

Microstructural and Compressive Strength Analysis for Cement Mortar with Industrial Waste Materials

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Abstract

Cement production uses large quantities of natural resources and contributes to the release of CO₂. In order to treat the environmental effects related to cement manufacturing, there is a need to improve alternative binders to make concrete. Accordingly, extensive study is ongoing into the utilization of cement replacements, using many waste materials and industrial. This paper introduces the results of experimental investigations upon the mortar study with the partial cement replacement. Fly ash, silica fume and glass powder were used as a partial replacement, and cement was replaced by 0%, 1%, 1.5%, 3% and 5% of each replacement by the weight. Compressive strength test was conducted upon specimens at the age of 7 and 28 days. Microstructural characteristic of the modified mortar was done through the scanning electron microscope (SEM) vision, and X-ray diffraction (XRD) analysis was carried out for mixes with different replacements. The tests results were compared with the control mix. The results manifested that all replacements present the development of strength; this improvement was less in the early ages and raised at the higher ages in comparison with the control specimens. Microstructural analysis showed the formation of hydration compounds in mortar paste for each replacement. This study concluded that the strength significantly improved by adding 5% of silica fume compared with fly ash and glass powder.

Keywords: Cement Replacements; Fly Ash; Silica Fume; Glass Waste; Recycling Materials; Compressive Strength.

1. Introduction

Production of cement implicates a high consumption of energy and therefore is responsible for almost (7%) of the world's (CO₂) emission. It's well known that (CO₂) is the main contributor to the greenhouse influence and subsequently being responsible for the global warming of the earth. Thus, research upon the usage of by-product cementing materials, like silica fume, metakaolin, fly ash, waste glass and rice husk ash in place of cement has been increased in concrete technology [1].

Currently, researches on sustainable development on concrete have been carried out on the following aspects: extension of "concrete structure and development of low-carbon concrete material and structure [2]. Contemporary mixed cement types also use pozzolan as a cement replacement material or a mineral additive that is inter-ground or mixed with Portland cement. Pozzolans are defined as "Siliceous or Siliceous-Aluminous materials that have a slight or no cementitious effect, but due to their fine separated shapes and with the existence of moisture. They are chemically reacting with the calcium hydroxide under normal temperature to produce compounds having cementitious characteristics [3]. Krishnaraj et al. (2017) explored the influence of fly ash on the durability characteristics and compressive strength of the mixed cement mortar. The compressive strength test results and the (SEM) analysis

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depicted that incorporating fly ash in mortar accelerates the (C-S-H) gel development via using the content of (C-H), causing better durable and mechanical properties of mortar [4]. Khalaf et al. (2018) investigated the influence of the local fly ash on the cement mortar characteristics. This study found that the compressive strength values were declined for all the fly ash mixes at the early ages, 3 days. However, the compressive strength was improved remarkably at the later ages 28 days, [5]. Yerramala et al. (2012) evaluated the strength characteristics of the fly ash mortars; results revealed that at the early age of all replacements of fly ash, the strength reduced to the ordinary mortar. Nevertheless, after 28 days and beyond, mortars prepared with the replacement of fly ash up to 15% caused a higher strength than the ordinary mortar [1].

Bhandari and Tajne (2013) investigated the mechanical characteristics of mortar blocks possessing different levels of replacements of fine and coarse waste glass with fine aggregate, the results of the test showed that the fine aggregate replacement via fine glass has an essential influence upon the mortar blocks compressive strength in comparison with the control sample due to the pozzolanic nature of (FG). The results elucidated the pozzolanic reactivity of such waste and the open potentials for the use of this material in mortars [6]. Aseel et al. (2016) studied the mechanical characteristics and thermal conductivity of mortar cement produced from glass waste at different values of glass to cement. Ultimately glass waste can be used as an environmentally and cost-effective cement substitute in mortar cement production [7].

Amudhavalli et al. (2012) considered the influence of the partial cement replacement by (0, 5, 10, 15 and 20%) silica fume. The results of the test depicted that the silica fume usage in concrete enhanced the concrete performance in strength and durability features [8]. Sasiakalaa et al. (2012) studied the mortar compressive strength for Ferro cement containing ternary mixes of silica fume, Portland cement, and superplasticizer as a water reducing agent. The results revealed that the compressive strength cement mortar enhanced with silica fume content [9]. Tayeh et al. (2019) confirmed that the use of glass powder in mortar explored the strength properties of mortar and its sulphate resistance, moreover, the results indicated that replacing glass powder in the control mixture increased the unit weight by 29% at 60 days [10]. Czapik and Mateusz et al. (2018) evaluated the effect of silica fume on the properties of cement mortar. This study confirmed the improvement in compressive strength achieved when added silica fume to cement paste [11]. Ortega et al. (2018) studied the long-term effects 400 days of the addition of waste glass powder on the microstructure and service properties of mortars, which incorporate up to 20% of this addition as clinker replacement. And that showed good service properties until 400 days, similar to or even better than those made with ordinary Portland cement without additions, with the added value to contributing to sustainability [12].

