International Journal of Material Science & Engineering – IJMSE
Volume 1 : Issue 1
Publication Date : 09 January 2014

Calcium Carbonate Efflorescence on Pozzolanic Modified Mortar

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***Abstract*—This study focused on the effectiveness of Fly Ash (FA) and Silica Fume (SF) as cement replacements in reducing efflorescence on the surface of Ordinary Portland Cement (OPC) mortar. Previous studies have shown that FA and SF can potentially enhance the performance of cement mainly due to their reaction with calcium hydroxide (C-H) to develop more of the strength-carrying compound in cement structure namely calcium silica hydrate (C-S-H). This pozzolanic reaction is crucial in the mitigation of efflorescence by its relation to C-H leaching. The early hydration behavior of Pozzolanic Modified Mortar (PMM) that hypothetically affects efflorescence has been investigated through physicochemical characterization namely Puddle Test (PT), Standard Chemical Method (SCM), Compressive Strength Test (CS) and Scanning Electron Microscopy (SEM). PMM mortar samples were prepared with water-to-cement ratio (w/c) of 0.3, 0.4 and 0.5. FA and SF were used as 10%, 20% and 30% cement replacement by weight. Samples were cured at room temperature (32^{o} C) and 90% relative humidity. Powdered and polished samples were prepared and tested at 28 days of hydration. Compared with conventional mortar, PMM exhibited lesser efflorescence whereby validation from CS and SEM images showed the evidence of pozzolanic reaction.** **Keywords— Efflorescence, Fly Ash, Silica Fume, Mortar**

I. Introduction

Efflorescence as shown schematically in Fig. 1, is a deposit of crystalized calcium carbonate ($CaCO_3$) on the exposed concrete and cementitious materials manifesting from hazy white layers to thick white crusts [1, 2].This manifestation as shown in Fig 2, is caused primarily by the leaching of Calcium Hydroxide (C-H) or Portlandite, one of Portland cement hydration products which is slightly soluble in water. It migrates to the concrete surface through the capillary system of the concrete and evaporates to leave the solid C-H which then reacts with atmospheric carbon dioxide (CO_2) to form $CaCO_3$[1,2,3,4,5].

Despite the fact that aesthetic problem is more obvious on coloured surfaces than on the grey ones, and causes economical implication due to products rejection by customers, efflorescence is indirectly related to durability problem in a way that the leaching occurred within the concrete can cause an increase in porosity, increase in permeability and decrease in strength, thereby increases its vulnerability to aggressive chemicals ingress [7, 8].

This phenomenon has received much description and discussion in the literature, not to mention speculation to its causes and prevention. However the concern of this study was the in depth understanding of the underlying mechanism that may lead to creating solution that can mitigate its occurrence and by extension minimize the previously mentioned implication [3, 5]. To date there is no effective method that can guarantee the prevention of efflorescence [6, 7, and 8]. Since C-H leaching is the main cause of efflorescence therefore theoretically, efflorescence can be minimized by minimizing C-H leaching through pozzolanic reaction. Therefore, it is the motivation of this study to investigate how these cement replacements affect efflorescence. Moreover, there are limited data available in the study of the microstructural interaction of Fly Ash and Silica Fume as Pozzolanic Modified Mortar (PMM) that relate the reduction of efflorescence. Hence the purpose of this study was to investigate the effect of PMM on efflorescence by focusing on finding the optimal level of cement replacement that may mitigate efflorescence and the morphology of PMM in comparison to conventional mortar.

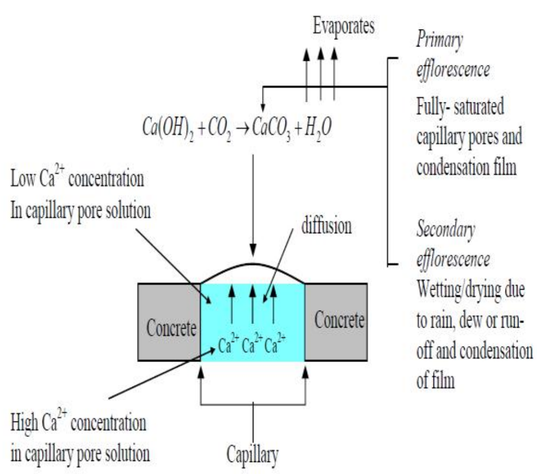


Figure 1: Schematic diagram of Efflorescence from cross sectional view of concrete block

