The effect of coarse aggregate inclusion on the performance of reactive powder concrete exposed to oil products.

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Abstract. Reactive powder concrete, RPC, is an ultra-high strength and high ductility cementitious composite with advanced mechanical and physical properties. RPC is composed of very fine powders (cement, sand, quartz powder and silica fume), steel fibers (optional) and superplasticizer. A basic principle, which was presented by the first developers of RPC, is to eliminate the coarse aggregate in mixture to enhance homogeneity. The main purpose of the present investigation is to modify RPC by including natural graded coarse aggregate in the mixture in different approaches. The investigation was devoted to monitor the strength development, total absorption and permeability of RPC after exposure to two types of oil products, kerosene and gas oil, up to 180 days. The current results showed that eliminating coarse aggregate as a principle could be gone-beyond. The better way for including coarse aggregate in RPC was to replace the fine sand and the most useful FA/A ratio is expected to be within the range of 0 - 25 % by weight. This range was found to create an optimum situation between strength and absorption for kerosene and gas oil exposures. It is not recommended to include the coarse aggregate in RPC mixtures as a replacement to the binder specially when dealing with oil products storage.

1. Introduction

Reactive Powder Concrete, RPC, was first developed in France in the early 1990s and the world’s first Reactive Powder Concrete structure, the Sherbrook Bridge in Canada, was erected in July 1997. RPC is an ultra-high strength and high ductility cementitious composite with advanced mechanical and physical properties. It consists of a special concrete where the microstructure is optimized by precise gradation of all particles in the mix to yield maximum density. It uses extensively the pozzolanic properties of highly refined silica fume and optimization of the Portland cement chemistry to produce the highest strength hydrates [1].

RPC is composed of very fine powders (cement, sand, quartz powder and silica fume), steel fibers (optional) and superplasticizer. The superplasticizer, used at its optimal dosage, decreases the water to cement ratio (w/c) while improving the workability of the concrete. A very dense matrix is achieved by optimizing the granular packing of the dry fine powders. This compactness gives RPC ultra-high strength and durability. RPCs have compressive strength ranging from 200 MPa to 800 MPa [2].

The following basic principles were highly emphasized by the developers of the RPC [3]:
- Enhancement of homogeneity by elimination of coarse aggregates;
- Enhancement of compacted density by optimization of the granular mixture, and application of pressure before and during setting;
- Enhancement of the microstructure by post-set heat-treating;
- Enhancement of ductility by incorporating small-sized steel fibers;
- Maintaining mixing and casting procedures as close as possible to existing practice.

Collepardi et al. [4] stated that this prescribed mixture could not be considered as concrete due to the
absence of coarse aggregate. Moreover, the cement factor of the RPC is as high as 900-1000 kg/m$^3$ due to the use of very fine sand instead of ordinary aggregate. This unusual cement factor could increase drying shrinkage and creep strain of the RPC with respect to ordinary concrete with cement factor usually in the range of 300-500 kg/m$^3$. As a results of these claims, Collepardi et al. had modified the original RPC mixture to include some coarse aggregate and investigated the properties of the modified RPC mixtures in terms of required mixing water, compressive and flexural strength, shrinkage, swelling and creep. They concluded that the replacement of the fine ground quartz sand by an equal volume of well graded natural aggregate did not change the compressive strength of the RPC at the same water-cement ratio and that was in contradiction to Richard and Cheyrezy [3].

In the present work the same modification that proposed by Collepardi et al [4] has been adopted to study the effect of coarse aggregate inclusion on the performance of RPC when being exposed to kerosene and gas oil. In addition to that the high-reactivity Metakaolin was used as supplementary cementitious material instead of Silica fume and the butadiene styrene rubber was used as high-range water reducer. The investigation was devoted to monitor the strength development, total absorption and permeability of RPC.

2. Experimental Work

2.1 Materials

The binder in the current RPC mixes consists of 80 percent by weight of ordinary Portland cement (ASTM C150- Type I) [5] and 20 percent high reactivity Metakaolin (ASTM C618- Type N) [6]. Table 1 lists the chemical and physical properties of both materials.

