Experimental Evaluation of Modified Sulfur Concrete for Achieving Sustainability in Industry Applications

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Received: 10 November 2018; Accepted: 11 December 2018; Published: 23 December 2018

Abstract: Portland cement concrete (PCC) has been the most widely used concrete in the construction industry. However, PCC has a short service life under some aggressive environments, leading to the need of costly repairs. The purpose of this research was to implement local materials to produce a modified sulfur concrete (MSC) with better performance in industrial applications. Several modified sulfur concrete mixtures were prepared using natural aggregates from the northern region of Colombia, and sulfur cement by combination of sulfur with a modifier, with the objective of achieving the best performance based on mechanical strength and chemical resistance. To achieve this purpose, an experimental program based on a k-factorial design was used to determine the optimal mix design based on the results of the compressive strength. The mixture presenting the best results was then examined further with standardized tests to determine its physical, mechanical, and chemical properties (compressive strength, abrasion resistance, bulk density, absorption, and chemical resistance). Final results showed that the sulfur concrete mixture is very resistant to chemical attack and an outstanding substitute for PCC. The results indicated that there is no significant loss in weight and no relevant variation in compressive strength after the specimens were immersed in sulfuric acid and sulfate solutions. In addition, similar results were obtained for the slabs located in chemicals plants whose conditions were assessed during a 60-day period of exposure.

Keywords: modified sulfur concrete; sulfur cement; compressive strength; abrasion resistance; chemical attack

1. Introduction

Sulfur and its derivatives are considered as being among the most important elements used as industrial raw materials all over the world [1]. The main use of sulfur includes agricultural industry as fertilizer and other chemical processes, but it also has potential applications in the manufacturing industry (pharmaceuticals, personal care products, cosmetics, water treatment, etc.). Sulfur is found naturally in the environment and has been ranked between the tenth to fourteenth most abundant elements in the Earth’s crust [1–3]. Despite that, during the last century, as consequence of technological progress and industrial developments, sulfur production from petroleum and gas resources has generated large accumulation of this mineral [4]. Since 2011, the global production of sulfur in all forms has been around 69 million tons [5]. China, for instance, produced 8.8 million tons in 2015, while Colombia produced 69,000 tons during the same period [6]. As fossil fuel consumption is increasing all over the world, petroleum and gas production are yielding sulfur as a by-product, which is being applied as a binder in composite construction materials such as asphalt and concrete [7]. In fact, sulfur cements have been recognized for providing good resistance to chemical attack, quick
hardening, i.e., reaching the required properties in only 24 h, high strength and fatigue resistance, very low water permeability, and exceptional resistance to acid and salt agents, which allows its use in highly aggressive environments [4,7,8]. In addition, the mechanical properties of sulfur cements may be improved by the inclusion of a variety of admixtures to produce what is known as modified sulfur cements.

On the other hand, modified sulfur concrete (MSC) is a thermoplastic material composed of aggregates, sulfur cement, and additives, which, upon solidifying, rapidly gains resistance in about one day of curing; consequently, the early strength gain is one of the most important and desired properties of MSC as a construction material [9,10]. Sulfur is usually heated and mixed with additives and aggregates to produce MSC. During the cooling process, the sulfur changes from a liquid state to monoclinic sulfur crystals (S\(\beta\)-phase) at \(-114\) °C; then, when the temperature reaches approximately 96 °C, the S\(\beta\)-phase transforms to orthorhombic sulfur (S\(\alpha\)-phase), which is the stable phase of sulfur at room temperature [4,7,11]. As this transformation occurs in less than 24 h and S\(\alpha\)-phase has greater density than the S\(\beta\)-phase, inducing high internal stresses in the material due to the solidification of sulfur, this allows for premature failure, cracks, or defects at early ages. As the total volume contraction upon solidification is about 7%, it is necessary to modify the sulfur to ensure the durability of elements constructed with MSC [4,11].