Figure 2: Efflorescence on concrete wall

II. Materials and Methods

A. *Materials*

The two types of pozzolanic cement replacements chosen for this research were Fly Ash (PFA) Class F according to ASTM C 618, Standard Specification for Fly Ash, and Silica Fume (SF) according to ASTM C 1240 .Cement used was Ordinary Portland Cement (OPC) (ASTM Type 1 recognized by ASTM C150) manufactured by Cahaya Mata Sarawak Cement Sdn. Bhd (CMS) and it exceeded the quality requirements specified in the Malaysian Standard MS 522: Part 1: 1989 Specifications for OPC. The chemical and mineralogical characteristics of the OPC binder are given in Fig. 3. Table 1 shows the chemical and physical properties of Fly Ash Class F and Silica Fume.

To study the effect of PMM on efflorescence, comparative physicochemical analyses were performed using Puddle Test (PT), Standard Chemical Method (SCM), Compressive Strength Test (CS) and Scanning Electron Microscope (SEM). The mix proportion was set at 1:0.4:1.67 for all samples that were casted into Universal Container 30ml, 28 X 85mm for PT, SCM and SEM, and 150 mm X 150 mm X 150 mm cubes for CS test. All samples were dry-cured in the concrete laboratory with average temperature (T) of 32^{o} C and average relative humidity (RH) of 90%. PMM mortar samples were prepared with water to cement ratio of 0.4 and 10%, 20% and 30% of FA and SF cement replacement by weight.

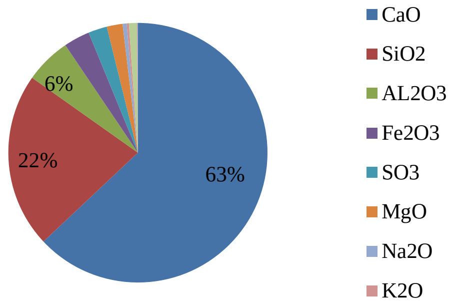


Fig 3: Chemical Composition of OPC

Table 1: Chemical Composition of Fly Ash Class F and Silica Fume

Composition (%)	CaO	SiO_2	Al_2O_3	Fe_2O_3
Fly Ash Class F	5.0	52	23	< 1
Silica Fume	< 1	97	-	-

B. *Test methods*

Efflorescence Tests

PT and SCM were performed at day 7, 14, 21 and 28. PT is an accelerated efflorescence test where distilled water of 10 ml was added on samples surfaces in the form of circular drops [1,2].The water in the circle could vaporize or be absorbed by the samples. On specified day, the surface of the samples was scraped to obtain powder of 1gram in weight. SCM was used to quantify the amount of $CaCO_3$ from the extracted powder by dissolving it in a diluted hydrochloric acid solution. The dissolved salts was then placed on a filter paper and weighed before it was oven dried for 24 hours at temperature between 90°C-100°C. Then the sample was taken out and weighed again. The weight loss indicates the amount of the dissolved efflorescence formed on the mortar samples surface.

Morphology by SEM

Polished samples were prepared and analyzed using SEM at day 28. Acetone was used to stop the hydration process of these samples. SEM images for all prepared samples were captured by Scanning Electron Microscope (JSM-6701F) supplied by JEOL Company Limited, Japan that followed the ASTM C 1723-10 (2010) code of practice.

Compressive Strength Test

28-day CS test was performed according to BS 1881-116 (1983) on 150 mm cubes samples. It was used to determine the maximum compressive load that a sample can carry per unit area. The compressive strength gives the overall picture of the quality of concrete.

III. Results and Discussions

Fig 4 to 6 show the efflorescence intensity in terms of percentage of $CaCO_3$ collected from the surfaces of FA and SF modified mortar samples in comparison with unmodified mortars (Control) of 0.4 w/c ratio for 7, 14, 21 and 28 days.

