Malaysian Journal of Civil Engineering 26(2):198-208 (2014)

EFFLORESCENCE MITIGATION BY POZZOLANIC INDUSTRIAL BY-PRODUCTS

Norsuzailina Mohamed Sutan[1]*, Sinin Hamdan[2], Ibrahim Yakub[3] & Zainal Abidin Talib[4]

[1]*Department of Civil Engineering, Faculty of Engineering, Universiti Malaysia Sarawak, 94300 Kota Samarahan, Sarawak, Malaysia*
[2]*Department of Mechanical Engineering, Faculty of Engineering, Universiti Malaysia Sarawak, 94300 Kota Samarahan, Sarawak, Malaysia*
[3]*Department of Chemical Engineering and Energy Sustainability, Faculty of Engineering, Universiti Malaysia Sarawak, 94300 Kota Samarahan, Sarawak, Malaysia*
[4]*Department of Physics, Faculty of Science, University Putra Malaysia, 43400 Serdang, Selangor, Malaysia*

*Corresponding Author: *msnorsuzailina@feng.unimas.my*

Abstract: This study focuses on the effectiveness of pozzolanic industrial by-products namely Fly Ash Class F (FA) and Silica Fume (SF) as partial cement replacements in reducing efflorescence on the surface of Ordinary Portland Cement (OPC) mortar. The early hydration behaviour of Pozzolanic Modified Mortar (PMM) that hypothetically affects efflorescence has been investigated physically using Puddle Test (PT), Standard Chemical Method (SCM), and Electrical Conductivity Test (ECT); mechanically using Compressive Strength Test (CS); and morphologically using Scanning Electron Microscopy (SEM). FA and SF of 10%, 20% and 30% cement replacements as PMM and Unmodified Cement Mortar (UCM) samples were prepared with water-to-cement ratio (w/c) of 0.4. All samples were cured in the concrete laboratory at daily room temperature (T) and relative humidity (RH) in the range of 18-28^{o}C and 65-90%, respectively. Results showed 10%SF reduced efflorescence up to 52.9% in comparison to UCM. The decreased in ECT and the 12.64% increased strength evidently substantiated the EI results. Based on this study, more than 30% cement replacement is detrimental for efflorescence mitigation. It might be due to the lack of water content to initiate pozzolanic reaction because of the agglomeration of fine SF particles.

Keywords: *Efflorescence, fly ash, silica fume, mortar*

1.0 Introduction

Efflorescence is a deposit of crystallized calcium carbonate ($CaCO_3$) on exposed surfaces of cement based products (CBP) manifesting from hazy white layers to thick white crusts (Kresse,1987; Kresse, 1991). This manifestation as shown in Figure 1(a), is caused primarily by leaching of one of Portland Cement hydration products called Calcium Hydroxide (CH) or Portlandite. Figure 1(b) shows the rhombohedric morphology from SEM image of the manifestation which is typical of calcite, the most common species of $CaCO_3$. As shown schematically in Figure 2, free CH diffuses to the surface through capillary system of CBP and evaporates to leave solid CH which then reacts with atmospheric carbon dioxide (CO_2) to form $CaCO_3$ (Kresse,1987; Kresse, 1991; Higgins,1982; Neville,2002; Bensted,2000).

Despite the fact that efflorescence is regarded as aesthetic problem since its appearance is more obvious on coloured surfaces, and causes economical implication due to products rejection by customers, it is indirectly related to durability problem in a way that the excessive and long term CH leaching indicates and causes the increase in porosity, increase in permeability and decrease in strength, thereby increases its vulnerability to aggressive chemicals ingress (Kresse,1989; Dow,1989).

Figure 1:(a) Efflorescence on concrete wall (b) Scanning Electron Micrograph (SEM) image of efflorescence collected from the wall.

Since CH leaching is the main cause of efflorescence therefore hypothetically, efflorescence may be minimized by reducing CH through pozzolanic reaction. Pozzolanic reaction in a pozzolan modified cement system is the reaction between pozzolan with CH to produce more C-S-H gel (Kresse,1989). It consumes CH which lowers the amount the system and then lowers the permeability and porosity of the system. This is due to the extra C-S-H produced that hinders the transportation of the remaining CH.