Alternately, if unmodified sulfur (elemental) is used as a binder, concrete may present some problems regarding durability and stability; in particular, sulfur concrete degradation and failures have been reported after exposure to repeated freezing cycles. This phenomenon occurs because sulfur transformation occurs during concrete preparation, which induces high internal stresses and micro cracking within the material [8]. From a mechanical point of view, elemental sulfur provides lower resistance to water and higher brittleness than PCC [12–15]. To overcome these problems, and seeking to enhance mechanical properties and long-term performance, sulfur concrete needs to be modified. However, MSC may still present some disadvantages. For instance, the thermal expansion of MSC specimens is substantially larger than has been reported for PCC. Indeed, this high thermal expansion coefficient may imply significant expansion and contraction of concrete due to temperature changes eventually promoting micro-cracks in the concrete [7]. The most common modifiers used in order to avoid the transformation of sulfur from monoclinic to orthorhombic states are dicyclopentadiene, or a combination of (a) dicyclopentadiene, cyclopentadiene, and dipentene, and (b) olefinic polysulfide additives [12,15]. However, the limited use of MSC in industry applications has been ascribed to the fact that the reaction between sulfur and dicyclopentadiene is exothermic and requires close temperature control; in addition, dicyclopentadiene-modified sulfur cement is unstable when exposed to high temperature conditions [12].

One of the main advantages of MSC over PCC is its durability to most acid and salt environments, especially in industrial plants where conventional PCC has a short service life. Besides industry applications, other uses of MSC include structures under freezing and thawing cycles, food industry facilities, sewage pipes, drainage channels, and marine structures. Regarding sustainability, MSC may be considered an eco-friendly material as it can replace Portland cement in several construction applications. In fact, among several causes of global warming, the construction industry is responsible for a significant portion of greenhouse gases emissions (GHG) [7]. In fact, PCC is responsible of about 5% of global CO\(_2\) generation, which is usually linked to the heating process of raw materials in kilns at temperatures higher than 1400 °C [10]. On the other hand, since a large amount of sulfur used in the industry is obtained from the distillation of oil as a by-product, using the sulfur as binder in concrete mixtures will reduce the use of water and environmental impacts related with Portland cement production. Finally, in order to quantify the potential environmental benefits of MSC, further analysis must consider life cycle assessment comparing production and use phases of PCC and MSC respectively.

MSC has been massively implemented in United States, Canada, and recently in Europe, but not in South America [4], so the developments of this material in Colombia have been limited. This paper
aims to propose an optimal mix design of MSC by implementing a factorial experiment in order to study the effects each design factor on the response variable (compressive strength). Based on the optimal mix formulation, a complete characterization of MSC was performed, including compressive strength, density, abrasion resistance, chemical attack, and durability.

2. Materials and Methods

Elemental sulfur with 99.9% purity, a specific weight of 1.032 g/cm³, and other components as shown in Table 1 were used as a binder. The sulfur was modified using an additive with a specific weight of 1.69 g/cm³; this additive is made of olefinic hydrocarbon polymers such as Excopol to produce modified sulfur cement that allows the sulfur to stabilize in an orthorhombic form (Sα phase). The aggregates were sand and siliceous river gravel as fine and coarse aggregates respectively, and were obtained from local stone quarries with maximum particle size of $\frac{3}{4}$". Several test methods were implemented in order to examine the physical, chemical, and mechanical properties of the fine and coarse aggregate sources, including bulk specific gravity, water absorption, sieve analysis, abrasion, and chemical resistance, which are in accordance with specifications established on the ASTM C33/33M standard [16]. Results are shown in Table 2.

| Table 1. Chemical Analysis of Sulfur. |
|---------------------------------------|
| S (%) | 99.9  |
| Ash (%) | 0.0004 |
| H₂O (%) | 2.4  |
| H₂SO₄ (%) | 0.007 |
| Apparent density g/cc | 1.032 |
| Organic Content | 0.002 |
### Table 2. Aggregates Characterization Summary.