| No. | Property                              | OPC  | HRM  |
|-----|---------------------------------------|------|------|
| 1   | Oxide Content, %                      |      |      |
|     | CaO                                  | 60.5 | 0.28 |
|     | SiO$_2$                               | 21.4 | 54.36|
|     | Al$_2$O$_3$                           | 5.98 | 32.76|
|     | Fe$_2$O$_3$                           | 3.9  | 1.32 |
|     | MgO                                  | 1.36 | 0.18 |
|     | SO$_3$                                | 1.82 | 0.12 |
| 2   | LOI                                   | 1.80 | 7.97 |
| 3   | IR                                    | 1.04 | ---- |
| 4   | LSF                                   | 0.92 | ---- |
| 5   | Specific surface (Blaine method), cm$^2$/gm | 3650 | 5741 |
| 6   | Soundness, % (Autoclave method)       | 0.25 | ---- |
| 7   | Setting time (vicat's apparatus):     |      |      |
|     | Initial, hrs: min.                    | 1:15 | ---- |
|     | Final, hrs: min.                      | 2:45 |      |
| 8   | Compressive strength:                 |      |      |
|     | 3days, N/mm$^2$                       | 25   | ---- |
|     | 7days, N/mm$^2$                       | 32.3 |      |
| 9   | Specific gravity                      | 3.15 | 3.11 |
| 10  | Strength activity index, %            | 100  | 164  |

Very fine sand, with maximum size of 0.6 mm, was used as fine aggregate (BS 882:1992 - fine) [7], meanwhile natural gravel, with maximum size of 14 mm, was incorporated as coarse aggregate (BS 882:1992, 5 –14 mm) [7]. Table 2 shows the sieve analysis and some characteristics of the used aggregates. Straight steel fibers, with a length of 13 mm and diameter of 0.18 mm, were used to produce RPC. These fibers have 1800 MPa tensile strength and 210 GPa modulus of elasticity.
Styrene butadiene rubber, SBR, emulsion was employed successfully as high-range water reducer to control the workability of the RPC mixes [8]. This latex is typically included in concrete in the form of a colloidal suspension polymer in water.

| No. | Test | FA | CA | BS 882:1992 Limitations [7] |
|-----|------|----|----|-----------------------------|
| 1   | Sieve Analysis | % Cum. Passing | FA | CA |
| 14  | ---  | 100 | --- | 90-100 |
| 9.5 | 100  | 85  | 100 | 50-85 |
| 4.75| 100  | 10  | 100 | 0-10 |
| 2.36| 100  | 2   | 80-100 | --- |
| 1.18| 100  | --- | 70-100 | --- |
| 0.6 | 100  | --- | 55-100 | --- |
| 0.3 | 45   | --- | 5-70  | --- |
| 0.15| 5    | --- | 0-15  | --- |
| 2   | Sp. Grav., SDD | 2.73 | 2.75 | --- |
| 3   | SO₃ Content, % | 0.08 | 0.05 | --- |
| 4   | Absorption, % | 0.70 | 0.63 | --- |

The mix proportioning procedure, which was recommended by Richards and Cheyrezy [3], was adopted. To achieve 130 MPa compressive strength at 28 days with workability of 150 ± 5 mm, flow table, many trials were done. Finally the proportions shown in Table 3 were used for the reference RPC mix, M₀.

Two approaches were adopted to investigate the effect of coarse aggregate inclusion in the mix. The first is changing the fine aggregate to total aggregate by weight ratio, FA/A. The following ratios were investigated: 100, 52, 25 and 0 percent for mixes M₀, M₁, M₂ and M₃ respectively (series I).

In the second approach the effect of variation in the aggregate to binder by weight ratio, A/B, on the behavior of RPC was studied. Only the coarse aggregate content was varied in this part of the study. The investigation included four weight ratios: 0.91, 1.11, 1.23 and 1.35 for mixes M₀, M₄, M₅ and M₆ respectively (series II). Table 3 lists the details of the all investigated mixes.

| Mix | Binder, kg/m³ | Aggregate, kg/m³ | FA/A ratio % | A/B ratio by weight | Water, kg/m³ |
|-----|---------------|-----------------|--------------|---------------------|--------------|
| M₀  | 920 230       | 1050 0          | 100          | 212                 |
| M₁  | 550 500       | 52              |              | 212                 |
| M₂  | 260 790       | 25              | 0.91         | 203                 |
| M₃  | 0 1050        | 0               | 194          |
| M₄  | 835 210       | 111 90          | 1.11         | 201                 |
| M₅  | 790 197       | 1050 87         | 1.23         | 198                 |
| M₆  | 750 185       | 215 83          | 1.35         | 203                 |

A constant volume fraction for steel fibers of 2 % was used for all mixes. The SBR content was kept constant also at 14 % by weight of cement.
2.2 Curing procedure
RPC mixes were cured in water at 90 °C for 48 hrs and after that in water at 20°C for 24 hrs and finally in air at 20°C till the age of 28 days. After the age of 28 days, tested specimens were grouped in three series. The first continued to be cured with water, meanwhile, the second cured with kerosene and the third with gas oil until the age of test. Kerosene and gas oil were used to simulate the exposure conditions in structures for storage or transportation of oil. Table 4 shows the properties of these products.

