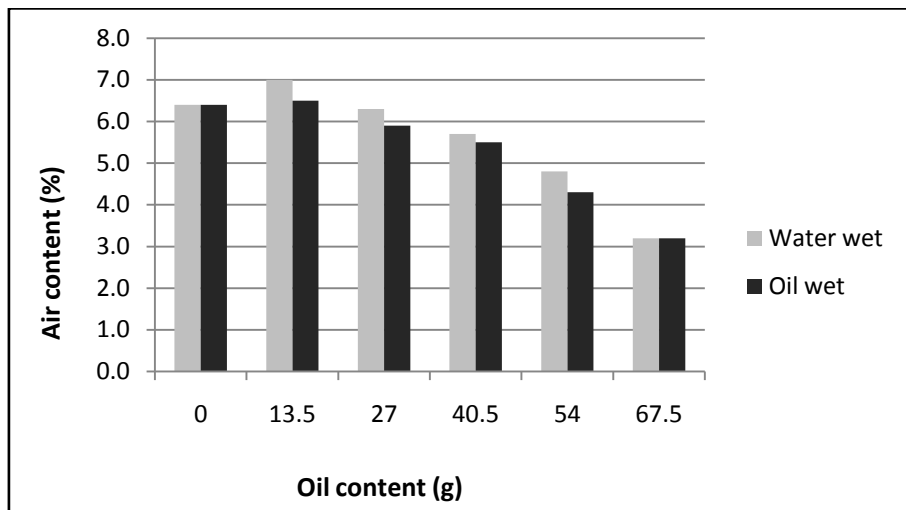


Figure 4: Effect of mixing method on the air content

### 3.2. Setting time and compressive strength

Setting time was observed to increase with the increase in the oil fraction regardless of the type of mixing method used (Figure 5). This behaviour of setting retardation is believed to occur as a result of the hydration inhibition due to the incorporation of oil with mortar [16]. Nevertheless, it was noted that the mixing method had no significant effect on the setting time. Therefore, coating the cement with oil prior to hydration (OW method) did not significantly alter the hydration process compared to the cement having more direct contact with water (WW method) during mixing.

The behaviour of 1, 7 and 28 days compressive strength of specimens incorporating mineral oil was quite different from the behaviour without oil. All mixes follow a similar trend where higher oil content in mortar results in decreased compressive strength irrespective of the mixing method used (Table 2). All the specimens showed an increase in compressive strength from 1 to 28 days by both mixing methods with the rate of strength development decreasing maturity shows that cement hydration occurs in all mixes regardless of the mixing method but this occurs to varying degrees. The little strength development from 7 to 28 days suggested that all strength development after 7 days was mainly due to the strength forming phases present at 7 days and that the mechanisms causing CSH inhibition at 7 days were still occurring at 28 days. It should be noted that the difference in the compressive strength at 28 days between the two methods varied from 6% to 12% with the incorporation of 2% and 10% mineral oil respectively. This indicates that the mixing method has a slight impact on the compressive strength.

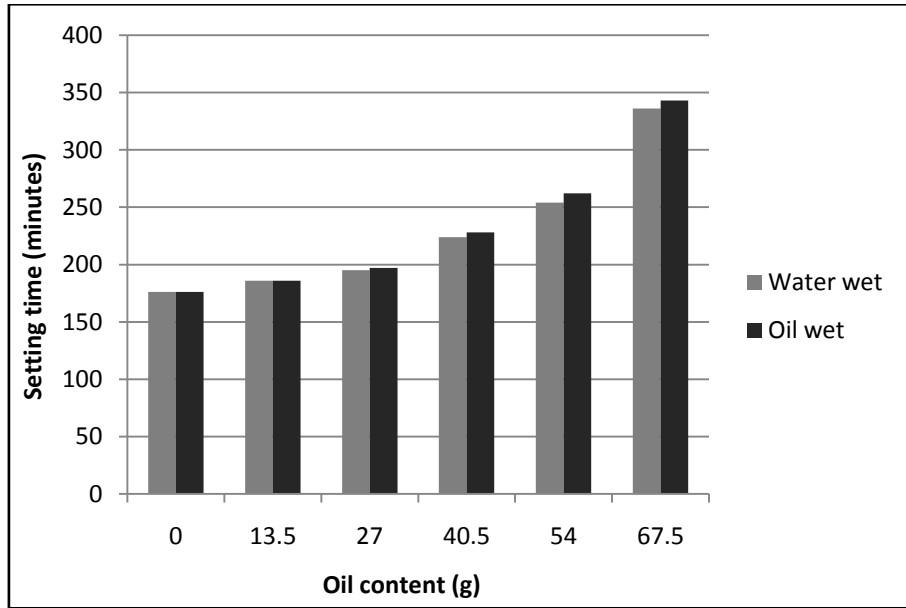


Figure 5: Effect of mixing method on the setting time

Table 2: Effect of mixing method on the compressive strength

Oil content		Mixing method	Compressive strength				
			1 day (MPa)	7 days (MPa)	28 days (MPa)	Percent increment	
(%)	(g)					1 to 7 days	7 to 28 days
0	0	Control	16.0	43.0	46.1	168.8	7.2
2	13.5	WW	15.3	31.1	32.0	103.3	2.9
4	27.0	WW	13.9	24.4	28.5	75.5	16.8
6	40.5	WW	8.6	19.6	26.3	127.9	34.2
8	54.0	WW	5.6	14.5	22.0	158.9	51.7
10	67.5	WW	1.3	7.5	11.7	476.9	56.0
2	13.5	OW	15.2	30.4	31.2	100.0	2.6
4	27.0	OW	13.4	23.3	26.4	73.9	13.3
6	40.5	OW	8.0	18.0	22.2	125.0	23.3
8	54.0	OW	5.2	15.4	18.5	196.2	20.1
10	67.5	OW	1.2	8.4	10.4	600.0	23.8