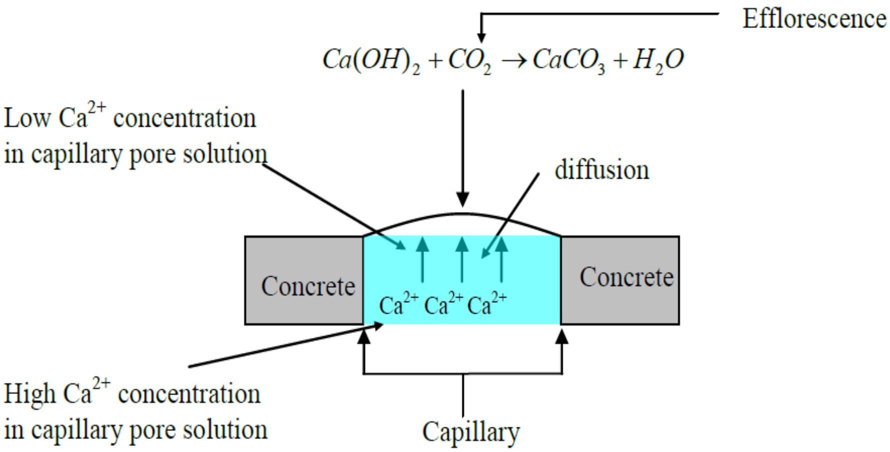

Figure 2: Schematic diagram of efflorescence from cross sectional view of concrete block.

Equation 1 and 2 show the behavioural difference between hydration of OPC system and pozzolan modified cement system with respect to the consumption of CH and the formation of extra C-S-H (Kollmann and Strubel,1979).

Hydration of OPC system:

$$\mathrm{C_3S + H \rightarrow C - S - H + CH} \quad (1)$$

Hydration of Pozzolan Modified Cement System:

$$\mathrm{Pozzolan + CH\ (from\ (1)) \rightarrow C - S - H} \quad (2)$$

Therefore, it is the motivation of this study to investigate how these pozzolanic cement replacements affect efflorescence. Moreover, there are limited data available in the study of the microstructural interaction of FA and SF as Pozzolanic Modified Mortar (PMM) that can be related to 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 pozzolanic cement replacement and the correlation between physical, mechanical and morphological properties with the phenomenon.

2.0 Materials and Methods

2.1 Materials and Samples Preparation

The two types of pozzolanic industrial by-products as cement replacements chosen for this study were Fly Ash Class F (FA) Class F according to ASTM C 618, Standard Specification for Fly Ash and Silica Fume (SF) according to ASTM C 1240. Their chemical and physical properties are shown in Table 1. Ordinary Portland Cement (OPC) (ASTM Type 1 recognized by ASTM C150) manufactured by Cahaya Mata Sarawak Cement Sdn. Bhd (CMS) was used. It's chemical and mineralogical characteristics are given in figure 3. To study the effect of PMM on efflorescence, comparative physicochemical analyses were performed using Puddle Test (PT), Standard Chemical Method (SCM), Electrical Conductivity Test (ECT), Compressive Strength Test (CS) and Scanning Electron Microscope (SEM). Mix proportions were set at (cement: sand: water) 1:1.67:0.4 by weight of OPC, natural sand, and water. The samples for PT and SCM were cylindrical (28mm diameter, 85mm height) from where small samples were extracted and polished for SEM analysis after 28 day. For EC and CS tests 100mm x 100mm x 100 mm and 150mm x 150mm x 150mm cubes samples were prepared, respectively. All samples were cured in the concrete laboratory of Universiti Malaysia Sarawak at daily room temperature (T) and relative humidity (RH) in the range of 18-28oC and 65-90%, respectively. PMM samples were prepared with water to cement ratio of 0.4 and 10%, 20% and 30% of FA and SF cement replacement by mass.

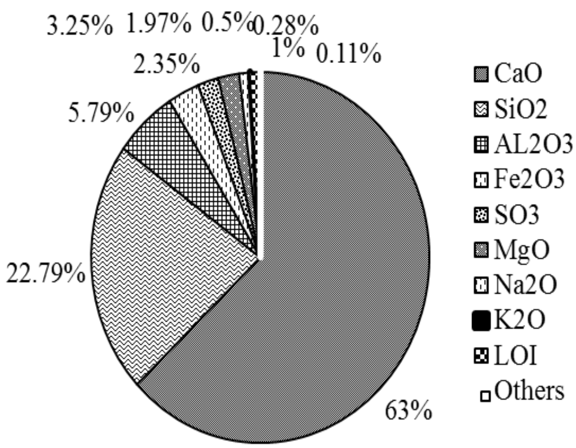

Figure 3: Chemical Composition of OPC

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	-	-

2.2 Test methods

2.2.1 Efflorescence quantification by PT and SCM

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 (Kresse,1987 ;Kresse,1991). The water in the circle could vaporize or be absorbed by the samples. On specified day, the surface of the samples was scraped to obtain1gram samples in powder form. SCM was then used to quantify the amount of CaCO3 from the extracted powder by dissolving the collected samples in a diluted hydrochloric (HCl) acid solution. The dissolved CaCO3 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 it was taken out and weighed again. The weight loss indicated the amount of the efflorescence formed on the sample surface.