| Test                                      | Standard                  | Units | Fine Aggregate Type A | Fine Aggregate Type B | Coarse Aggregate | Acceptance Limits |
|-------------------------------------------|---------------------------|-------|-----------------------|-----------------------|------------------|-------------------|
| **Physical properties**                   |                           |       |                       |                       |                  |                   |
| Sieve analysis                            | ASTM C136 [17]            | –     | –                     | –                     | –                | –                 |
| Water Absorption                          | ASTM C 127, C128 [18,19] | %     | 2.78                  | 1.64                  | 1.64             | <2%               |
| Bulk density by rodding                   | ASTM C29/C29M [20]        | kg/m³ | 1660                  | 1780                  | 1570             | –                 |
| Voids (compacted by rodding)              |                           | %     | 35                    | 28                    | 37.9             | –                 |
| Loose bulk density                        |                           | kg/m³ | 1370                  | 1600                  | 1420             | –                 |
| Voids (loose aggregate)                   |                           | %     | 46.3                  | 35.2                  | 43.9             | –                 |
| Specific gravity                          | ASTM C 127, C128 [18,19] |        | 2.62                  | 2.52                  | 2.57             | –                 |
| SSD                                       |                           |        | 2.55                  | 2.47                  | 2.53             | –                 |
| Dry                                       |                           |        | 2.74                  | 2.60                  | 2.64             | –                 |
| Clay lumps and friable particle           | ASTM C142 [21]            |        |                       |                       |                  | <2%               |
| **Mechanical Properties**                 |                           |       |                       |                       |                  |                   |
| Los Angeles Abrasion, LA                  | ASTM C535 [22]            | %     | –                     | –                     | 13.18            | <40%              |
| **Chemical Properties**                   |                           |       |                       |                       |                  |                   |
| Corrosion Resistance                       | ASTM C1370                | %     | 1.8                   | 1.9                   | 1.8              | <2%               |
| $\text{H}_2\text{SO}_4$                   | [23]                      |        | 1.7                   | 1.7                   | 0.5              | <2%               |
| $\text{SO}_4(\text{NH}_4)_2$              |                           |        |                       |                       |                  |                   |
Based on ACI 548-2R standard [24], in order to reduce sulfur cement content on the mixture design, well graded aggregates should be used. In this research, three aggregate sources were used: fine aggregate type A (17.6%), fine aggregate type B (21.4%) and a coarse aggregate (61%). The selected aggregate gradation was based on reducing voids in the mineral aggregates and considering dense-graded aggregate gradation limits given by ASTM D 3515 standard [25]. Sieve analysis were conducted based on the ASTM C136 standard [17] for all aggregate sources and results are shown in Figure 1. The selected gradation corresponds to a combination of three aggregate sources to obtain a dense gradation meeting the specified limits.

A chemical resistance test for the three aggregate sources was performed based on the ASTM C1370 standard [23], which aimed to establish the acceptability of the aggregates for use in MSC. The aggregates were exposed to sulfuric acid (H$_2$SO$_4$) and ammonium sulfate SO$_4$ (NH$_4$)$_2$ solutions at a concentration of 40% in order to simulate industrial environment conditions. Weight loss of below 2% was obtained for all samples after being immersed for 24 h in the test solutions at an elevated temperature (60 ± 2 °C), as shown in Table 2.

