Sustainable Development of Highly Flowable Cementitious Grouts for Semi-flexible Pavement Mixture

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Abstract
Grout is a substance with a cement which is characterized by a high strength and it is a high workability, so it can be injected in places of small cracks or places of water leakage. Generally, it is used to repair cracks or defects of concrete, also it can be used to fill the voids under the metal bases or digging anchors. Moreover, grout is used efficiently in the process of production the semi-flexible pavement mixtures. The purpose of this study is to design a sustainable grout using waste and by-products materials, to end as flowable grout for semi-flexible pavement mixtures. The mixture of the grout was prepared using Ordinary Portland Cement (OPC), silica fume (SF), paper sludge ash (PSA), superplasticizer (SP), and water. Different proportions of mentioned materials were used to characterizing the developed grout through flowability, compressive strength, and flexural strength tests. Results showed that the flow time increases with increase in the surface area of cementitious materials. Also, the compression strength of the grouts comprised OPC+PSA, revels that there is an optimal dosage: i.e., 15% PSA. Whereas SF generally leads to increase the compression strength. On other side, flexural strength test confirmed the upgrading of developed grouts. As a conclusion, a sustainable grout can be produced with acceptable characteristics when grout constitutes are optimized.

Key words: Cementitious grout materials; flowability; paper sludge ash; semi-flexible pavement; silica fume.
1. Introduction

The increase in demand for cement plants leads to more CO₂ emissions that circulate in the air in the form of particles and prevent sunlight from leaving the Earth, leading to global warming that the world is currently suffering [1, 2]. The demand for cement plants must be reduced through the use of waste or by-products materials as a supplementary cement material to conserve the globule trend for sustainable construction materials.

Grout can be defined in the simplest form, as a mixture of cementitious materials with water and some time with superplasticizer admixture (SP), with or without aggregates [3, 4]. The blend is a completely consistent mixture without any separation between the components. In geotechnical engineering, grout is a form of soil improvement by injecting a liquid material into the subsurface soil or layers that contain voids or cavities, thus it can improve soil properties [5, 6]. Grouting is generally achieved through drilled holes and pump under a high pressure [7]. The injected material is referred as “grout material”.

The discovery of engineering injection technology dates back to the French civil engineer Charles Berigny. 1802 in the harbor of Dieppe, Charles noticed that the soil was exposed to erosion due to water as a result of the tidal process, thus adversely affected the constructed structure. At that time, the injection technology was used to protect the soil from the erosion process [8].

Grout types are used depending on where they are it is applied, such as bonded prestressed tendon grout, auger cast pile grout, masonry grout, and pre-placed aggregate grout [9-11]. Grout can also perform some other functions, such as improving fire resistance, security, acoustical performance, termite resistance, blast resistance, thermal storage capacity, and anchorage capabilities [12, 13]. Grout can also be used in semi-flexible pavement, where the grout can penetrate the voids in the pavement to upgrade its performance [14, 15].
Semi-flexible pavement (SFP) was developed during the 1950s [16]. SFP blend is made by filling an open-graded asphalt skeleton, which has air voids of around 25–30%, with cement-based grouting material. This layer combines the pre-eminent qualities of bituminous pavements (flexible) and concrete (rigid) [17].

Grout injection technology has been used for porous asphalt due to the problems that exist in flexible and rigid paving, and one of the most important problems that exist in flexible pavement is the rutting due to high loads as well as the problems that exist in the rigid pavement of the joints that lead to the discomfort of the passengers during the use of the road, the construction time is long, and it also takes a long time to open because it needs curing to gain strength.

Different research studies focused on using supplementary cement materials in grout production. [18] Concluded that the cement grout comprised silica fume and natural pozzolan by (5, 10, and 15%) does not show any bleeding or separation between its components when it is in a fresh state, as well as, while the grout in a hard state, it gives high compression strength, good resistance to sulfate attack, and its performance of shrinkage is accepted.