This research was conducted to study the effect of using some sustainable materials which have pozzolanic properties such as silica fume, fly ash and fine glass powder which would be introduced to cement mortar technologies with different content. Therefore, research themes will focus on benefits and determinants of the implementation of these replacements in cement and concrete technology. Also, the optimum content for each type would be recognized.

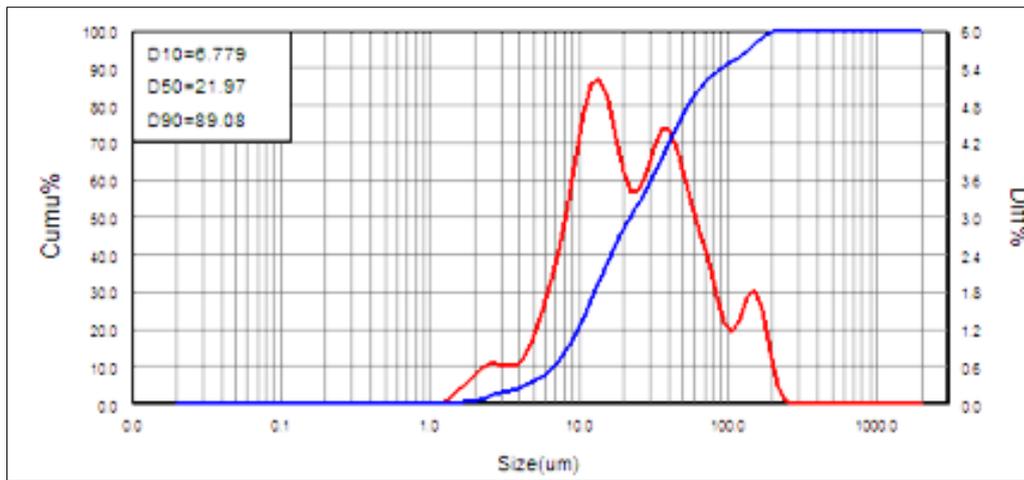
2. Experimental Method

2.1. Materials

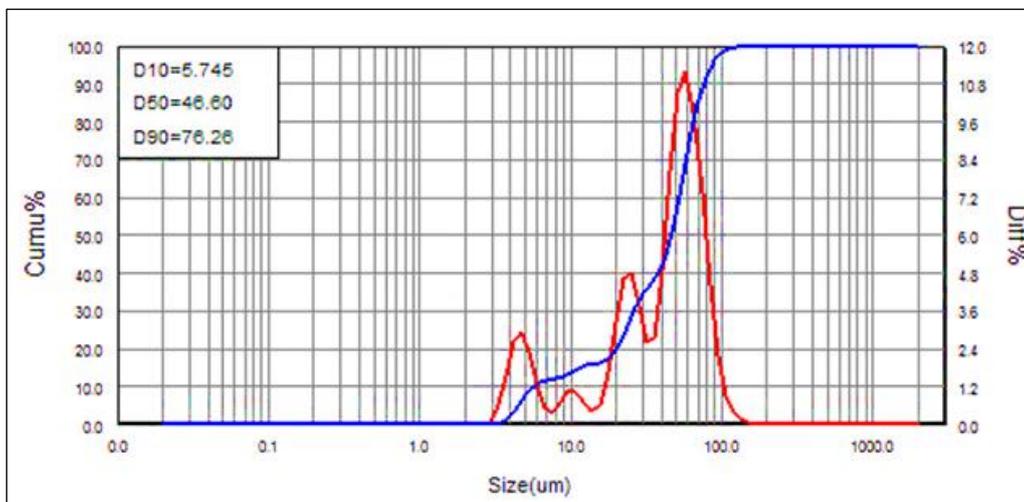
In the current research, the ordinary Portland cement (Type I) confirmed to the Iraqi Standard Specifications No. 5 [13] was used and the chemical compositions of cement shown in Table 1. Silica fume, fly ash and glass powder were used as a partial cement replacement, Figure 1 indicates the particle size analysis for each of them. The silica fume used in this experiment contained (94.6%) SiO_2 , the chemical compositions of fly ash and glass powder were analyzed by an (XRF) microprobe analyzer and depicted in Table 2. Glenium 54 (G54) high range water reducing admixture type F was introduced into the whole blends. Natural sand of river was utilized with the portion of sand that passes through 850 μm sieve according to Iraqi Standard specification no 2080 [14].