2.1. Sample Preparation

Sample specimens were prepared according to the ACI 548.2R guide [24]. The aggregates were preheated to 135° C in a mixer with temperature control, then the sulfur and sulfur modifier additive were added and the mixture was mechanically homogenized for approximately 20 min. The temperature in the mixer was always controlled to be in the range of 130 to 140 °C and the mixing energy was 28 rpm. Once this temperature was reached, the MSC mixture was ready and placed in the molds for casting. For specimen preparation, cylindrical steel molds 15 cm in diameter and 30 cm in height were used and preheated at 120 °C before the mixture was poured. Then, the specimens were manually compacted in three layers using a steel rod following the ASTM C-39 standard [26]. The specimens were allowed to cool to room temperature before being removed from the molds, and after 24 h, demolded, and subsequently, the specimen weight and compressive strength were determined, as shown in Figure 2.
were used for conducting a 3^k factorial experiment with 2 factors, to model the influence of mixture components on the compressive strength of the MSC in order to optimize the mixture design. These two factors were aggregate content and additive/sulfur ratio.

During the selection of levels for the analysis, i.e., maximum, intermediate, and minimum for the two variables, two conditions were implemented: condition 1 varying the aggregate content as 50, 60%, 70%, and 80% without changing sulfur cement proportions (% additive/sulfur = 0.05), and condition 2 varying the sulfur cement proportions with % additive/sulfur of 0.05, 0.10, 0.15 and 0.20. In both cases, two specimens were tested to determine the one-day compressive strength according to the ASTM C39 standard [26]. The range of sulfur cement and aggregate content in which the MSC mixtures performed satisfactorily was identified considering (i) good workability, (ii) high compressive strength, and (iii) sample finishing. Tables 3 and 4 below show the results for average compressive strength for condition 1 and 2 respectively.

### Table 3. Compressive strength results—Condition 1.

| Aggregate Content (% Volume) | Sulfur Cement (% Volume) | Average Compressive Strength (MPa) | Standard Deviation (MPa) |
|-----------------------------|--------------------------|-----------------------------------|-------------------------|
| 80%                         | 20%                      | 14.48                             | 0.31                    |
| 70%                         | 30%                      | 27.07                             | 0.21                    |
| 60%                         | 40%                      | 24.28                             | 1.09                    |
| 50%                         | 50%                      | 12.12                             | 1.79                    |

### Table 4. Compressive strength results—Condition 2.

| Additive/Sulfur Ratio | Average Compressive Strength (MPa) | Standard Deviation (MPa) |
|----------------------|-----------------------------------|-------------------------|
| 0.05                 | 29.23                             | 2.86                    |
| 0.10                 | 33.14                             | 0.78                    |
| 0.15                 | 12.85                             | 0.28                    |
| 0.20                 | 10.87                             | 0.16                    |

As shown in Table 3, an aggregate content of 70% in the MSC showed the highest compressive strength; conversely, increasing the aggregate content up to 80% reduced the compressive strength, and the workability and handling experienced during placement were also significantly affected. Therefore, the range of this variable for the factorial design was selected as 60% to 70%, with 65% as an intermediate value. Also, as shown in Table 4, the additive/sulfur ratio showing the highest
compressive strength was between 0.05 and 0.10; therefore, the range of values to be used in the factorial design were selected as 0.05, 0.10, and 0.075 as the minimum, maximum, and intermediate value respectively.

2.3. Factorial Experiment

In order to optimize the mix design of MSC, a $3^k$ factorial experiment with 2 factors was implemented. This statistical approach was used to evaluate the interaction between different treatment factors with the compressive strength as the experimental response. In this case, the two factors were aggregates content (A) and additive/sulfur ratio (B). Therefore, a $3^2$ factorial experiment was developed and each factor contained a high, low, and middle level, as shown in Table 5. The design matrix for the experiment including the nine mixture proportions and the response data obtained from four replicates tested at 3 days are shown in Tables 6 and 7. Specimen dimensions of 15 cm in diameter and 30 cm in height were used in all cases.