[19] Concluded that adding ultra-fine slag up to 15% reduces bleeding without affecting flowability, improves compression strength and also reduces shrinkage.

Another study has proven that mixing cement with methacholine improves compression strength but reduces fluidity for the same proportions of water and superplasticizer, while when mixing cement, methacholine, and ultra-fine granulated blast furnace slag, no improvement in compression strength appeared, but an increase in fluidity was noticed compared to cement mixed with methacholine [20].

The purpose of this study is to design more sustainable grout using another type of waste or by-products materials, namely Paper sludge Ash (PSA) and Silica Fume (SF) to end as flowable grout for semi-flexible pavement mixtures development.
2. Experimental work

2.1 Materials

Cement used in this study is ordinary Portland cement (CEM I 42.5R), confirmed the requirement according to Iraqi standard No: 5/1984 type I. Table (1) shows the chemical and physical properties of this type of cement.

Table 1 Physical and chemical properties of OPC.

| Property                  | Results | Requirement |
|---------------------------|---------|-------------|
| Blain (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        |

| Property   | Results | Requirement |
|------------|---------|-------------|
| SiO₂       | 19.91   | Not specified |
| Al₂O₃      | 4.58    | Not specified |
| Fe₂O₃      | 4.83    | Not specified |
| CaO        | 61.10   | Not specified |
| MgO        | 3.14    | ≤ 5%        |
| 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%      |

In Europe, approximately 11 million tons of paper sludge waste is producing during the pulp and paper making process [21], and this waste is treated by burning it to restore energy and reduce its size, thus paper sludge ash (PSA) is produced as waste materials. However, these factories do not available in Iraq to produce paper sludge waste yet due to availability of oily electrical power plants. However, multi power resources
and renewable energy are becoming a common strategy worldwide, whereas waste paper is available widely, so it decided to simulate PSA production to sustain this trend locally. The PSA was prepared in the form of three phases:

Firstly, burn large amounts of paper as a primary burn to reduce its size, expelling the oxides and converted to ashes. Secondly, burning obtained ash from the first stage in an electric oven at a temperature of 800 °C for two hours to be converted to pozzolanic materials as recommended by [22]. Finally, it was grinded by mill for a period of half an hour to simulate the real PSA [23]. Table 2 illustrates the chemical properties of the PSA, and the scanning electron microscope (SEM) can be illustrated in the Figure 1.

Silica fume or also called condensed silica fume or micro silica, is a very fine powder and contains a high content of amorphous silicon dioxide. This material is a by-product of silicon and ferrosilicon smelting processes [24]. The physical and chemical properties of used SF can be shown in Table 2, and the scanning electron microscope (SEM) can be illustrated in the Figure 1. It should be noted that the physical properties (surface area) of the test of SF in this study were adopted from [25] because same material was used in his research.

Table (2) Illustrates the physical and chemical of SF and PSA.

| Properties                      | SF   | PSA   |
|---------------------------------|------|-------|
| Surface area (m²/kg)            | 18100| 1517  |
| Density (kg/m³)                 | 700  | 2.63  |
| Chemical composition            |      |       |
| Na Sodium                       | 1.534| 0.584 |
| Mg Magnesium                    | 0.432| 0.082 |
| Al Aluminum                     | 0.091| 0.027 |
| Si Silicon                      | 92.05| 0.361 |
| Cl Chlorine                     | 0.001| 0.236 |
| Element | Name         | Concentration 1 | Concentration 2 |
|---------|--------------|-----------------|-----------------|
| K       | Potassium    | 1.886           | 0.093           |
| Ca      | Calcium      | 3.035           | 98.832          |
| Ti      | Titanium     | 0.002           | 0.002           |
| Mn      | Manganese    | 0.149           | 0.021           |
| Fe      | Iron         | 0.448           | 0.204           |
| Co      | Cobalt       | 0.006           | 0.002           |
| Cu      | Copper       | 0.017           | 0.015           |
| Zn      | Zinc         | 0.179           | 0.004           |
| Sr      | Strontium    | 0.016           | 0.114           |
| Y       | Yttrium      | 0.005           | 0.002           |
| Ba      | Barium       | 0.057           | 0.006           |