Table 1. Chemical composition of cement

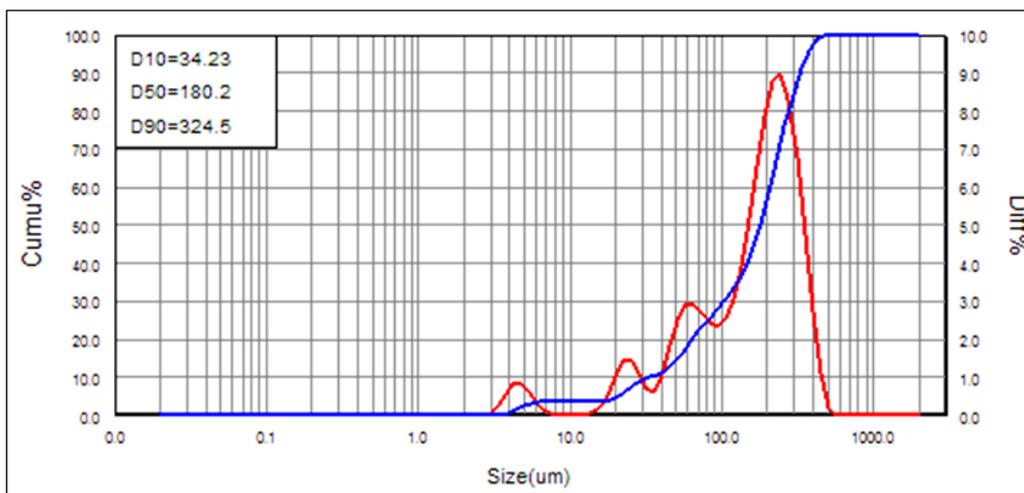
| Oxide | Content, % |
|-------------------------|------------|
| CaO | 66.11 |
| SiO_2 | 21.93 |
| Al_2O_3 | 4.98 |
| Fe_2O_3 | 3.10 |
| MgO | 2.0 |
| K_2O | 0.75 |
| Na_2O | 0.35 |
| SO_3 | 2.25 |



(a)



(b)



(c)

Figure 1. Particle Size Analysis for: (a) Silica Fume (b) Fly Ash and (c) Glass Powder

Table 2. Chemical compositions of fly ash and glass powder

| | SiO ₂ | MgO | K ₂ O | CaO | Al ₂ O ₃ | Cl | Fe ₂ O ₃ | Na ₂ O | SO ₃ | CuO | ZnO |
|---------|------------------|------|------------------|------|--------------------------------|------|--------------------------------|-------------------|-----------------|-------|-------|
| Fly ash | 88.6 | 3.21 | 3.26 | 2 | 0.75 | 3.26 | 1.72 | - | - | - | - |
| Glass | 72.5 | 3.72 | - | 10.2 | 0.7 | - | 0.31 | 9.9 | 0.244 | 0.227 | 0.133 |

2.2. Mixture Details

In this research, thirteen mixtures of mortar consist of (cement, sand, water, superplasticizer and replacements) to prepare the samples. Constant water/cement ratio (w/c) = 0.45 and superplasticizer Percentage=1.25% by the cement weight were used for all mixture. The mortar mixtures details illustrated in Table 3.

Table 3. Mortar mixes

| Mix Symbol | Cement (g) | Sand (g) | Fly ash (g) | Silica fume (g) | Glass powder (g) |
|------------|------------|----------|-------------|-----------------|------------------|
| Control | 500 | 1375 | - | - | - |
| 1F | 495 | 1375 | 5 | - | - |
| 1.5F | 492.5 | 1375 | 7.5 | - | - |
| 3F | 485 | 1375 | 15 | - | - |
| 5F | 475 | 1375 | 25 | - | - |
| 1SF | 495 | 1375 | - | 5 | - |
| 1.5 SF | 492.5 | 1375 | - | 7.5 | - |
| 3SF | 485 | 1375 | - | 15 | - |
| 5SF | 475 | 1375 | - | 25 | - |
| 1GP | 495 | 1375 | - | - | 5 |
| 1.5GP | 492.5 | 1375 | - | - | 7.5 |
| 3GP | 485 | 1375 | - | - | 15 |
| 5GP | 475 | 1375 | - | - | 25 |

F: is the mixes with Fly Ash. SF: is the mixes with Silica Fume. GP is the mixes with Glass Powder

2.3. Compressive Strength Measurements

According to ASTM C109, (50×50×50) mm mortar cubes were cast using the mix proportion shown in Table 3, w/c ratio of 0.45. Specimens were cast, and throughout the moulding, and the cubes were mechanically shaken. Beyond 24 hours, these specimens were de-moulded and subjected to curing in distilled water with two groups the first group cured for 7 and the second group were cured for 28 days. And, beyond curing, such specimens were examined for the compressive strength utilizing a compression testing machine. The tests carried out according to BS 1881-Part 101 [15] on three specimens, and the average values of compressive strength were determined. Compression test was done using BESMAK testing equipment.