### Table 5. Factor Levels for factorial experiment.

| Factor                | Level |
|-----------------------|-------|
| Aggregate content     | Low   | Middle | High |
|                       | 60    | 65     | 70   |
| Additive/sulfur ratio | 0.05  | 0.075  | 0.010 |

### Table 6. Mixture proportions for the factorial experiment.

| Additive/Sulfur Ratio | Aggregate Content (%vol) | Sulfur Cement (%vol) | Sulfur (kg) | Additive (kg) | Coarse Aggregate (kg) | Fine Aggregate A (kg) | Fine Aggregate B (kg) |
|-----------------------|--------------------------|----------------------|-------------|---------------|----------------------|----------------------|----------------------|
| 0.050                 | 60%                      | 40%                  | 6.22        | 0.52          | 15.46                | 4.63                 | 5.40                 |
| 0.050                 | 65%                      | 35%                  | 5.44        | 0.45          | 16.75                | 5.02                 | 5.85                 |
| 0.050                 | 70%                      | 30%                  | 4.66        | 0.39          | 18.04                | 5.40                 | 6.30                 |
| 0.075                 | 60%                      | 40%                  | 6.07        | 0.75          | 15.46                | 4.63                 | 5.40                 |
| 0.075                 | 65%                      | 35%                  | 5.31        | 0.66          | 16.75                | 5.02                 | 5.85                 |
| 0.075                 | 70%                      | 30%                  | 4.55        | 0.57          | 18.04                | 5.40                 | 6.30                 |
| 0.100                 | 60%                      | 40%                  | 5.93        | 0.98          | 15.46                | 4.63                 | 5.40                 |
| 0.100                 | 65%                      | 35%                  | 5.19        | 0.86          | 16.75                | 5.02                 | 5.85                 |
| 0.100                 | 70%                      | 30%                  | 4.45        | 0.74          | 18.04                | 5.40                 | 6.30                 |

### Table 7. Compressive strength test results for the factorial experiment (MPa).

| Additive/Sulfur Ratio | Additive/Sulfur | | | | | | |
|-----------------------|----------------|---|---|---|---|---|---|
| 60                    | 25.06          | 23.05 | 27.54 | 32.56 | 24.26 | 27.07 |
| 0.050                 | 39.86          | 30.85 | 21.48 | 19.15 | 31.61 | 30.36 |
| 0.075                 | 36.11          | 30.36 | 23.57 | 27.52 | 29.84 | 33.83 |
| 0.100                 | 46.28          | 41.92 | 24.3 | 26.89 | 32.91 | 32.98 |
| 0.050                 | 35.80          | 32.55 | 31.96 | 29.55 | 34.83 | 36.82 |
| 0.075                 | 32.68          | 35.30 | 31.71 | 29.71 | 33.69 | 36.08 |
| 0.100                 | 32.68          | 35.30 | 31.71 | 29.71 | 33.69 | 36.08 |

Analysis of the data is presented as a normal probability plot of the residuals, as shown in Figure 3a. The data in this plot lies reasonably close to the straight line, and only three residuals are showing a value higher than 2, lending support to our hypothesis that the underlying assumptions of the analysis are satisfied. In order to better interpret the model and use the results to optimize the mix design of MSC, a graphic representation of the relationship between compressive strength and the two factors, aggregate content and additive/sulfur ratio, is shown in Figure 3b. This figure shows the
contour lines of constant response (compressive strength) for the aggregate content and additive-sulfur ratio, indicating that the compressive strength is maximized when aggregate content is close to 60% (lower level) and the additive-sulfur ratio is at the intermediate level. Given a design compressive strength of 35 MPa at 3 days, the selected optimum mix design consisted of 70% aggregate in volume and 30% sulfur cement, where the additive/sulfur ratio was equal to 0.10, reducing significantly the cement content in the MSC mixture. However, from Figure 3b, different alternatives can be selected for the required compressive strength, but optimization of the mix design followed the most economical mixture with a higher aggregate content in order to reduce sulfur cement content.

