Characterizing the Mechanical Properties of Sustainable Modified Cementitious Grout for Semi-Flexible Mixture

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Abstract: Grout could be a cement-based fabric with high quality and workability that permitting it to be infused into little splits or cracked zones. It is ordinarily utilized to fix concrete splits or gaps, but it may be utilized to fill voids beneath metal bases or dig anchors or soil gravities filling. Moreover, grout is utilized effectively within the generation preparation of the semi-flexible asphalt blends. This research aims to characterize the impact of incorporating proposed ash as a supplementary cement material (SCM) on the mechanical properties of developed grout. The developed grout comprised Ordinary Portland Cement (OPC), silica fume (SF), palm frond waste ash (PFWA), superplasticizer (SP), and water. Diverse extents of specified materials were utilized to characterizing the created grout through flowability, compressive strength, and flexural strength tests. Results disclosed that the stream time diminishes with an increased water/binder ratio (W/B). On the other hand, the compressive strength and flexural strength of the grouts comprised OPC+PFWA reveals an ideal dose that shows superior mechanical properties, namely, 10% PFWA. As the main conclusion, the mechanical properties of sustainable modified cementitious grout are highly affected by grout ingredient types, dosage, and properties.

Keywords: Cementitious grout materials; flowability; palm frond waste ash; semi-flexible pavement; silica fume.

1. Introduction
Cementitious grouts have been used commonly by engineers to solve foundation problem as infilling voids and water tightness of underground soil mass. Cement grouts have been extended to use as sealing hazardous and low-level radioactive wastes within environmental areas. Literature indicates that the grout can perform its intended role as stabilizing burial waste trenches and solidifying agents [1,2]. The grout injecting in the soil is back to the year 1802 when the French civil engineer “Charles Berigny” noted that the soil exposed to erosion due to water is affecting the bearing capacity and all structure above; he concluded that injecting soil by grout may decrease soil erosion [3]. Grout can also improves fire resistance, blast resistance, termite resistance, and thermal storage capacity [4]. Grout can be used in semi-flexible pavement (SFP) to filling the voids at the porous asphalt layer [5]. SFP is a pavement technology that consists of open-graded asphalt concrete with high air void (25-35)% that injects with a special grouting material with the fluidity of (11-16) seconds [6-8]. SFP could be used as a surface layer with a depth of (30-60) mm, while some work made it up to 80 mm thickness [9]. On the other hand, some grout can be used with 200 mm thickness [10]. For this depth, grout should fill the voids in the porous asphalt layer, for that fluidity of the grout that needed (11-16) second. Grout using in SFP should have sufficient resistance to stress and strain without failure. Also, it should have good fluidity to filling...
the voids in the porous asphalt layer. Table 1 shows that the fluidity is changed significantly with grout material ingredients and water content.

### Table 1. Various design properties of grout from literature.

| Ref. | Proportions of the materials used in designing the grout | Fluidity (s) |
|------|---------------------------------------------------------|--------------|
| [11] | 95% OPC + 5% SF + 1% SP + 0.28% W/B                   | 9 – 11       |
| [12] | 38.5% OPC + 19.2% Fly ash + 12.7% Sand + 26.8% Water + 2.8% additive (strength + water-reducing) | 8.1          |
| [13] | 95% OPC + 5% SF + 0.3 W/C + 2.0% SP                   | 15.0         |
| [7]  | 36.6% Portland cement + 17.1% Fly ash + 17.1% Sand + 25.7% Water + 3.5% Resin modifier | 9.0          |
| [14] | (0.92% OPC + 0.08 UEA*) as binder + 0.45 W/B + 0.25 S/B + 0.3 SP/B | 11.51        |
| [15] | 0.65 W/C + 14% Sand + 10% Mineral powder + 6% Fly ash | 11.4         |
| [16] | 0.65 W/C + 10% polymer/C + 20% Sand + 10% Mineral powder | 11.1         |
| [17] | (Cement + Water + additives), by trial and error       | 9 – 11       |
| [18] | 0.80% Portland cement + 0.05% SF + 0.15% Fly ash + 0.29% W/B + 8 (ml/kg) SP + 3 (ml/kg) SRA**    | 23.0         |
| [19] | (720 Water + 1000 Portland cement + 497 Sand + 249 Filler + 30 Latex powder) by g | 12.8         |
| [20] | 46.5% OPC + 7% Fly ash + 14% EA + 27.9% S + 4.7% Water + 0.6% SP (OPC + SF + W + SP) trial and error | 11.87        |

* Expansion agent type UEA, ** Shrinkage-reducing admixture (SRA).