Superplasticizer (SP) was supplied from CONMIX company (under the trade name MegaFlow 1000). MegaFlow 1000 is a modified polycarboxylate ether-based superplasticizer which has a unique carboxylic ether polymer with long lateral chains. It is an effective cement dispersant, fluidified and high range water reducer. The specific gravity of SP is 1.08, it is confirm to ASTM C494-15 type D and G [26] and the recommended dosage is (0.5-2) % by weight of cement.

Figure 1. SEM of the: (A) SF and (B) PSA.
Finally, the used water in the mixture is RO water, while the water used during the curing time was tap water.

2.2 Methods

Developed grouts were designed to be with different OPC replacement by waste and by-product materials and various water/binder (W/B) ratio, that facilitate investigation aim in two stages, namely:

1st phase is specifying the optimum blend by using thirteen mixtures in different replacement proportions, with a constant (W/B) ratio of 0.5%, and a superplasticizer dosage of 1% of the weight of the binder. To confirm reliability, the PSA and SF replacement proportions decided from previous research works [27, 28].

2nd phase is specifying the optimum fluidity by depending on the optimum mixtures (M 4, M 6, M 11) that were nominated from the 1st phase. The work has been already conducted with a change in the superplasticizer and keeps the W/B ratio constant. The ratios used in the 1st and 2nd phases are indexed in Table (3).

| Mix | Phase | % OPC | % PSA | % SF | % W/B | % SP |
|-----|-------|-------|-------|------|-------|------|
| M1  | 1     | 100   | 0     | 0    | 0.5   | 0    |
| M2  | 1     | 95    | 5     | 0    | 0.5   | 1    |
| M3  | 1     | 90    | 10    | 0    | 0.5   | 1    |
| M4  | 1     | 85    | 15    | 0    | 0.5   | 1    |
| M5  | 1     | 80    | 20    | 0    | 0.5   | 1    |
| M6  | 1     | 97.5  | 0     | 2.5  | 0.5   | 1    |
| M7  | 1     | 95    | 0     | 5    | 0.5   | 1    |
| M8  | 1     | 92.5  | 0     | 7.5  | 0.5   | 1    |
| M9  | 1     | 90    | 0     | 10   | 0.5   | 1    |
| M10 | 1     | 92.5  | 5     | 2.5  | 0.5   | 1    |
| M11 | 1     | 85    | 10    | 5    | 0.5   | 1    |
| M12 | 1     | 77.5  | 15    | 7.5  | 0.5   | 1    |
| M13 | 1     | 70    | 20    | 10   | 0.5   | 1    |
| M14 | 2     | 85    | 15    | 0    | 0.5   | 1.5  |
| M15 | 2     | 85    | 15    | 0    | 0.5   | 2    |
| M16 | 2     | 97.5  | 0     | 2.5  | 0.5   | 1.5  |
2.3 Preparation of sample grouts

All grout samples were prepared at laboratory temperature and under standard conditions. Firstly, the dry materials were mixed well and homogeneously depending on the different proportions for each mixture. Secondly, mix the superplasticizer with the water until it is well homogeneity. Then, dry materials were added to the liquid materials and mix with a regular speed. Next, 1750 ml. of the grout was inserted into a flow cone test to determine the flow of the grout time. Finally, the grout was poured into cubes of 50 x 50 x 50 mm or beam of 160 x 40 x 40 mm depends on the required test. After pouring the grout into the mold, the samples remain in the mold for one day, then de-molded, finally curing was achieved in water until the day of the test.

2.4 Test of cementitious grout

There are two types of tests that were performed on grouts:

1- Fluidity test: which is the test of the flow time of the grout with the time using a mold of flow cone test of standard dimensions that conform to the ASTM C 939-10 [29]. This test is done when for the grout in a fresh state.