![Normal Plot of Residuals](image1)

![Contour Plot for the model](image2)

**Figure 3.** (a) Normal Plot (Design Expert Software); (b) Contour Plot for the model (Design Expert Software).

### 3. Results and Discussion

#### 3.1. Compressive Strength

MSC develops about 70% of the ultimate compressive strength at only few hours after casting, and usually 75 to 85% after 24 h [27]. In this study, tests were performed on duplicate identical specimens cured at ages 3, 7, 14, and 28 days. Average compressive strength and its evolution as time progressed for all specimens are shown in Table 8. As seen on Table 8, the average compressive strength obtained at 3 days was 33 MPa, and 41 MPa at 28 days. Results agree with the percentage of the compressive strength obtained at early ages calculated as 80.5% compared to the 28 days value as reported by others [28–30].

| Time (Days) | Average Compressive Strength (MPa) | Standard Deviation (MPa) |
|-------------|-----------------------------------|-------------------------|
| 3           | 33.14                             | 0.15                    |
| 7           | 34.8                              | 0.65                    |
| 14          | 34.35                             | 2.16                    |
| 28          | 41.26                             | 0.96                    |

#### 3.2. Density

The unit weight of the MSC varies depending on the quantity and the relative density of the aggregate, the amount of air entrapped, and the amount of sulfur cement paste in the mixture. This test was performed based on the ASTM C 642 standard [31]. Average values are shown in Table 9 for MSC specimens. As observed in Table 9, the unit weight values for the MSC specimens were, on average,
between 2513–2594 kg/m$^3$. For conventional PCC, the unit weight ranges from 2240–2400 kg/m$^3$, making the MSC denser than conventional concrete as reported by others [11,29,30].

| Time (Days) | Average Density (kg/m$^3$) | Coefficient of Variation (CV) |
|------------|----------------------------|-----------------------------|
| 3          | 2580                       | 4%                          |
| 7          | 2594                       | 4%                          |
| 14         | 2529                       | 2%                          |
| 28         | 2513                       | 2%                          |

### 3.3. Abrasion Resistance

Extensive literature reports have focused on the investigation of the abrasion resistance of PCC but few data is available for MSC. The abrasion resistance of MSC is one of the most relevant advantages of this material and it has been reported as twice that of the PCC [32]. Given that one of the major applications of MSC are industrial floors and pavements, this procedure aims to simulate the abrasive service loads under traffic, impact by equipment and other abrasive actions or chemicals on concrete surfaces by the sandblasting method using a sand that meets the type and size distribution given in ASTM C 778 standard [33]. The abrasion test on MSC specimens was performed according to the ASTM C 418 Standard [34]. For the test, the specimens were first immersed in water for 24 h, then the abrasion resistance of the test specimens was determined based on the volume loss of concrete from a sandblasted area expressed in cubic centimeters per square centimeter, known as the abrasion coefficient. The mass or volume loss on the concrete surface of the specimen after the test gives an indication about its resistance to abrasion (See Figure 4). An example of the results of the abrasion test are shown in Table 10, with an average abrasion coefficient of 0.22 cm$^3$/cm$^2$.

![Appearance of MSC specimen surface after abrasion test.](image)

**Table 9.** Density for MSC specimens.

| Time (Days) | Average Density (kg/m$^3$) | Coefficient of Variation (CV) |
|------------|----------------------------|-----------------------------|
| 3          | 2580                       | 4%                          |
| 7          | 2594                       | 4%                          |
| 14         | 2529                       | 2%                          |
| 28         | 2513                       | 2%                          |

**Table 10.** Example of abrasion test results for MSC specimens.

| Cavity | Volume (cm$^3$) | Affected Surface Area (cm$^2$) | Abrasion Coefficient (cm$^3$/cm$^2$) |
|--------|-----------------|--------------------------------|--------------------------------------|
| 1      | 14.87           | 44.34                          | 0.34                                 |
| 2      | 9.91            | 52.26                          | 0.19                                 |
| 3      | 9.91            | 46.91                          | 0.21                                 |
| 4      | 14.87           | 56.19                          | 0.26                                 |
| 5      | 9.91            | 42.33                          | 0.23                                 |
| 6      | 14.87           | 55.62                          | 0.27                                 |
| 7      | 9.91            | 109.49                         | 0.09                                 |
| 8      | 14.87           | 77.25                          | 0.19                                 |