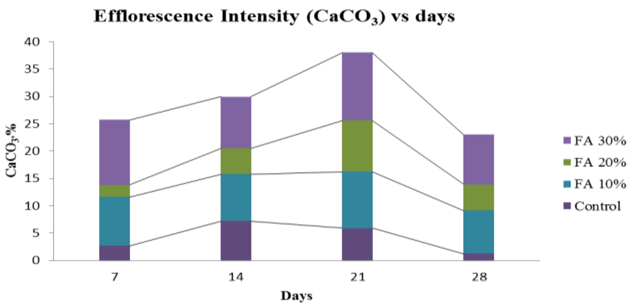


Figure 4. Percentage (%) of CaCO3 versus days for FA modified mortars and Control

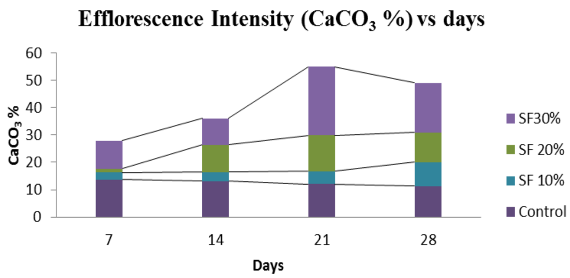


Figure 5. Percentage (%) of CaCO3 versus days for SF modified mortars and Control

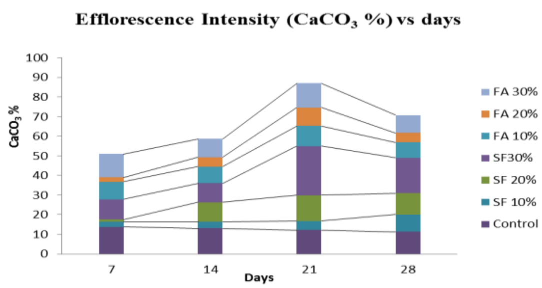


Figure 6. Comparison of Percentage (%) of $CaCO_3$ versus days for FA and SF modified mortars with Control

Based on Fig. 4, the low efflorescence intensity in terms of percentage of $CaCO_3$ in comparison to control can be found from 10% SF and 20% FA samples, whereas the high efflorescence intensity were given by 30% SF and FA replacements. These are the expected results since previous studies have confirmed that mortar contains FA or SF as cement replacements can produce denser and relatively watertight microstructure which is preferable in mitigating efflorescence [9, 10]. Furthermore, FA and SF mortar can reduce porosity and have better durability properties due to densification of interfacial transition zone. This is due to the consumption and the reduction in amount of calcium hydroxide (C-H) as pozzolanic reaction by FA and SF particles to form additional calcium silicate hydrate CSH gel which contribute to the densification of interfacial transition zone and lesser porosity of the matrix [11, 12, 13].

Fig 5 and 6 show that efflorescence intensity was reduced as hydration period increased especially after 21 days. In other words, longer hydration period produced denser microstructure at the later age with the reduction of the amount of calcium hydroxide (C-H) and the increase of Calcium Silicate Hydrate (CSH). Fly ash in mortar samples generally retards the rate of hydration thus the development of dense microstructure is not yet completed at earlier age [13]. Previous studies also showed that concrete with silica fume generally exhibits better performance of properties and tends to produce denser interfacial transition zone compared to concrete with fly ash. However, results showed in Fig 6 illustrates that it gave poorer performance compared to control and FA samples.

This phenomenon shows the ability of silica fume to densify interfacial transition zone, accelerate hydration and consume C-H for formation of additional CSH was not enough to compensate for the deficiencies in water content for the hydration process. As suggested in the previous studies, fly ash and silica fume with very fine particle can reduce the void space and correspondingly the water requirement for plasticizing the system. Despite that, with regard to micro fine filler such as silica fume which consists of very large proportions of very fine particles, the filler particle must be well dispersed with the aid of plasticizing agent before any enhancement properties from the effect of increase particle packing can be materialized. Hence, incorporation of silica fume requires usage of superplasticizer or water reducing admixtures to its very fine particle [13]. There was no superplasticizer used in this experimental works so there was lack of plasticizing agent to disperse the micro fine particles. Furthermore, the reduction in water content also gave rise to poor performance of silica fume modified mortar because the dry silica fume would absorb certain amount of water content and lower the available water content for cement hydration process. The water absorption is higher on silica fume particles compared to on fly ash particles due to high fineness of silica fume particles (higher specific surface area).