3. Results and Discussion

3.1. Results of Compressive Strength

Figures 2 to 4 demonstrate the effect of replacement type on the compressive strength of mortar's specimens after 7 and 28 days of curing. In Figure 2, the results show clearly the mortar mixes, where the silica fume was used as partial replacement, in this figure the compressive strength increased gradually with the increase of silica fume contents, it raised from 27 MPa for control mortar to 41 MPa with the increasing percentage of (5%). The behaviour of improving the compressive strength with the increase of silica fume in mortar mixtures is due to that if the silica fume introduced, the cement hydration rate raises at the initial hours owing to release of (OH) ions and alkalis into the pore fluid. Also, additional SiO_2 will react with CH to produce more CSH gel [16]. This further formation of CSH gel in the cement paste enhanced the mortar strength properties and gave a more dense structure due to the filler effect of silica fume. These results agreed with Al Ghaban et al. (2018) [17] and Rostami and Behfarnia (2017) [18].

In Figure 3, the results indicate that the mixes with fly ash have no appreciable early strength. However, those mixes have gained the strength more than the target strength in later days this phenomenon of gaining strength with late age when using fly ash as partial replacement of cement was demonstrated by Yerramala et al. (2012) [1] and İlhami et al. (2017) [19] and other researchers. The compressive strength was increased from (27) MPa for control mortar to (39.5) MPa for mixes with (5%) fly ash at 28 days. This development in compressive strength might be attributed to the chemical reactivity of fly ash with CH to form CSH gel and thus gain binding property in cement paste according to Krishnaraj et al. (2017) study [4]. In Figure 4, the results reveal, similarly to those of mixes with silica fume and fly ash, that the powder of waste glass incorporation enhanced the cement mortars mechanical strength. Due to the pozzolans in the glass powder composition which would react with the available (CH), chiefly making (CSH) similar to that formed in the hydration reactions of cement, the results of an approach obtained by Sofiane et al. (2019) [20] and Tamanna et al. (2016) [21]. Thus, confirming that waste glass powder can further contribute to sustainability in construction. Comparing the results of compressive strength yielded that silica fume showed more significant improvement in strength by analogy with fly ash and glass powder respectively, this distinction for silica fume can be attributed to its high silica content with the high pozzolanic effect. However, it can

be concluded that all the studied replacements can be considered as a viable replacement for cement and is thus an economical construction material.

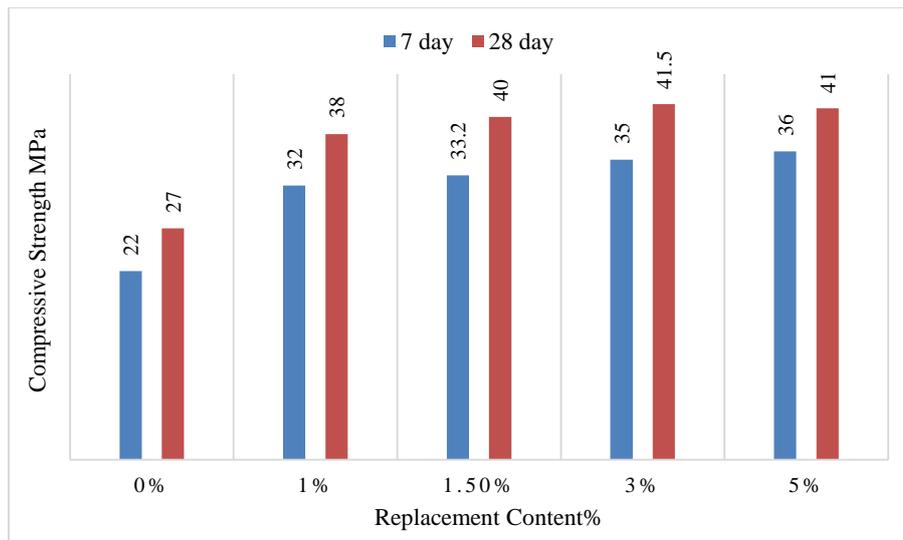


Figure 2. Results of the compressive strength test for the concrete mixes with silica fume as cement partial replacement