2- Mechanical properties tests: compressive strength test for the grout cubes for ages of 1,3,7,14 and 28 days, conforms to the ASTM C 942-10 [30]. As well as, flexure strength test for the grout at age of 28 days using standard-dimensional molds that conform to the ASTM C 348-14 [31]. This test is done for the grout in a hard state.

3. Results of cementitious grout

The results obtained from the flow test, compression and flexure strength of - all mixtures at different ages are discussed in the following subsections:
3.1 Fluidity of cementitious grout

Fluidity results that obtained from the 1st phase, according to the mixing proportions for each mixture can be illustrated in the Figure (2).

![Figure 2. Fluidity of cementitious grout (1st phase).](image)

The results demonstrate that mixtures M2 to M5, which contain PSA values of (5 - 20) % as a replacement OPC, showed increase in fluidity with increase in PSA, as a results of both increase in total binder surface area, further to the nature of PSA morphology which suction additional water between its particles as suggested by [32, 33]. Simultaneously, this thing is identical to the mixtures M6 to M9 which contains SF values of (2.5 - 10) % and also for mixtures M10 to M13 that contain PSA values of (5 - 20) % and SF (2.5 - 10) % collectively. However, the high surface area of SF is governing the fluidity of the developed mixtures significantly.

Consequently the mixtures M4, M6, and M11 can be nominated as the optimum mixtures obtained from the 1st phase, because the required flow time is ranged between 11-16 sec as recommended by [34], where to ensure complete penetration of the porous asphalt sample low flow time is preferable. However, M4 containing 15% of PSA was
chosen from the mixtures group that contained PSA only, as the change in fluidity due to increase PSA is very limited. Moreover, increase used PSA dosage is promoted the aim of the study for enhance environment and reduce the percentage of used OPC and reduce CO₂ emissions. Likewise, the selection of the M6 which contained the SF dosage of 2.5% is based on fluidity within the permissible time and gives properties that are very close to the traditional mix. The same also is true for the selection of M11 which contains a mixture of 10% PSA and 5% SF. In summary, the base of selection was dependent on getting as much as possible OPC replacement by supplementary materials but with lower fluidity time value, of course using higher PSA is preferable than SF in term of economic issue.

The 2\textsuperscript{nd} phase, which is focused on optimizing the proportions of the superplasticizer, while the W/B ratio remaining constant. the results of this phase can be illustrated in Figure (3).

![Figure 3. Fluidity of cementitious grout (2\textsuperscript{nd} phase).](image)
According to the results of the 2nd phase, there is a general trend in all mixtures groups revels that increase SP led to an increase in fluidity time then decrease is noticed. However, it can say that the change is very limited and within the average recommended fluidity mentioned range, this is corresponded to the results of [35]. Nevertheless, however, the explanation for increasing the fluidity by adding the SP is that when the SP particles are contacted with the cement particles, the SP molecules are adsorbed on the surface of the cement particles, which leads to the formation of a negative charge on OPC particles that repels each other so that cement particles do not flocculate with each other [36]. In general, the fluidity of the 2nd phase mixture confirms the selection of the optimum mixture and facilitate other mechanical tests to advise the optimum supplementary materials dosages.

3.2 Compressive strength of cementitious grout

The compressive strength characteristic was investigated on cementitious grout cubes 50 x 50 x 50 mm with 3 cubes for each age of 1, 3, 7, 14 and 28 days. Figures (4, 5, and 6) shows the compressive strength for mixtures with OPC replacement by PSA, SF, PSA+SF, respectively, while Figure 7 combines all mixtures at the age of 28 days.

Figure 4. Compressive strength of control mixtures comprising OPC and OPC+PSA.
Figure 5. Compressive strength of control mixtures comprising OPC and OPC+SF.