During mixing and compaction procedure, mortar samples with SF exhibited abnormal dryness and sandy surface condition. Even though additional mixing water was added but the sample was still unable to reach certain workability for better compaction effort and thus it produced mortar samples with porous surface. It might be due to very low mixing with water, and caused large amounts of unhydrated cement and high porosity of the mortar. These types of porous mortar samples can have excessive leaching or ingress of moisture or water and thus increased the severity of efflorescence. Therefore it is proposed to replace the cement with silica fume in term of volume, not in term of mass. The reduction of water content due to water absorbed by the dry surface of silica fume particles before mixing makes it needful to evaluate and adjust the water content in order for the mortar mixtures to have sufficient water content for hydration process. Super plasticizer or water reducing agent is recommended for mortar modified with silica fume to access the true potential of silica fume. Based on the observation of the mortar samples during puddle test, mortar sample with silica fume absorbed water very quickly. It can be extrapolated based on the observation during puddle test that, silica fume mortar have higher absorption rate compared to mortar samples with fly ash and control mortar sample. It was also found that, the higher percentage of cement replacement by silica fume, the faster the water was absorbed during puddle test due to porous surface of mortar sample. Fig.7 and 8 confirmed the findings by displaying the increase in compressive strengths of FA and SA and the evidence of pozzolanic activity by the presence of CSH in SEM images respectively.

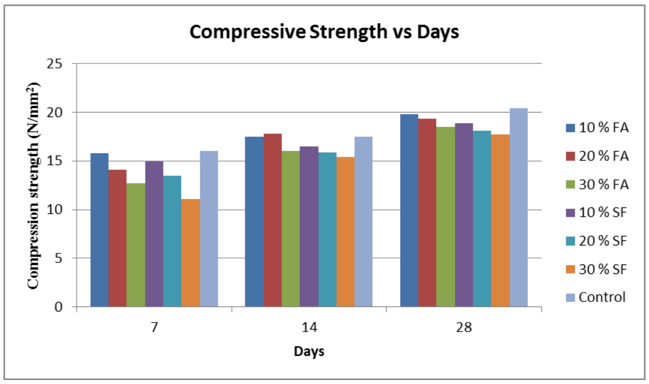
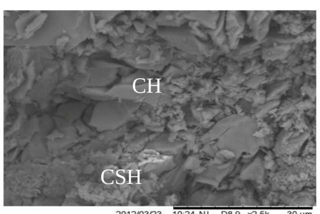
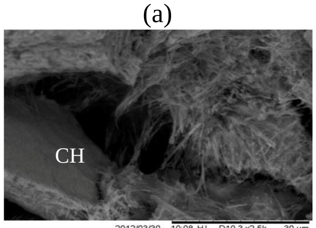


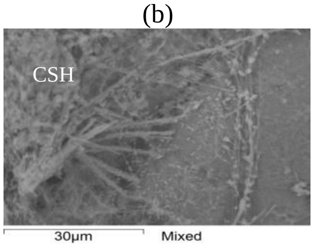
Figure7.Comparison of Compressive Strength versus days for PMM and Control



(a)



(b)



(c)

Figure 8: SEM images of (a) OPC, (b) 20% FA, (c) 10% SF at day 28

IV. Conclusion

From the PT and SCM investigations performed, it can be concluded that PMM has influenced the efflorescence formation on mortar surfaces to a significant extent through pozzolanic reaction in PMM. This behavior was reflected in the SEM images and the stronger CS results for PMM in comparison to conventional mortar. It was further validated that the optimum level of FA and SF replacement to mortar was 20% and 10% by weight respectively. Future investigation on the extent of reactivity of FA and SA with $Ca(OH)_2$ is needed to understand and solve efflorescence phenomenon and indirectly improve concrete durability and sustainability.

Acknowledgment

The research work reported in this paper has been funded by the Ministry of Higher Education Malaysia: (FRGS/03(04)/772/2010(53)) and University Malaysia Sarawak (SGS/03(S113)/894/2012(26)).

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