WW: Water wet OW: Oil wet

### 3.3. Hydration process

Due to the extended nature of the mixing process, the initial heat evolution (A) which is mainly due to dissolution and aluminate hydration ( $C_3A$ ) was not captured for all mixes. Also the dormancy period (B) was not clearly observed. After reaching the greatest temperature change in stage C, the hydration temperature peaks begins to decline during deceleration period (D) followed by the curing stage (E). As a result, the important properties to note from the semi-adiabatic calorimeter temperature profile are the maximum recorded temperature and the time at the maximum temperature.

The thermal profile from semi-adiabatic suggests that there is a similar trend for the water wet and oil wet plot (Figure 6). There is a threshold below (4%) which there is little impact and above which there is an impact on the hydration process. The hydration is strongly affected by the addition 10% of oil, where there is both a lagging and a decrease in the temperature peak. It was observed that the mixing method has a relatively small impact overall on the hydration process. Despite the fact that with the oil wet method the oil is coated the cement prior to contacting the water, there is little impact on the hydration reaction compared to the water wet method. Table 3 shows the summary of the hydration characteristics for all the mixes.

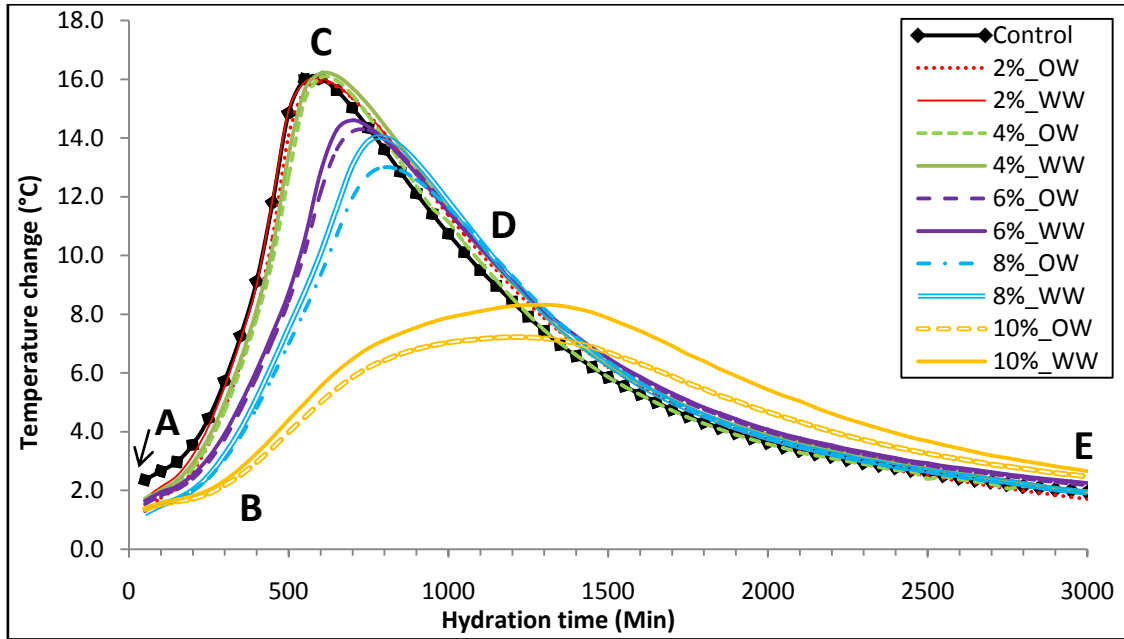


Figure 6: Temperature evolution of mortar containing mineral oil using different mixing methods (Water wet, WW and Oil wet, OW)

Table 3: Summary of hydration characteristics of mortar containing mineral oil using water wet (WW) and oil wet (OW) method for mixing

Oil content		Water wet (WW)		Oil wet (OW)	
		Peak temperature	Time at peak temperature	Peak temperature	Time at peak temperature
(%)	(g)	(°C)	(Min)	(°C)	(Min)
0	0	16.11	571	16.11	571
2	13.5	16.06	592	15.93	605
4	27.0	16.23	608	16.11	622
6	40.5	14.60	696	14.3	730
8	54.0	14.03	779	13.01	811
10	67.5	8.31	1239	7.23	1194

#### IV. CONCLUSION

Cement-based stabilisation/solidification of oil contaminated material is an emerging technology however there is limited knowledge in relation to the effect of the mixing method on the fresh, hardened properties and the hydration of the resultant cementitious mix. The results of this study show that mixing protocols have relatively little impact on the behaviour of the mortar mixes both in the fresh and hardened states. Furthermore, the study reveals that the fraction of oil in mortar played a more important role in terms of the inhibition of cement hydration than the mixing method. While oil coats the cement prior to contacting the water in the oil wet method, the two mixing methods actually exhibit the same trend. At lower oil content ( $\geq 4\%$ ) there is little impact but at higher oil content (6% - 10%), the hydration is strongly impacted. More research is required (using more complex and realistic oily materials to the petroleum industry) as this will help to pinpoint whether the fundamental knowledge generated is applicable to these materials.

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