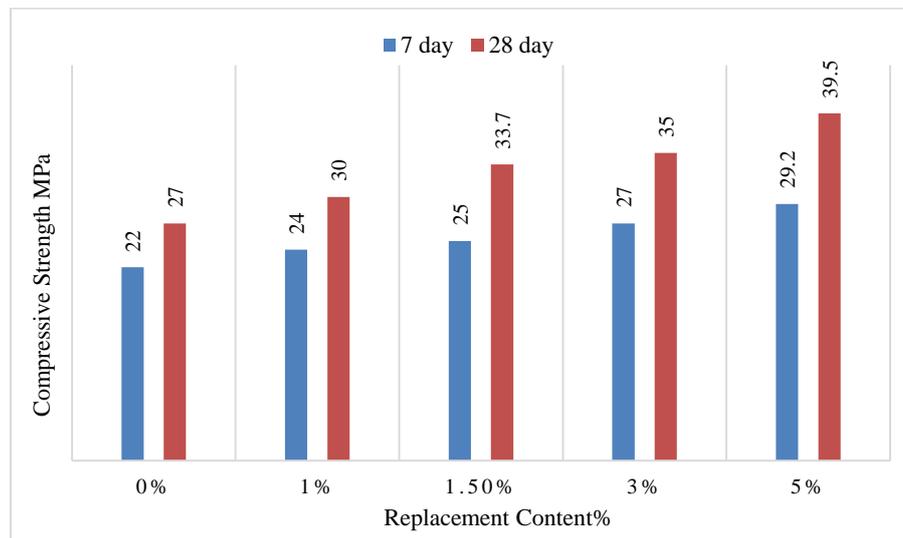


Figure 3. Results of the compressive strength test for the concrete mixes with fly ash as cement partial replacement

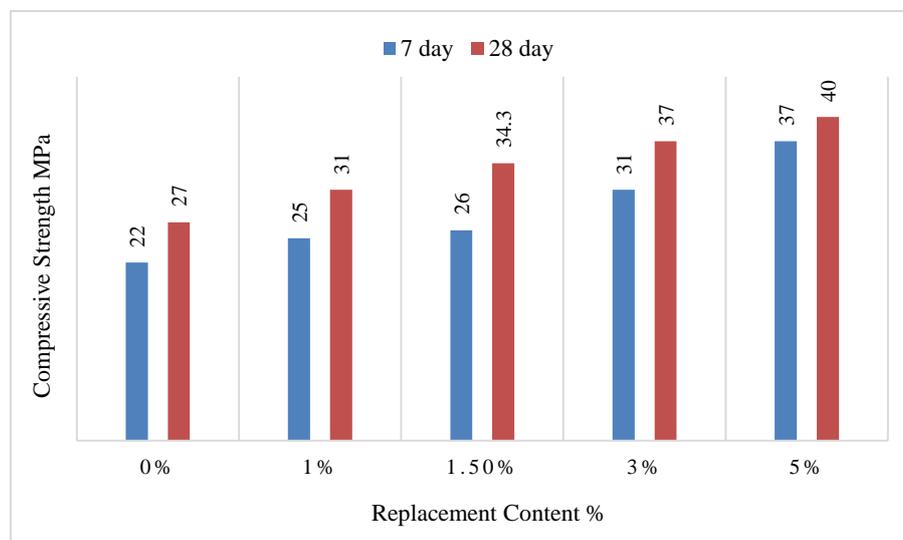


Figure 4. Results of the compressive strength test for the concrete mixes with glass powder as partial cement replacement

3.2. Results of SEM Analysis

From the SEM analysis of blended cement mortar samples, it is evident that no clear particles of silica fume, fly ash or fine glass powder are seen; hence, it proved that all the pozzolanic materials contribute to the hydration process. Reference sample microstructure is shown in Figure 5(a). When these displays were examined, it was observed that the (C-S-H) gels started to form in the cement pastes, Figure 5(b) views that the formation of (C-S-H) gels increased in samples with the silica fume. In Figure 5(c, d) for the (SEM) images of mixes with fly ash or fine glass powder, the (C-S-H) gels observed to be more significant and that they covered most of the internal structure. These findings confirm by Ana Mafalda Matos et al. carried out a study concerning the microstructure of mortar containing waste glass powder [22].