Table 4. Properties of used oil products.

| Property                          | Kerosene | Gas oil |
|----------------------------------|----------|---------|
| Moisture content, % by volume    | 0        | 0       |
| Sulfur content, % by weight      | 0.31     | 0.80    |
| pH                               | 7.6      | 6.3     |
| Specific gravity at:             |          |         |
| 25°C                             | 0.708    | 0.825   |
| 40°C                             | 0.770    | 0.813   |
| Viscosity (centipoises) at:      |          |         |
| 25°C                             | 1.092    | 3.960   |
| 40°C                             | 0.855    | 2.943   |

2.3 Testing program
Table 5 lists the conducted tests, the adopted standards, types and dimensions of tested specimens and age of test for the hardened RPC mixes.

Table 5. Details of tests, adopted standards, specimen’s types and testing age.

| Test   | Adopted standard | Specimen type | Dimensions, mm | Age of test, days |
|--------|------------------|---------------|----------------|-------------------|
| Compressive Strength | BS EN 12390-3 [9] | Cube          | 100*100*100    | 3, 7, 28, 30, 90 and 180 |
| Splitting Tensile Strength | ASTM C496 [10] | Cylinder      | d=100, h=200   | 3, 7, 28, 30, 90 and 180 |
| Absorption         | ASTM C 267 [11] | Cube          | 100*100*100    | 7, 28, 60, 90 and 180 |

The permeability test, a non-standard test, was performed by the oil research and development center/Ministry of Oil. The test was carried out on cylindrical specimens of 38 mm in diameter and 50 mm in thickness. The specimens were oven dried for one week, then cooled for 24 hrs and placed in the pressure vessel, to accelerate the saturation process. The sample was subjected to a pressure of 3.5 N/mm² for a period of one week under the effect of the oil products after which, the testing sample was inserted in the core holder. Compressed air was used to pressurize the specimens to 0.241 N/mm², equivalent of a head of 30.01 m gas oil and 31.01 m kerosene, hydrostatic pressure was maintained for 4 days.

Darcy's law [12] was used to determine the permeability coefficient from the following equation:

\[
\frac{dq}{dt} \left( \frac{1}{A} \right) = K_o \left( \frac{dh}{L} \right)
\]

Where:
- \( dq/dt \): rate of flow through the sample, cm³/sec.
- A: the cross sectional area of the specimens exposed to the fluid, cm².
- dh: thickness of the specimen, cm.
- \( K_o \): coefficient of permeability, cm/sec.
3. Results and Discussion

3.1 Strength development

Irrespective of the exposure conditions, all mixes show a strength gain with aging and that is the result of continuous hydration of the high cement factor. Fig. 1 displays the compressive strength development for all mixes which were cured in water after the age of 30 days. Mixes in series I: M1, M2 and M3, have yielded higher strength values than the reference mix M0 for the same age. For series I, the amount of fine sand was gradually replaced by natural graded coarse aggregate without changing the cement factor. The decrease in the FA/A ratio caused a reduction in the aggregate total surface area and consequently a reduction in the water requirements or in other words a decrease in the W/B ratio. Also increasing the coarse aggregate content inherited denser concrete mass and better bond. In series II, the graded coarse aggregate, had replaced part of the binder, Portland cement and HRM. Mixes M4, M5 and M6 attained lower compressive strength values than mix M0 for the same age. The probable cause for this reduction in strength is the increase in the W/B ratio or the reduction in the binder content.

![Fig. 1. Compressive strength development for RPC mixes.](image1)

![Fig. 2. Splitting tensile strength development for RPC mixes.](image2)

The effect of graded natural coarse aggregate inclusion on the splitting tensile strength development is shown in Fig. 2. The mixes in the two investigated series I and II reached lower ultimate strength values with respect to the mix M0 for the same age of test. This trend could be attributed to the effect of smooth surfaces of used aggregate on the bond strength between aggregate and mortar [13, 14] and many researchers [14, 15] had stated that tensile strength of high strength concrete is more sensitive to microcracking due to coarse aggregate inclusion than compressive strength.
In order to magnify the effect of exposure to kerosene and gas oil on the compressive strength development of RPC mixes, the value of strength for specimens soaked in oil products were compared to those attained by specimens cured in water for the same ages. Figs. 3 and 4 show the development of the ratios \( f_{c,K}/f_{c,W} \) and \( f_{c,G}/f_{c,W} \) with curing age. For mixes in series I, Figs. 3a and 4a, curing with kerosene and gas oil have positive effect on strength gain till the ages of 120 – 150 days. Afterward, the curing in water has overtook and yielded higher values. Such behavior could be attributed to the densification of concrete caused by the ingress of oil product which have lower viscosity than water [16, 17]. After the assigned period, 120 – 150 days of soaking, the oil products have a slight negative effect in decreasing strength. This trend is diagnosed in literature but higher rates were reported [18, 19]. The gas oil showed more harmful effect than kerosene and that may be due to its higher sulphate content.