**Average abrasion coefficient (cm$^3$/cm$^2$)**: 0.22
3.4. Mass loss and Durability of MSC

The resulting durability of MSC samples was assessed by measuring the mass of the specimen at 3, 7, 14, and 28 days and comparing it with the initial value (before the immersion), as shown in Figure 5. The mass loss was calculated for MSC cylinders immersed in a sulfuric acid and ammonium sulfate solution of 40% \( \text{H}_2\text{SO}_4 \) and \( \text{SO}_4(\text{NH}_4)_2 \) respectively following the ASTM C 579 standard [35], and the results are shown in Table 11. Reported values of change of mass due to immersion were low, indicating that the samples, after being exposed to acid and sulfate environments, did not show any deterioration, as reported by others [7,36].

![Figure 5](image-url)

**Figure 5.** Change of mass due to immersion in chemical solutions.

In order to further evaluate the effect of the acid- and sulfate-containing environments on the durability of MSC, two approaches were used: evaluation of the compressive strength test results after immersion in two solutions as time progressed, and in situ performance test of MSC slabs subjected to real industrial environment in a petrochemical plant located in the city of Barranquilla.
The resulting deterioration of MSC was assessed by comparing the compressive strength results at different immersion times in the solutions evaluated in this study, as shown in Figure 6, and the compressive strength of control specimens immersed in regular water. The compressive strength loss was calculated, and results are shown in Table 11. The compressive strength results seem to be slightly affected by the immersion in the aggressive media, and the control specimens are showing similar behavior to the other samples as the immersion time increases, demonstrating the high chemical resistance of the MSC. The average mass values after immersion in solution were a little higher in both cases, but remained within expected values. Additionally, no cracks or deterioration were observed after specimens were subjected to the extreme exposure conditions of the test. Regarding the compressive strength results as shown in Table 11, average values decreased by 3–6% in the 40% H$_2$SO$_4$ solution and 0.4–1.3% in the 40% SO$_4$(NH$_4$)$_2$, similar to the results reported by others [4,7,8].

![Compressive strength of MSC under different exposure conditions.](image)

Prism-shaped samples with dimensions 40 × 40 × 7 cm$^3$, as shown in Figure 7a, were prepared and located in an industrial environment where such chemical attacks usually take place (See Figure 7b). The specimens were monitored every 15 days for a month and then after 2 months of exposure in each test environment. Visual examination and qualitative description of the evolution of physical parameters such as color change, voids, or defects and appearance, were recorded at 1, 15, 30, and 60 days of exposure. In Figure 8, the surface condition of the test specimens after exposure to sulfuric acid and ammonium sulfate in chemical plants are shown as an indication of durability of MSC as time progresses.
Table 11. Durability test results by immersion of specimens in chemical solutions: Mass loss and compressive strength loss after immersion.

| Time (Days) | Solution        | Weight before Immersion (kg) | Weight after Immersion (kg) | Mass Loss (%) | Compressive Strength Control (Mpa) | Compressive Strength Immersion in Solution (Mpa) | Compressive Strength Loss (%) |
|------------|-----------------|------------------------------|----------------------------|---------------|-----------------------------------|---------------------------------|-------------------------------|
| 3          | 40% H₂SO₄       | 13,515                       | 13,518                     | 0.02%         | 31.155                            | 33.135                          | −5.97%                        |
| 7          |                 | 13,305                       | 13,355                     | 0.38%         | 33.71                             | 34.8                            | −3.01%                        |
| 14         |                 | 13,280                       | 13,405                     | 0.94%         | 36.75                             | 34.345                          | 6.91%                         |
| 28         |                 | 13,530                       | 13,555                     | 0.19%         | 42.305                            | 41.26                           | 2.58%                         |
| 3          | 40% SO₄(NH₄)₂   | 13,260                       | 13,288                     | 0.21%         | 33.14                             | 33.00                           | −0.41%                        |
| 7          |                 | 13,580                       | 13,665                     | 0.63%         | 34.80                             | 34.38                           | −1.27%                        |
| 14         |                 | 13,505                       | 13,655                     | 1.10%         | 34.35                             | 34.58                           | 1.09%                         |
| 28         |                 | 13,745                       | 13,855                     | 0.80%         | 41.26                             | 43.11                           | 4.50%                         |
Figure 7. (a) Specimen configuration for in-situ durability test; (b) Specimen at test location.