The SEM images of the hardened cement paste show that the distribution of (CSH) nearly increased compared to the reference mix due to the replacement of cement by pozzolanic replacements (silica fume, fly ash and glass powder). In the mixtures, the development in the (CSH) occurred as a result of the reaction between (CH) and pozzolanic replacing materials to produce extra (CSH) and ettringite. This mechanism was accordance with the mechanism proposed by Henry Limantono et al. (2016), explaining excellent hydration between cement, glass powder, and silica fume resulted in a massive layer of paste due to the pozzolanic effect [23]. Also, the filler action of presence the replacement particles which play the primary role in increasing the density of modified mortar compared with reference mortar. This increasing explains the reason behind the strength development when using these materials as partial cement replacement which can be analyzed and explained based on the growth of hydration products in the microstructure of mortar mixes [23, 24].

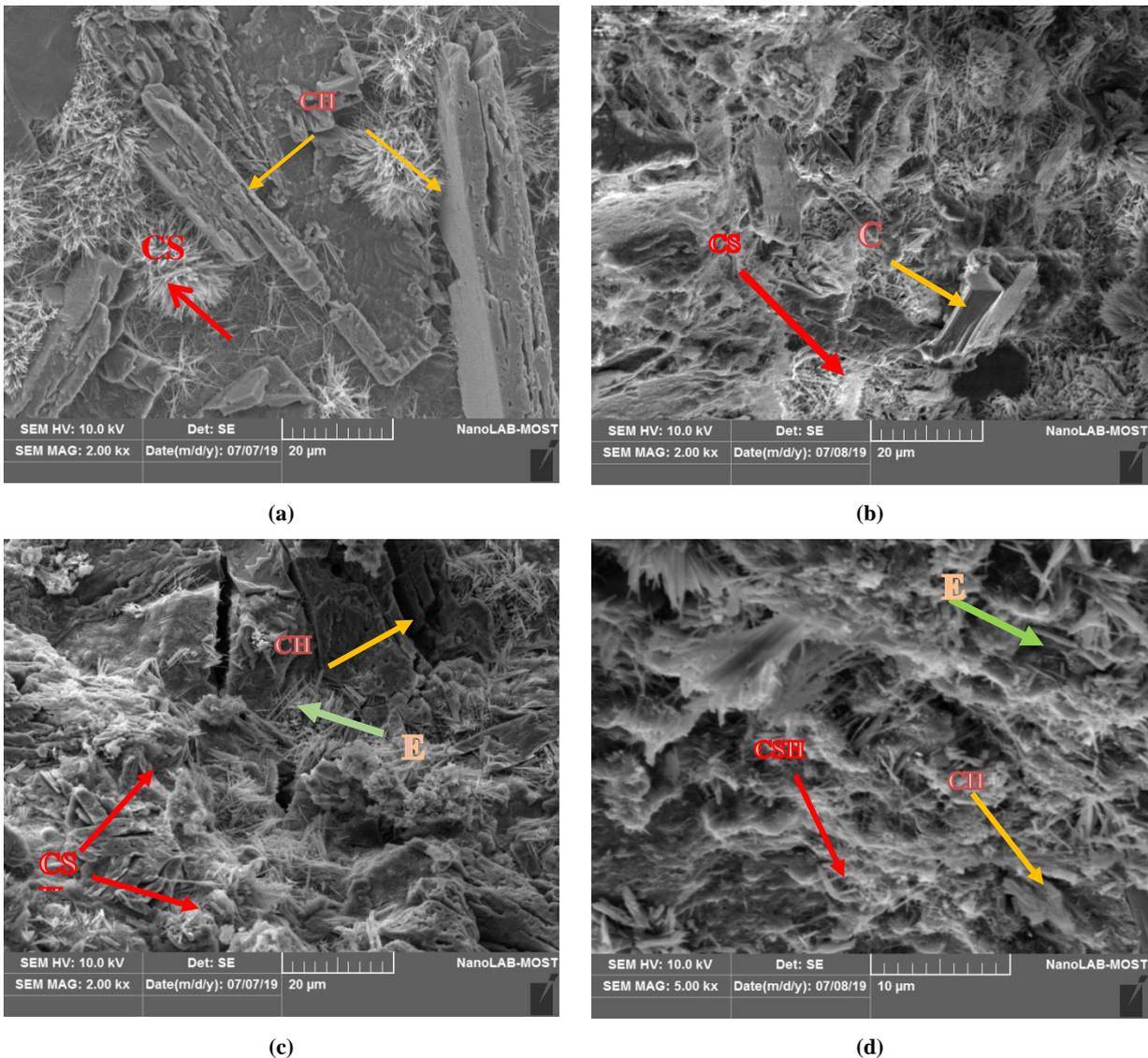


Figure 5. SEM photomicrograph for mortar mix with: (a) Reference Mix, (b) Silica Fume Mix, (c) Fly Ash Mix, (d) Glass Powder Mix