2.2.2 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.

2.2.3 Physical property by ECT

ECT was performed on 100mm cubes samples on day 7, 14, 21 and 28. Conductivity test circuit for DC measurement is based on basic resistivity shown in Figure 4.

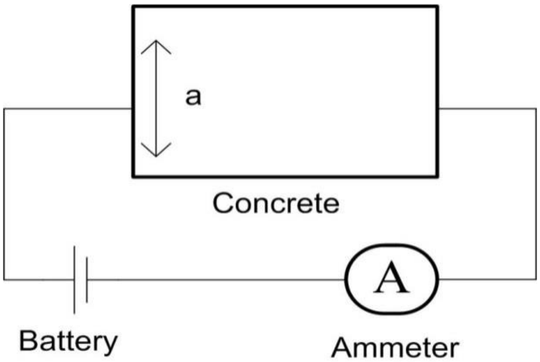

Figure 4: Basic Measure Resistivity

During testing, a low-frequency direct electrical current passes between the two electrodes and the voltage is measured. The electrical conductivity observed based on the resistivity value is

$$\text{Resistivity}, \rho = 2\pi aV/I \qquad (3)$$

Electrical conductivity is inversely proportional to the resistivity, where

$$\text{Conductivity} = 1/\rho = I/2\pi aV \qquad (4)$$

where a is the electrodes spacing, V is voltage drop, and I is the current. This equation is applicable for an identical semi-finite volume of material (Sengul, 2008).

2.2.4 Mechanical property by CT

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 CT results give the overall picture of the quality of concrete.

3.0 Results and Discussions

Figure 5 and Table 2 show the efflorescence intensity (EI) in terms of percentage of CaCO3 collected from the surfaces of FA and SF modified mortar samples in comparison to unmodified cement mortar (UCM) samples of 0.4 w/c ratio for 7, 14, 21 and 28 days. Generally, PMM manifested significantly lower EI in comparison to UCM.

10%SF reduced efflorescence up to 52.9% in comparison to UCM (%EI reduction= Total EI – UCM EI/ UCM EI) x 100 . This proved the hypothesis of this study that pozzolanic reaction in terms of reduction of CH to produce more C-S-H can significantly reduce efflorescence. This is due to the consumption and the reduction in the amount of CH to form additional C-S-H gel which contributes to the densification of interfacial transition zone and lesser porosity of the matrix (Toutanji,1995;Mehta,2004;Malhotra,1996). Furthermore, previous studies confirmed that mortar containing pozzolanic materials as cement replacements can produce denser and relatively watertight microstructure which is preferable in mitigating efflorescence (Tobon *et al.*,2010 ; Freeda *et al.*,2010).

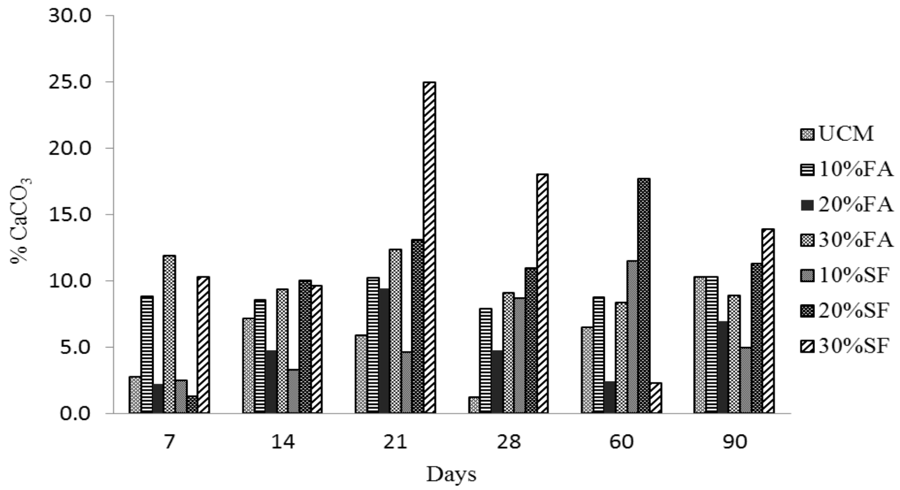

Figure 5:Comparison of Percentage (%) of $CaCO_3$ versus days for FA and SF modified mortars with Control