Figure 8. (a) Specimen at test location –SO$_4$ (NH$_4$)$_2$; (b) Specimen at test location –H$_2$SO$_4$.

4. Conclusions

The following conclusions can be made based on the experimental research results:

The results indicate that after 60 days of exposure, the appearance of the slabs was slightly affected, revealing the exceptional durability of the MSC (See Figure 8a,b). Under these exposure conditions, a certain change in surface coloring was noted, although it was washed out easily. However, the slabs located at the ammonium sulfate plant (Figure 8b) revealed small defects as small cracks and local
dissolution of the sulfur cement paste in areas where the surface presented initial defects such as pores and voids, which may be from poor compaction; however, this was limited and insignificant compared to the total area exposed. Additionally, given that the chemical concentrations at site locations are expected to be close to 100% and at higher temperatures than those of the laboratory specimens, the aggressiveness for these slabs was higher than for the laboratory test. Furthermore, under site conditions, the test solutions are changing with time; thus, more severe conditions are expected as a new solution is in direct contact with the specimen over time. Further confirmation of trends will be useful for future analyses and long-term performance evaluation of MSC structures in the case of in situ exposure, as such information is limited.

4. Conclusions

The following conclusions can be made based on the experimental research results:

- The optimal mix design of modified sulfur concrete consisted of an aggregate content of 70% of the total volume, with 61% of coarse aggregate, 17.6% of fine aggregate A, and 21.4% of fine aggregate B, and with an additive/sulfur ratio of 0.100.
- Statistical analysis of the mix design showed a threshold value for the additive/sulfur ratio and aggregate content at which the compressive strength and the workability of the MSC mixture reaches an optimum balance. Based on the factorial design, the threshold value is about 30% of modified sulfur cement, with an additive/sulfur ratio of 0.05.
- On the basis of the results obtained from the factorial experiment, different mixture designs of MSC can be formulated depending on the desired compressive strength and to minimize cost. Results can be used to project optimal mix design for similar applications.
- Modified sulfur concrete reaches approximately 73% of the ultimate strength at one day of casting. This resistance continues to increase with time but at a lower rate. No significant change in the unit weight or compressive strength was observed for the specimens after the durability test in two aggressive media, demonstrating the high chemical resistance of the MSC. Compressive strength results decreased by 3–6% in the 40% H$_2$SO$_4$ solution and 0.4–1.3% in the 40% SO$_4$(NH$_4$)$_2$ similar to results reported by others.
- In situ performance of MSC was assessed by exposure of prism-shaped samples to two aggressive media located in chemical plants. Results indicate good performance of MSC elements up to 60-days. Long-term performance is pending.

Author Contributions: The authors confirm contribution to the paper as follows: study concept and design: M.D., J.T.; data collection: M.D.; analysis and interpretation of results: M.D., J.T., G.M.-A.; draft manuscript preparation: M.D., G.M.-A. All authors reviewed the results and approved the final version of the manuscript.

Funding: This research was funded by Departamento Administrativo de Ciencia, Tecnología e Innovación, grant number 745/2016.

Acknowledgments: The authors are grateful for the cooperation of the Company Monomeros Colombo Venezolanos S.A in providing the sulfur, additives and chemical solutions.

Conflicts of Interest: The authors declare no conflict of interest.

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