3.3. Results of XRD Analysis

(XRD) analysis performed to investigate the replacements of cement reactivity in the mortar after a curing time of 28 days. Figures 6 to 9 depict the XRD with angular range ($2\theta = 0 - 60$) degree for reference mix and mixes with silica fume, fly ash and glass powder, correspondingly. The primary phases present in the Portland cement after the hydration process were calcium silicate hydrate gel (C-S-H), Calcium Hydroxide ($\text{Ca}(\text{OH})_2$), calcium aluminate (CaAl_2O_4), calcium silicate (CaSiO_3) and ettringite phases. In all XRD spectra, the same three main types of the observed peaks were Calcite (CaCO_3) with a hexagonal crystal system [26], Quartz (SiO_2) with a hexagonal crystal system [27], Portlandite ($\text{Ca}(\text{OH})_2$). Among them, SiO_2 mainly came from the siliceous sand, and CaCO_2 produced from the addition mixed with the cement during its production process and carbonation of cement hydration products, and $\text{Ca}(\text{OH})_2$ was formed from the hydration of cement clinker. This result confirms the finding by Huang and Zhao (2019), who studied the correlation between strength and durability of mortar with fly ash [28]. The hydration process in a mortar with mineral admixture is very complicated as compared with the hydration of Portland cement. Therefore, all (XRD) charts in this research present the calcium silicate hydration (C-S-H) unmanageable to distinguish the glassy phase (lack in crystallinity). Calcium hydroxide (CH) is also difficult to differentiate due to the pozzolanic reaction, which forms calcium silicate hydration (C-S-H) that leads to depleting unknown amount of calcium hydroxide (CH) this was accordance with the results proposed by Sang-Hwa Junget.al, explaining the phases corresponding to hydration of mortar with fly ash and RHA [29]. The results indicate that calcium hydroxide appears at very low peak intensity, as shown in the previous figures because of the consuming of calcium hydroxide (CH) by the pozzolanic reaction that indicates the pozzolanic reactivity of the partial replacement materials, and that is supported by the compressive strength and scanning electron microscope results [30, 31].

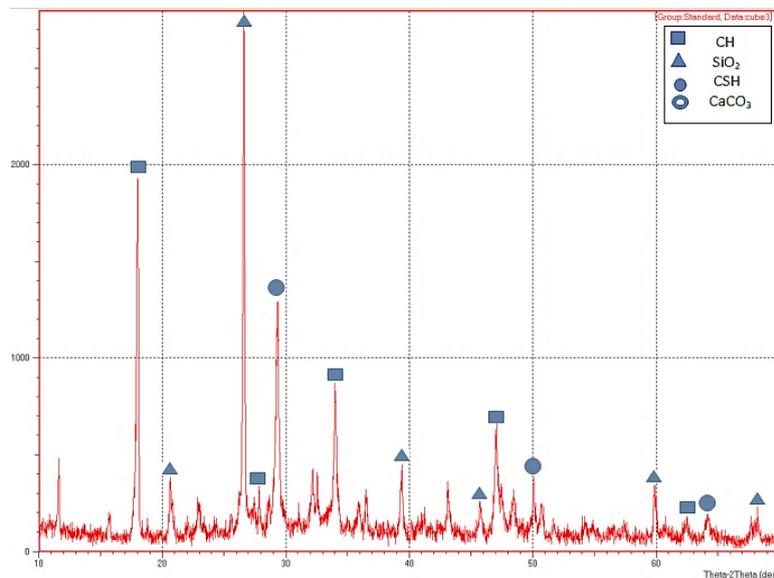


Figure 6. The characterization measurement (XRD) pattern of reference mortar mix

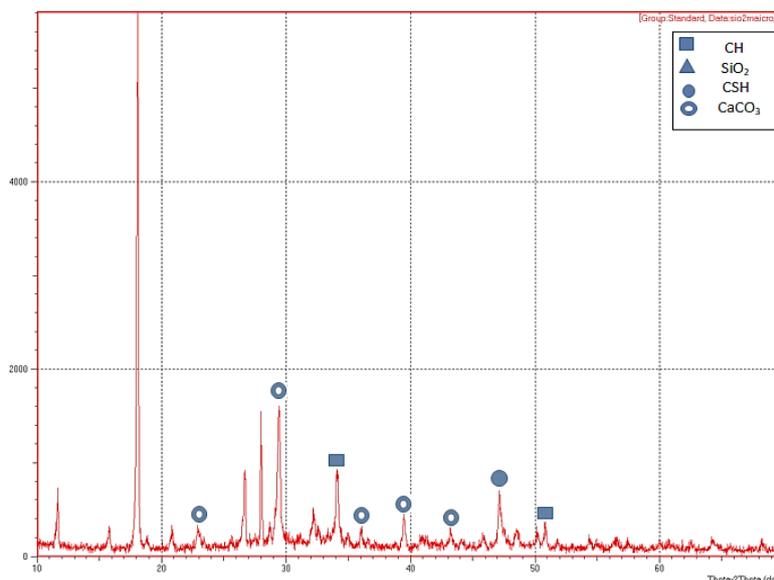


Figure 7. The characterization measurement (XRD) pattern of mortar mix with silica fume

