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Influence of Na_2SO_4 on the Mechanical Properties of High Volume Calcined Clay Pozzolan Cement

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Abstract: Calcined clay pozzolan, the major mineral admixture used in Ghana to partially substitute ordinary Portland cement has gained massive recognition in the local construction industry because of its technical advantages and affordability. The most prominent undesirable property associated with the use of calcined clay pozzolans is slow setting and strength development, especially at early ages. This paper presents a study of the effect of Na_2SO_4 on the mechanical properties of high volume pozzolan cements. The cements containing 30 wt. %, 40 wt. %, 50 wt. % and 60 wt. % calcined clay pozzolan was activated with 1%, 2%, 3% and 4% Na_2SO_4. Some physical properties such as Blaine index, specific gravity, water demand and setting times were determined. The chemical compositions of the blended cements were also analyzed. The effect of Na_2SO_4 on compressive strength of blended cements was determined after 2, 7, 28 and 180 days. Durability studies on activated blended cements in aggressive media were also conducted. Activation with a minimum of 2% Na_2SO_4 improved the 2 days compressive strength of cement containing 60% calcined clay pozzolan by 66.2% and caused it to behave as Class 32.5N cement, at 28 days. As Na_2SO_4 dosage increased, compressive strength also increased. The optimum Na_2SO_4 dosage was 2%. Activated blended cements resisted $MgSO_4$ environment better than unactivated cements.

Keywords: Pozzolan Cement, Activation, Admixture, Durability, Compressive Strength, Setting Time

1. Introduction

The incorporation of supplementary cementing materials to generate specific technical properties in concrete has become a common practice throughout the world. These materials may be naturally occurring, industrial waste or by-products or specially manufactured using less energy. Pozzolans are used as a partial replacement in ordinary Portland cement up to 40% in concrete and masonry works for constructional applications [1, 2, 3]. They are sometimes preferably used for construction because of their resistance to alkali-aggregate reaction, improved durability due to resistance to sulfate attack, improved workability, and lower cost among others [4, 5, 6].

Calcined clay pozzolan, the major mineral admixture used in Ghana to partially substitute ordinary Portland cement has gained massive recognition in the local construction industry because of its technical advantages and affordability. Research on various Ghanaian clay deposits [7] has shown that clay pozzolans when milled to cement fineness can replace up to 30% of ordinary Portland cement in structural applications [8]. These clay pozzolan cement mixes have been successfully used for various housing construction projects in Ghana [9].

The most prominent undesirable property associated with the use of calcined clay pozzolans is slow setting and strength development, especially at early ages. These undesirable properties rise from the slow reaction rate of the active pozzolan constituents with the liberated $Ca(OH)_2$ from the Portland cement [10]. Methods such as mechanical [11], thermal [12] and chemical activation [13, 14] have been proposed by researchers to accelerate and improve strength development and setting of cements containing pozzolans. Shi and Day [15] in their comparison of different methods of enhancing the reactivity of pozzolans have reported that chemical activation improves strength of cements better than thermal and mechanical activation. Use of chemical activators changes hydration products and accelerates pozzolanic reaction, which leads to faster strength developments and higher ultimate strength. Although the addition of activators increases the cost of raw materials, the cost per unit strength increases. Numerous investigators have utilised chemical activation to activate fly ash systems. Two different methods commonly utilised include alkali activation and sulfate activation [16].

Alkali activation involves the breaking down of the glass phases in an elevated alkaline environment to accelerate the reaction [17]. Sulfate activation is based on the ability of sulfates to react with aluminium oxide in the glass phase of fly ash to form sulfates (AFt) that contribute to strength at early ages [18, 19]. The possibility of fly ash activation mainly lies in the breaking down of its glassy phases. The addition of 4% Na_2SO_4 increased both early and later strength of lime-pozzolan cement pastes from 23°C to 65°C [20]. When 30% cement was replaced with coal fly ash, the addition of 3% Na_2SO_4 increases strength by approximately 40% from 3 to 28 days. However, as fly ash replacement level is increased from 30 to 70%, the addition of 3% Na_2SO_4 increases strength by approximately 80% from 3 to 28 days. This means that the use of Na_2SO_4 is particularly effective with high fly ash replacement [19].