Table 2:.Efflorescence Intensity (EI) of 0.4w/c

		7	14	21	28	60	90	Total EI	% EI Reduction
SF	30%	11.9	9.4	12.4	9.1	8.4	8.9	79.2	-203.4%
	20%	2.6	10.0	13.1	11.0	17.8	11.3	65.8	-152.1%
	10%	1.3	1.2	2.1	2.4	2.6	2.7	12.3	52.9%
FA	30%	11.9	9.4	12.4	9.1	8.4	8.9	60.1	-130.3%
	20%	2.2	4.7	9.4	4.8	2.4	6.9	30.5	-16.9%
	10%	5.4	4.1	9.7	7.9	10.6	6.2	43.9	-68.2%
	0.4w/c	2.7	4.2	5.9	2.7	6.5	4.1	26.1	
		36.5	43.3	77.6	55.9	50.6	54.1	317.9	

To further substantiate PT and SCM results, ECT results, related to permeability and diffusivity of ions through porous materials, were used (Nagi,2004). The reduction of the capillary pores content of CH through the pozzolanic reaction of SF influences the conductivity of hardened concrete by reducing it (Doran,1992; Brameshube,2003). In addition, water within the pore becomes saturated with Ca^{2+} and OH^{-} ions making electrical conductivity to be increased (Abo *et al.*,1995). Futhermore,chemistry of pore solution and pore structure affect the electrical conductivity of water saturated concrete in which is related to the CH ions as a considerable conductive ion in pore solution (Shi,2004). Therefore, ionic movement of CH in cement matrix of 10%SF, 20%FA, 30%SF and UCM can be quantified using electrical conductivity as shown in Figure 6.

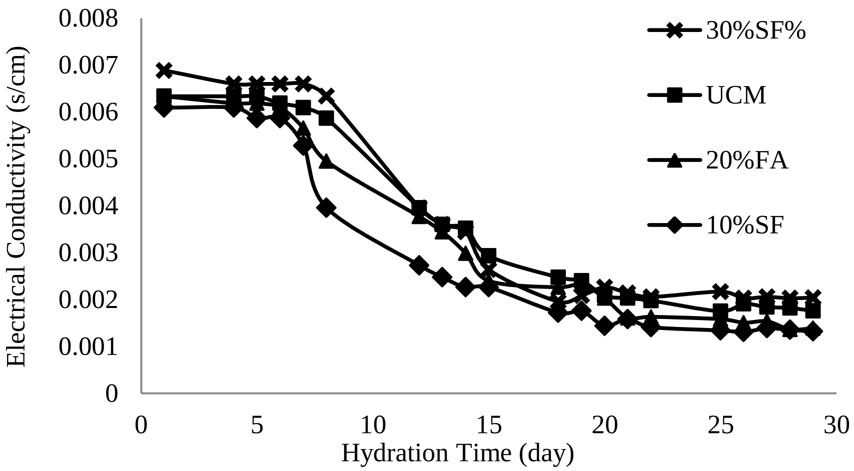

Figure 6:Electrical Conductivity of Mortar of PMM and UCM samples.

It can be observed that 10% SF has the lowest conductivity in comparison to 20%FA, 30%SF and UCM. 30%SF has the highest conductivity which is reflected on its worst EI .This is due to the increase in ionic concentrations and mobility of these ions as more water exists within the pores. PT, SCM and ECT results were in correlation with CS test results as the mechanical property of samples.

Figure 7 shows CS test results for 10%SF, 20%FA, and 30%SF samples in comparison to UCM sample. The highest strength was achieved by 10%SF sample (23.0MPa) at day 28 which is 12.64% improvement from UCM. This result substantiates EI and ECT results.