Figure 6. Compressive strength of control mixtures comprising OPC and OPC+PSA+SF.
According to the obtained results, traditional mixture M1 reveals that the increase in compressive strength is increased with age because cement needs time to complete its hydration process and reach relatively mature strength. M2 to M5 that contain OPC with partial replacement by PSA, in ratios of 5, 10, 15 and 20 %, relatively decrease in compressive strength compare with control M1 associated for all ages, mainly because the type of hydration process as confirmed [27]. However, M4 is the ideal mixture it shows relatively less reduction in compressive strength and significant replacement ratio, i.e. 15%.

M6 to M9 which contain OPC with partial replacement by SF with proportions of 2.5, 5, 7.5, 10% exhibited similar compressive strength increases with age to control M1, but with less value. This is interested as SF normally increase compressive strength of concrete mix; however, this could be due to high W/B that eliminate the gain in strength by increase the voids between the hydration process. Nevertheless, M6 show the most identical mixture to control M1 with very low dosage of SF of 2.5% which is sustain economics of the prepared mixtures.

Figure 7. Compressive strength of all mixtures at age of 28 days.
M10 to M13 mixtures which contain a blending of OPC, PSA, and SF in different proportions showed that, entering SF PSA collectively leads to improved compressive strength compared to that mixtures containing PSA individually. This is a result of consuming the extra water by the activation of both PSA and SF to the OPC, therefore, it can be seeing that M10 is very similar to M1.

The compressive strength of the optimum mixtures obtained from the 1st phase with the change in the ratio of the superplasticizer are presented in Figure (8).

Figure 8. Compressive strength for different ages (2nd phase).

Through the obtained results of M14, M15, M16, M17, M18, and M19. It can observe that the increase in SP dosage does not give significant enhancement to early age strength as the water content remain the same and the carboxylic polymer product networks unable to anchor hydration products. but at former ages the SP products works significantly specially for specific mixtures where extra hydration products are produced extensively, e.g. M16 and M17.
3.3 Flexure strength of cementitious grout

The flexure strength was tested for control mixture M1 and that mixtures nominated as optimum mixes of 1st phase, further to that of 2nd phase mixture for compression purpose, Figure (9) the shows mentioned mixtures at age of 28 days.

![Figure 9. Flexure strength of cementitious grout at 28 days.](image)

The flexural strength of a conventional mixture containing only OPC, is 8.63 MPa, however, when the PSA is introduced, no significant degradation happened with 1% SP, but increase SP reduce flexural strength. This is due to the fact that an increase in the dose of SP leads to more water in the mixture. Hence there is an optimum ratio of SP should use, when doses exceed this limit this leads to reduce strength as confirmed by [37]. On other side, introduce SF to the mix significantly increase the flexural strength for all mixtures with or without the presence of PSA. However, this is due to the fact that the surface area of SF is very high and requires high doses of SP in order to ensure its dispersal within the sample and thus distributed uniformly and thus ensure its effectiveness in giving high strength. Also, it can be noticed that there is un consistent in
the results of compressive strength and flexural strength, which can be explained by the
preparation and molding process that affects the strength results as confirmed by [14].
Finally optimizing the SP for these mixtures include PSA and SF could significantly
increase the flexural strength. This is can be explained by the addition of SP will improve
the binder particles dispersion action and the presence of the long lateral chains linked to
the polymer backbone generates a steric hindrance, which stabilizes the cement particles'
capacity to separate and disperse [38].

4. Conclusion

From the extensive testing program and analysis of the results, the following can be
concluded:

1- The fluidity has a very large relationship to the surface area of the cementitious
materials, the higher the surface area, the greater the flow time.
2- Increasing SP ratios is not always facilitating a batter fluidity. Optimum moderate
value is needed especially when PSA is introduced to OPC. However, high dosage up
to 2% is significant when SF is present in grout mixture
3- 15% PSA replacement of OPC in grout production is the optimum value when
mechanical properties is governed the required properties.
4- 2.5% of SF replacement value is the optimum value when mechanical properties are
governing the characteristics of grout.
5- Using PSA and SF with optimum SP all together in producing grout facilitate
significant characteristics than that with OPC only in term of mechanical properties.

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