The used material types in grout preparation have high effectiveness on grout fluidity, especially the superplasticizer. The superplasticizer carboxylic or polymers have more effectiveness to a depressing cementitious particle which cause high fluidity based on Sulphonated Naphthalene Formulation [21]. Also, water content has a high effect on grout fluidity and strength properties. A sustainable approach was suggested in this study. Modified grout was suggested by a blend of OPC, water, superplasticizer with partial replacement of OPC with SF and palm frond waste ash (PFWA). The incorporating of PFWA was due to its environmentally friendly, economic, and resource conversation issues [22]. PFWA is available widely in Arab countries regions as Iraq and Saudi Arabia. As an example, there are about 360,000 tons of date palm trees collected every year due to pruning the 12 million palm trees grown in the Kingdom of Saudi Arabia, according to a report published by King Abdulaziz City for science and technology [23]. Similarly, in Iraq, there are about 1703650 palm trees in 2019, according to the Ministry of Planning Central Statistical Organization [24]. Therefore, this study aims to find the effect of the use of silica fume, PFWA, and superplasticizer on the mechanical properties of the grouts used in SFP.

### 2. Experimental work

#### 2.1 Materials

Materials that are used to prepared sustainable modified grout are listed below:

**2.1.1 Ordinary Portland cement (OPC)**

According to Iraqi specification No: 5/1984 type I [25], OPC used in this study is (CEM I 42.5R) produced at Karbala cement plant. The chemical and physical properties are shown in Table 2.

**2.1.2 Silica fume (SF)**

The silica fume used in this study was produced by CONMIX company. It is a very fine powder called micro silica is by-product material of the smelting processes used in the silicon and ferrosilicon industry [26]. The chemical and physical properties of SF are shown in Table 3.
### Table 2. Physical and chemical properties of OPC.

| Property                  | Results | Requirement |
|---------------------------|---------|-------------|
| Fineness (m²/Kg)          | 339     | 250         |
| Density (gm/cm³)          | 2.00    | Not specified |
| Initial Setting Time (min)| 148     | ≥ 45        |
| Final Setting Time (hr.)  | 3.15    | ≤ 10        |
| Expansion (mm)            | 1       | ≤ 10        |

**Physical**

- SiO₂: 19.91 (Not specified)
- Al₂O₃: 4.58 (Not specified)
- Fe₂O₃: 4.83 (Not specified)
- CaO: 61.10 (Not specified)
- MgO: 3.14 ≤ 5%

**Chemical**

- SO₃: 2.23 C₃A ≤ 5 % SO₃ ≤ 2.5 %
- C₃A more than 5 % SO₃ ≤ 2.8 %
- Na₂O: 0.20 (Not specified)
- K₂O: 0.51 (Not specified)
- Chloride: 0.019 (Not specified)
- L.O. I: 2.91 ≤ 4.0 %

**Eq. Alkalies**

- 0.54 < 0.6 % for low alkalies

**I.R**

- 1.09 ≤ 1.5 %

**L.S.F**

- 0.9504 0.66 % - 1.02 %

**SM**

- 2.12 (Not specified)

**AM**

- 0.95 (Not specified)

**C₃A**

- 3.97 3.5 %>

### Table 3. Chemical and physical properties of SF.

#### Physical properties

| Surface area (m²/kg) | 18100 |
|----------------------|-------|
| Density (kg/m³)      | 700   |

#### Chemical properties

- Na₂O: Sodium 1.534
- MgO: Magnesium 0.432
- Al₂O₃: Aluminum 0.091
- SiO₂: Silicon 92.05
- Cl₂O: Chlorine 0.001
- K₂O: Potassium 1.886
- CaO: Calcium 3.035
- TiO₂: Titanium 0.002
- MnO: Manganese 0.149
- Fe₂O₃: Iron 0.448
- Co₂: Cobalt 0.006
- CuO: Copper 0.017
- ZnO: Zinc 0.179
- SrO: Strontium 0.016
- Y₂O₃: Yttrium 0.005
- BaO: Barium 0.057
2.1.3 Palm frond waste ash (PFWA)

PFWA is not available as a product yet; therefore, a process of producing PFWA was designed to stimulate its production in a heat recovery power plant. Preparation of PFWA includes: collecting the palm frond from orchards of Karbala; it has been fallen in the autumn season. Then burn it in the open area to minimize its volume. The produced ash is grinded then sieved on #200. Finally, the ash is re-burn at 900°C in a furnace to achieve the required chemical properties to use as PFWA. The process of preparation PFWA is shown in Figure 1.