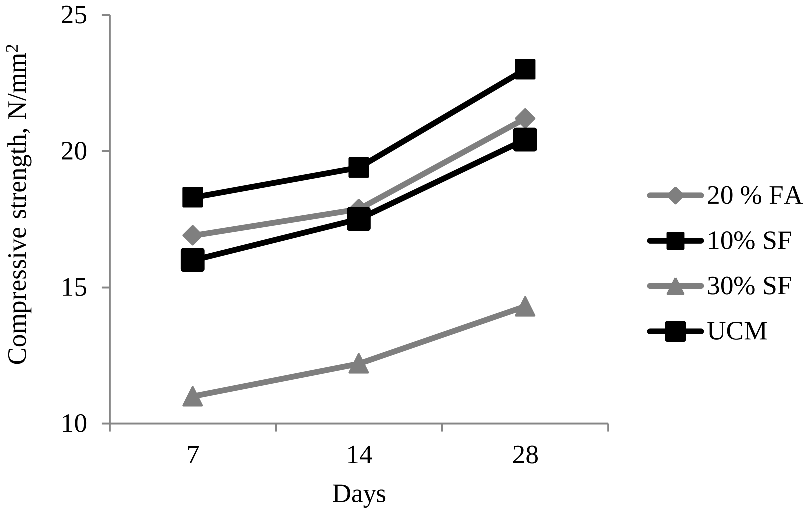

Figure7:Comparison of CS versus days for PMM and UCM

Since CH and C-S-H are the components of pozzolanic reaction and the main contributors to efflorescence, SEM morphological analysis was used to further substantiate the results from PT, SCM, ECT and CS test. SEM images shown in Figure 8, focus on the presence of CH and C-S-H that are related to EI of the respective samples. By referring to Figure 8(a), CH crystals solid hexagonal plate-like morphology are found in the sample as it is the by-product from the cement hydration besides the abundant presence of C-S-H flowery-like morphology (Malhotra *et al.*, 2001). It is observed in Figure 8(d) the SF particles agglomeration and existence of pores hence the high EI due to large capillary pores. Furthermore, evidence of pozzolanic interaction between CH and SF producing C-S-H can be observed by abundant presence of C-S-H flowery-like morphology in Figure 8 (b) and (c) for 10%SF and 20%FA which are also reflected in low EI due to reduction of CH and its pathways to effloresce (Malhotra *et al.*,2001).