This paper presents a study of the effect of Na_2SO_4 on the mechanical properties of high volume calcined clay pozzolan cement.

2. Materials and Methods

2.1. Materials Preparation

Calcined clay pozzolan used for this study was obtained from the pozzolan plant at CSIR-Building and Road Research Institute, Kumasi, Ghana and conformed to ASTM C-618 [21]. A Class 42.5N Ordinary Portland cement (OPC), conforming to EN 197-1 [22], manufactured by Ghacem, the leading cement producer in Ghana was used as the reference cement for the preparation of the mortar and paste specimens. The OPC was intimately blended with 30 wt. %, 40 wt. %, 50 wt. % and 60 wt. % calcined clay pozzolan to produce Portland pozzolan cements labeled P_{30}, P_{40}, P_{50} and P_{60}. Laboratory grade Na_2SO_4 was used as the activator.

2.2. Methods

All experiments were carried out at the Building and Road Research Institute Laboratories, Kumasi, Ghana. The specific gravity of the OPC and blended cements were determined using the BS 1377:90 [23] standard method. Blaine indices were also conducted by the air permeability method as specified by ASTM C 204 [24]. The chemical compositions of the blended cements were determined by the X-ray fluorescence equipment (Spectro X-lab. 2000). Reagent grade Na_2SO_4 was used to activate the pozzolan blended cements at percentages of 1, 2, 3 and 4. The setting times and water demand were also determined using the Vicat apparatus according to EN 196-3 [25].

Ordinary pit sand satisfying BS 4550-6 [26] was used to prepare the mortar. Tests for the determination of the compressive strength of the activated pozzolan cement mortar cubes were carried out according to methods specified by EN 196-1 [27]. The mortar cubes were prepared using a water-to-cement ratio of 0.5 and a cement-to-sand ratio a 1:3. Measured quantities of the blended cement and sand were mixed with portable water containing the Na_2SO_4 in a motorized mixer and placed in 70mm × 70mm steel moulds. It was then vibrated on a vibrating table for 4 mins. Curing of cubes was done in a moisture cabinet and compressive strength test conducted on them after 2, 7, 28 and 180 days. Another set of mortar cubes were immersed in 5% $MgSO_4$ solution and the effect of the solution on the compressive strength determined after 180 days.

3. Results and Discussion

3.1. Physical Properties

The specific gravity, Blaine indices and water demand of the reference cement blended cements have been presented in Table 1. It is seen from Table 1 that ordinary Portland cement (OPC) recorded the highest specific gravity of 3.18 whereas clay pozzolan (CP) recorded the least value of 2.67. It is observed that specific gravity of the blended cements decreased as the calcined clay pozzolan content in the cement increased ranging from 2.92 to 2.53. On the other hand, OPC obtained the least Blaine index of 338m^2/kg whereas calcined clay pozzolan recorded the highest of 442m^2/kg. Also, the Blaine indices of the blended cements increased as the pozzolan content increased from 30% to 60%. This indicates that the surface area of the mix increased as the pozzolan content increased. A higher surface area is a good indication of higher reactivity of pozzolan and cement constituents.

Table 1. *Some physical properties of OPC, CP and blended cements.*

Property	OPC	CP	P_{30}	P_{40}	P_{50}	P_{60}
Specific gravity	3.18	2.67	2.92	2.82	2.75	2.53
Fineness, m^2/kg	33.8	442	420	432	445	453
Water demand, %	28.5	–	35.6	36.1	38.2	38.8

Water demand to form a workable paste increased with increasing pozzolan content. This progressive increase in water demand is due to the smaller particle size of the pozzolan which increases the surface area of the whole mix translating into higher water demand [28]. Therefore, as the pozzolan content increases, the surface area becomes greater thereby requiring greater volume of water to form a workable paste.