Table 4. Chemical component of PFWA.

| Palm frond waste ash | Oxide (%) |
|----------------------|-----------|
| 15.09                | CaO       |
| 38.49                | SiO₂      |
| 3.78                 | Al₂O₃     |
| 3.87                 | Fe₂O₃     |
| 2.69                 | SO₃       |
| 12.36                | MgO       |
| 5.21                 | Na₂O      |
| 8.79                 | K₂O       |
| 9.91                 | Loss on ignition |
| 3925                 | Blaine fineness(m²/kg) |

2.1.4 Superplasticizer (SP)

Superplasticizer was supplied by (LYKSOR) company “Nano flow 5500”, hydroxylamine compound with organic amine and Hydroxyl, its effect fluidity of grout and get more cement strength due to its
high water reducer, which is confirmed to ASTM C494-15 type G [27], the recommended dosage is (0.5-2) % by weight of cement.

### Table 5. Technical properties of LYKSOR SP.

| Property                          | Description/value          |
|----------------------------------|-----------------------------|
| Color                            | Colorless to light yellow liquid |
| Water content                    | 10-15%                      |
| Chemical formula                 | C₉H₂₁NO₃                   |
| Smelling                         | Very light ammoniac odor    |
| Physical form (25°C)             | Liquid                      |
| Molecular point                  | 163.22                      |
| Freezing point                   | 3-8°C                       |
| Boiling point                    | 104-107°C                   |
| Flashpoint                       | Min. 154°C on a dry base    |
| Specific gravity (25/4°C)        | 1.027                       |
| Viscosity                        | 400-500 cps                 |
| TIPA content                     | 85% Min.                    |
| MIPA and DIPA content            | 3% Max.                     |

#### 2.1.5 Water

The water used in this study is tap water.

#### 2.2 Methods

Three stages follow the method: the first stage determines the optimum SP content, the second stage determines optimum W/B content, and the third stage determines the effect of using PFWA as a SCM. At the first stage, the SP percent was (0.5, 1, 1.5, and 2) % with 30% W/B, while the second stage begun W/B percent of (30, 35, 40, and 45) %, and the third stage incorporates PFWA percent as cement replacement at (5, 10, and 15) % with another W/B content (more than 40% due to PFWA added that has a property to absorbing water during mixing [22] that caused flow time increase more than (16 second)) to reach grout fluidity within the range (11-16) sec. The SF percent as cement replacement 5% decided from previous research works [13,28]. The grout mixtures designation is shown in the test matrix present in Table 6.

### Table 6. Cementitious grout matrix.

| Mix. | Stage | OPC (%) | SF (%) | PFWA (%) | SP (%) | W/B (%) | Flow time (s) |
|------|-------|---------|--------|----------|--------|---------|---------------|
| M0   | one   | 100     | 0      | 0        | 0.5    | 30      | 53            |
| M8   | one   | 100     | 0      | 0        | 1      | 30      | 39            |
| M9   | one   | 100     | 0      | 0        | 1.5    | 30      | 26            |
| M10  | one   | 100     | 0      | 0        | 2      | 30      | 19            |
| M4   | two   | 95      | 5      | 0        | 2      | 30      | 19            |
| M5   | two   | 95      | 5      | 0        | 2      | 35      | 15            |
| M6   | two   | 95      | 5      | 0        | 2      | 40      | 11            |
| M7   | two   | 95      | 5      | 0        | 2      | 45      | 9             |
| M1   | three | 90      | 5      | 5        | 2      | 42      | 12            |
| M2   | three | 85      | 5      | 10       | 2      | 46      | 13            |
| M3   | three | 80      | 5      | 15       | 2      | 50      | 13            |

#### 2.3 Preparation of grouts

The steps of preparing the cementitious grout materials are illustrated as flows:

1. Add SP to the water and mix them well to ensure the homogenous and activate SP to add the dry materials.
2) Adding the dry materials to water while the mixing device is operated, as shown in Figure 2-a, ensures the homogenous paste and prevents the balling phenomenon.

3) The prepared mix is subjected to a flow time test.

4) The produced grout is cast into the cubic mold with (50×50×50 mm) or beam (200×50×50 mm), as shown in Figure 2-b.

5) The specimens are demolded after one day or two days depending on its hardening, the early curing temperature (when specimens in mold) is saved at 20°C according to ASTM C 109/C 109M [29] by heater fan device as shown in Figure 2-c, the specimens maturation in water with 20°C according to ASTM C 109/C 109M [29] too, curing temperature was controlled by the heater as shown in Figure 2-d.

2.4 Grouting materials tests
Two types of testing were nominated to characterize the cementitious grout at fresh and hard states as follows:

1) Fresh test (Fluidity test): flow cone test is used to calculate the flow time of grout using flow cone mold that conforms to ASTM C939-10 [30], as shown in Figure 3-a.