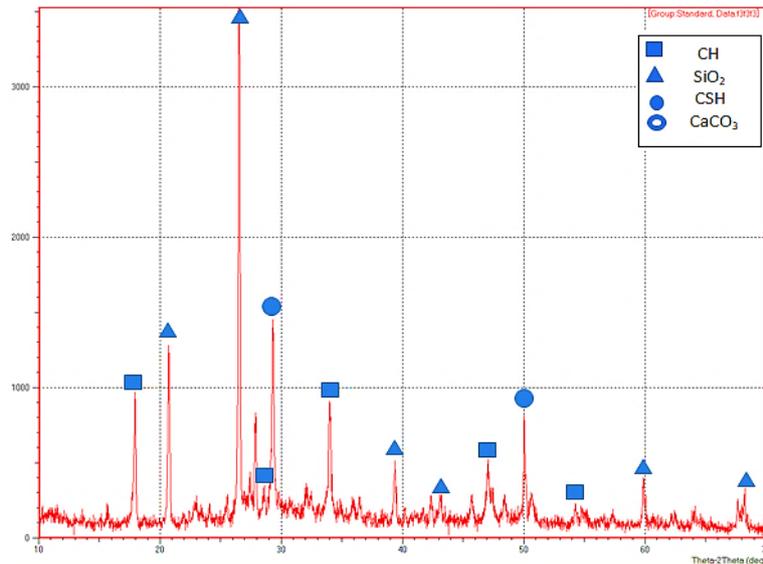


Figure 8. The characterization measurement (XRD) pattern of mortar mix with fly ash

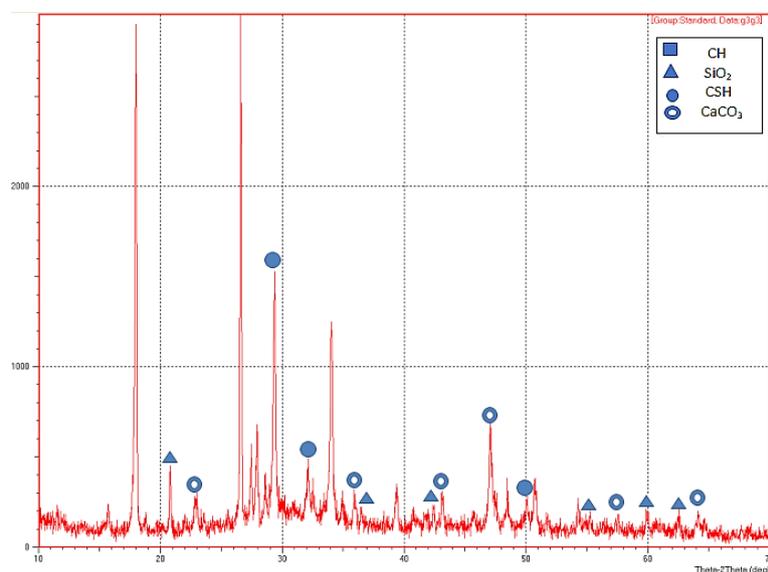


Figure 9. The characterization measurement (XRD) pattern of mortar mix with glass powder

4. Conclusions

The following conclusions are drawn from the obtained experimental study:

- Silica fume, fly ash and glass powder may be considered as a critical cement replacement admixtures in building up the physical strength of hardened cement paste.
- Silica fumes considered to be a proper admixture added to improve early and later strengths of mortar, furthermore present significant improvements to the compressive strength compared with fly ash and glass powder.
- The presented results obtained using (SEM) and (XRD) manifested the reasonable chance for observing the variations in the maturing cement pastes microstructure depending upon the differences in the morphology of the main phases arising throughout the cement paste hydration which help in predicting the properties of cement mortars.
- In the (SEM) micrographs, the dominant phases present in microstructure were Portlandite (CH), (CSH), and ettringite, which considered the evidence of hydration process, it was clear that the microstructural behaviour of mortar influences the strength characteristics of the mix.
- The XRD results elucidated that the addition of silica fume, fly ash and glass powder effects on the hydration product quantity and distribution.

5. Conflicts of Interest

The authors declare no conflict of interest.

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