Figs. 3b and 4b illustrate the development of the ratios \( f_{c,K}/f_{c,W} \) and \( f_{c,G}/f_{c,W} \) with curing age for mixes in series II. These mixes were produced with a varying A/B ratio. The degradation in strength was recorded in earlier ages than mixes in series I, as early as 60 days. Lowering the binder content could be the cause of such a behavior. Firstly, the hydration process will be reduced in rate and value. Secondly, reducing the content of finely divided binder, cement and HRM, would decrease the pore refinement physical effect in the microstructure of RPC [20].

![Fig. 3a. Comparison of compressive strength development between kerosene and water curing for series I mixes.](image)

![Fig. 3b. Comparison of compressive strength development between gas oil and water curing for series II mixes.](image)
3.2 Total absorption and permeability

Figs. 5a, b and c show the results of total absorption for tested RPC mixes in water, kerosene and gas oil respectively for the assigned ages. Absorption values depend mainly on the viscosity of the penetrating fluid and the porosity of the absorbing media. The values of absorption were the highest in water and the lowest in gas oil. This descendant order is due to the variation in viscosity of these fluids. For all exposure conditions, there was a deceleration in the rate of absorption. The rate was higher for the period 7 - 90 days than that of the period 90 – 180 days. This trend could be resulted from the continuous hydration of the binder.

Figs. 6a, b and c display the relationship between the FA/A ratio and total absorption for the studied mixes, series I, when they are cured in water, kerosene and gas oil respectively. For the investigated ratios, 52, 25 and 0 %, mix 2, with the FA/A = 25%, has the lowest absorption values when the curing liquid is water or kerosene. For gas oil, mix 3, with no fine sand, showed the lowest absorption values. This behavior could be related to the viscosity of curing liquid. Based on these observations, it could be concluded that replacing fine sand with natural graded coarse aggregate could be most useful with FA/A = 25% although the strength was not the highest, mix 2. Absorption is a crucial property when dealing with oil products storage.

Furthermore, Figs. 7a, b and c show the relationship between the A/B ratio and total absorption for the studied mixes, series II, when they are cured in water, kerosene and gas oil respectively. The studied ratios were 1.11, 1.25 and 1.35 by weight of binder. This approach leads to the increase in total
absorption for all mixes and for all ages. Therefore, it is not recommended to make the inclusion of coarse aggregate as a replacement of the binder.

![Fig. 5a. Total absorption of water-cured RPC mixes.](image)

![Fig. 5b. Total absorption of kerosene-cured RPC mixes.](image)

![Fig. 5c. Total absorption of gas oil-cured RPC mixes.](image)
Fig. 6a. Relationship between absorption and FA/A for water-cured RPC mixes.

Fig. 6b. Relationship between absorption and FA/A for kerosene-cured RPC mixes.

Fig. 6c. Relationship between absorption and FA/A for gas oil-cured RPC mixes.
Fig. 7a. Relationship between absorption and A/B for water-cured RPC mixes.

Fig. 7b. Relationship between absorption and A/B for kerosene-cured RPC mixes.

Fig. 7c. Relationship between absorption and A/B for gas oil-cured RPC mixes.

The results of the conducted permeability test for the RPC at ages of 60 and 90 days, cured in water or exposed to oil products, have no conclusive indication. All mixes have zero value for the coefficient of permeability. These values could not be considered as an indication for the impermeability of RPC mixes to water or oil products. In spite of the dense microstructure and low porosity of these mixes due to heat treatment and their high ability to be pressurized, but it seems that this test is not sensitive to the variation in microstructure between tested mixes.
4. Conclusions
The following findings are based on the results of the current work:

a. The basic principle of eliminating coarse aggregate in RPC to minimize the heterogeneity of the mixture could be gone-beyond.

b. The approach of replacing fine sand partially or totally by natural graded coarse aggregate without changing the binder content could be adopted. The most useful FA/A weight ratio is expected to be within the range of 0 - 25 %. This range was found to create an optimum situation between strength and absorption for kerosene and gas oil exposures.

c. It is not recommended to include the coarse aggregate in RPC mixtures as a replacement to the binder specially when dealing with oil products storage.

Abbreviations
A/B Aggregate to binder ratio by weight.
ASTM American society for testing and materials.
BS British standards.
CA Coarse aggregate.
FA/A Fine aggregate to total aggregate ratio by weight.
$f_{CG}$ Compressive strength of gas oil-cured concrete, MPa.
$f_{eK}$ Compressive strength of kerosene-cured concrete, MPa.
$f_{CW}$ Compressive strength of water-cured concrete, MPa.
HRM High reactivity Metakaolin.
RPC Reactive powder concrete
W/B Water to binder ratio by weight.

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