2) Hard tests (Mechanical tests): compressive strength test at 3, 7, 14 and 28 days conform to ASTM C942-10 [31] as shown in Figure 3-b, and flexural strength test at 3, 7 and 28 days conform to ASTM C348-14 [32] as shown in Figure 3-c, are nominated to determine the mechanical properties of the developed grouts.
3. Results and discussion
The results that characterized fresh and hard grout properties using flow cone, compressive strength, and flexural strength tests are shown below:

3.1 Fluidity of cementitious grout
Flow test for different grout mixes is conducted to ensure flow time of (11-16) sec as recommended by other studies [6,8] for appropriate injection of grout into porous asphalt layer. As shown in Figure 4, the maximum flow time is 53 s with 0.5% SP for mix M0 and the minimum flow time is 19 s with 2% SP for M8. This is because SP worked to separate the grains of dry content of the grouting materials caused high workability and fluidity of SP in mortar. This result agrees with other studies’ findings [33, 34]. Results revealed that as needed for semi-flexible pavement for success injecting of grout into the open-graded asphalt mix, the flow time should be (11-16) sec, although no mix with W/B of 0.3 is reached this level, M10 with 2% SP is the best grout fluidity. On the other hand, using more than 2% SP is limited as recommended by manufacture where the highest dosage is used.

![Figure 4. Flow time at SP % dosages with 30 % W/B.](chart4)

To reach the required level of fluidity, increasing W/B ratio is mandatory, although it could affect the mechanical properties of the developed grout. As shown in Figure 5, increases W/B from 30% to 45% minimizes the flow time from 19 to 9 s, due to the high workability and fluidity of water in a mortar. This result agrees with other studies [34,35]. However, the upper and lower limit of (11-16) sec for SFP grout can be achieved by (35-40) % W/B as more appropriate percentages.

![Figure 5. Flow time at different water content and 2% SP.](chart5)
3.2 Compressive strength of cementitious grout

The results that were achieved from testing of 50×50×50 mm cubes at ages of 3, 7, 14, 28, 56, and 96 days for different water content are shown in Figure 6, and for different PFWA replacement content are shown in Figure 7.

![Figure 6. Compressive strength for W/B % change.](image1)

![Figure 7. Compressive strength for PFWA change.](image2)

As shown above, in Figure 6, grout with 40% W/B has gained the highest compressive strength for all ages, followed by 35%, then 45%, then 30%. That is might be related to the water quantity needed for hydration reaction where the optimum value is 40%, and when increased to 45%. The compressive strength decreases due to excessive W/B that negatively works on paste structure full with a void formed after extra water evaporation. However, 40% W/B was adopted for further grout development.

As shown in Figure 7, the compressive strength is decreased with 5% PFWA replacement, then increased with 10%, after that return to decrease when PFWA increased to 15%. At 5%, the SCM effect is still low, and the pozzolanic activity is still low and uncompleted. The improvement in strength at 10% could be due to the denser microstructure of mortar caused by the PFWA effect. Additionally, the increase in grout strength with 10% PFWA may be attributed to the continuous formation of additional
calcium silicate hydrate (C–S–H) upon the reaction of reactive silica of pozzolan and calcium hydroxide (CH) produced by the cement hydration. This provides additional strength, particularly at later ages, as confirmed by a previous study [36]. At 15% PFWA level, the strength decreases due to interconnected micro pores that cause weak microstructure than 10% without these pores, as suggested by previous study [22], and agreed with others [35].

3.3 Flexure strength of cementitious grout

The results of molded in 200×50×50 mm prism with three prisms for every single result at 3, 7 and 28 days for different water content are present in Figure 8, while developed grout with different PFWA replacements is shown in Figure 9. As shown for a water change and PFWA change, the flexural strength results behaved similarly to compressive strength. However, the sense after the reason is the same, where the extra water left the grout structure with a high void minimizing the ability to resist flexural stresses. At the same time, the pozzolanic action of optimum value of PFWA facilitates an extra C–H–S that sustains the hydration products and improves flexural strength. Therefore 40% W/B ratio with 10% PFWA as OPC replacement in the presence of SF and SP finalizing best grout for SFP mixture.

![Figure 8. Molded with different water content.](image)

![Figure 9. Developed grout with different PFWA replacements.](image)
4. Conclusions
The aim of this research included characterizing the mechanical properties of cementitious grout materials that used for SFP using a sustainable approach by replacing part of OPC by PFWA, and the main conclusion is shown below:

- The cementitious grout's flux increases with an extreme limit of SP%, i.e., 2% SP is the best-achieved percent. Also, fluidity is decreased when PFWA% increase which implies increases in the W/B ratio.
- The required flow time limit of (11-16) seconds suitable for grout of SFP mixture is achieved with 2% SP and more than 0.35 W/B.
- The fresh properties of grout affect the mechanical properties, whereas optimum W/B% content according to compressive strength and flexural strength is achieved with 40%.
- In terms of mechanical properties, the 10% PFWA replacement of OPC is the optimum percent according to compressive strength and flexural strength results.

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