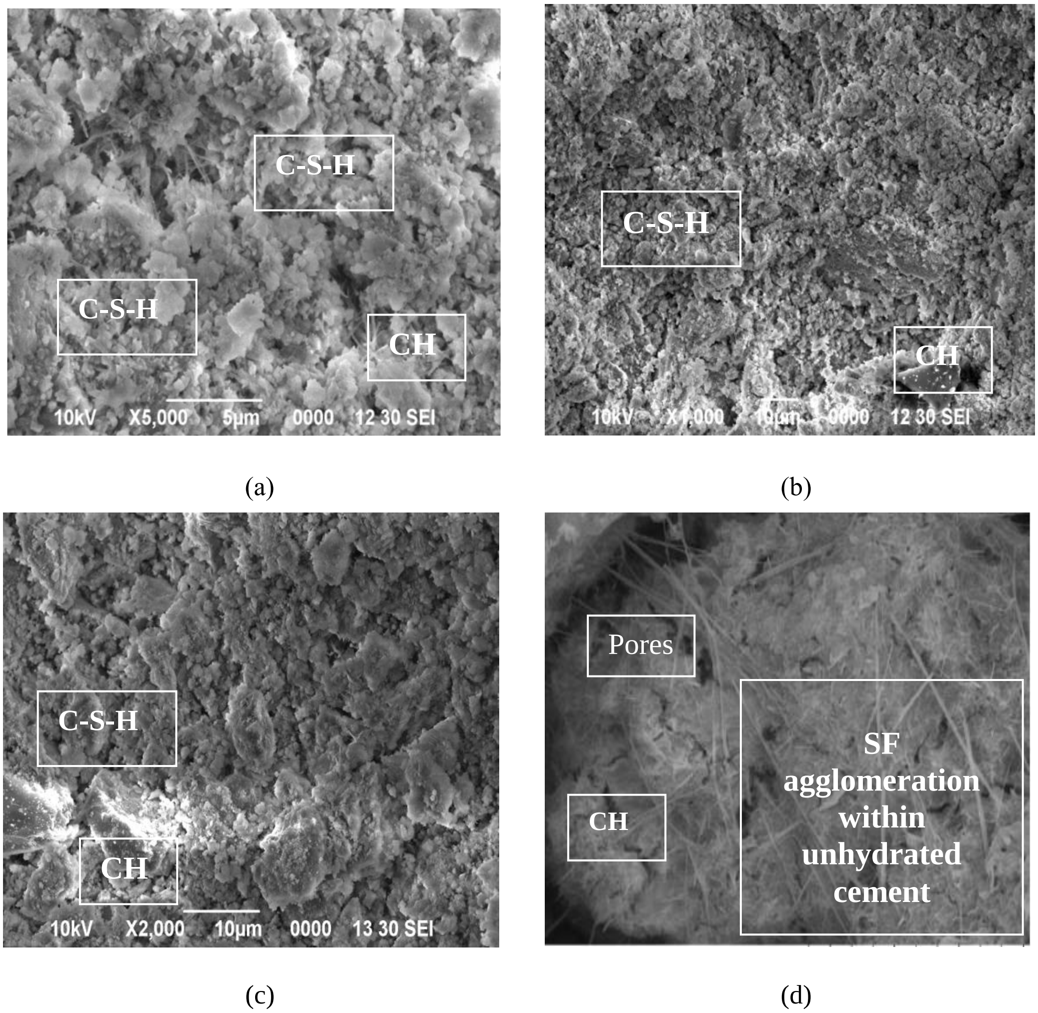

Figure 8: SEM images of (a) UCM (b) 20%FA and (c) 10%SFand (d) 30%SF samples at day 28

4.0 Conclusions

In general, PMM manifested significantly lower EI in comparison to UCM. Specifically, 10%SF reduced efflorescence up to 52.9% in comparison to UCM proving the hypothesis of this study that pozzolanic reaction can significantly reduce efflorescence by consumption of CH to produce more C-S-H that block the pathways of CH to effloresce. The physical and mechanical properties in terms of electrical conductivity and compressive strength of UCM were extensively improved with 10%SF cement replacements. The decreased in electrical conductivity and the 12.64% increased strength evidently substantiated the EI result. More than 30% cement replacement is detrimental for efflorescence mitigation. It might be due to the lack of water content to initiate pozzolanic reaction because of the agglomeration of fine SF particles.

5.0 Acknowledgements

The research work reported in this paper has been funded by the Ministry of Higher Education Malaysia: (FRGS/03(04)/772/2010(53) and ERGS/TK04(02)/1011/2013(08))

References

Bensted, J.(2000) Efflorescence-prevention is better than cure, Concrete, 34:40-41.

Brameshuber, W. and Raupach, M. (2003). Non-destructive Determination of the Water-Content in the Concrete Cover using the Multiring-Electrode ,Part I: Aspects Of Concrete Technology. International Symposium (NDT-CE 2003) Non-Destructive Testing in Civil Engineering..

Doran, D. K. (1992) Construction Materials Reference Book London ; Boston : Butterworth-Heinemann.12-34

Dow,C.& Glassier, F.P.(2003) Calcium carbonate efflorescence on Portland cement and building materials, *Cement and Concrete Research*, 33:147-154.

Freeda C. & Christy T. (2010) Effect of class-f fly ash as partial replacement with cement and fine aggregate in mortar, International Journal of Engineering & Materials Sciences, 17:140-144.

Higgins,D.D.(1982) Appearance Matters (No.4.).Cement and Concrete Association.

Kresse, P. (1987) Efflorescence-Mechanism of Occurrence and Possibilities of Prevention, Betonwerk+Fertigteil-Technik,53:160-168.

Kresse,P. (1991) Efflorescence and its prevention, *Betonwerk+Fertigteil-Technik*, 57: 84-88.

Kresse,P.(1989) Coloured concrete and its enemy: efflorescence. Chemistry and Industry, 93-95.

Malhotra, V.M. & Mehta, P.K. (1996) Pozzolanic and cementitious materials: Advances in Concrete Technology. , London and New York Taylor & Francis, pp. 4-5

Mehta, P. K. (2004) International Workshop on Sustainable Development and Concrete Technology, 21(11): 47-52.

Nagi , M (2004) Resistivity of Concrete: State of the Art Construction Technology Labs.David Whiting, CTL Inc. CORROSION 2004, March 28 - April 1, 2004 , New Orleans, USA.

Neville, A (2002) Efflorescence-surface blemish of internal problem? Part 1: The knowledge,Concrete International,24.

Neville, A. (2002) Efflorescence-surface blemish of internal problem? Part 2: Situation in practice,Concrete International,24.

Tobon, J.I.,Restrepo,O. & Paya,J.(2010) Comparative analysis of performance of portland cement blended with nanosilica and silica fume, Dyna, 33:37-46.

Toutanji, H. A. & El-Korchi, T. (1995) The influence of silica fume on the compressive strength of cement paste and mortar, Cement and Concrete Research, 25 (7):1591-1602.