The activation of the blended cements with Na_2SO_4 had no effect on the water demand. The water demand of the mixes remained unchanged at all dosage levels.

The initial and final setting times of the pozzolan cement increased significantly because of the incorporation of large volumes of pozzolan. Addition of Na_2SO_4 accelerated both initial and final setting times of the blended cement pastes. As pozzolan content in the mix increased, higher dosages of Na_2SO_4 was required to reduce the setting times.

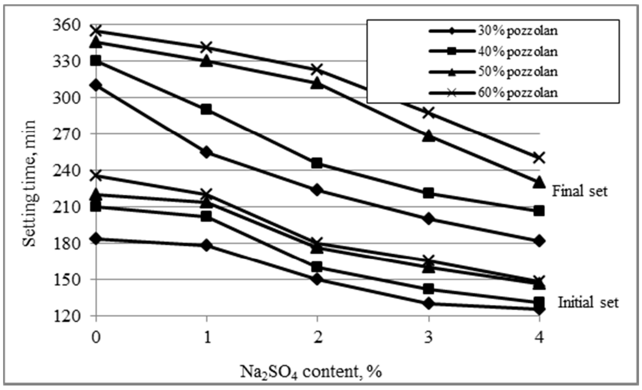

Figure 1. *Setting times of blended cements activated with different percentages of Na_2SO_4.*

3.2. Chemical Composition

The results of the chemical analysis of OPC, calcined clay pozzolan and blended cements as determined by X-ray fluorescence (XRF) have been presented in Table 2. The composition of the cements shows the blended cements are chemically suitable.

Table 2. *Chemical composition of OPC and blended cements.*

Constituents, %	OPC	Clay Pozzolan	P_{30}	P_{40}	P_{50}	P_{60}
SiO_2	18.88	62.77	18.37	16.53	15.07	19.05
Al_2O_3	3.57	18.71	2.65	4.93	4.58	4.13
Fe_2O_3	3.36	11.68	4.46	3.07	2.66	4.41
CaO	59.64	0.25	59.18	58.44	58.8	55.01
MgO	1.89	1.46	1.54	2.64	3.98	1.44
MnO	0.14	0.46	0.2	0.06	0.04	0.1
TiO_2	0.14	0.41	0.09	0	0.34	0.32
SO_3	4.93	0.19	3.29	3.06	3.23	3.44
Na_2O	4.7	0.21	0.15	0.09	0.24	0.12
P_2O_5	0.22	0.03	0.03	0.01	0.01	0.04
Cl	0.01	0	0.01	0	0.01	0
K_2O	2.12	1.08	0.55	0.66	0.62	0.72
L. O. I	3	2.75	9.5	10.5	11.1	12

P_{30} – 30% pozzolan cement; P_{40} – 30% pozzolan cement P_{50} – 50% pozzolan cement; P_{60} – OPC with 60% pozzolan cement

3.3. Compressive Strength

The compressive strength of 30% pozzolan cement (P_{30}) at 2 days was 16.1 MPa and decreased as pozzolan content increased to as low as 6.5 MPa at 60% replacement. This could be due to dilution effect and the slow reactivity of the pozzolan constituents and Portlandite from the OPC at early ages [29]. However, the activation of the mortar with 1% Na_2SO_4 caused 6.8%, 20.3%, 25.6% and 24.6% respectively increase in strength at all replacement levels. As Na_2SO_4 content in the mortar increased, compressive strength increased. The optimum Na_2SO_4 dosage was 2%, improving compressive strength of 60% pozzolan cement by 66.2% and causing it to pass the minimum standard for 2 days (10 MPa) by 8%. This is because at early ages, the introduction of Na_2SO_4 speeds up the consumption of $Ca(OH)_2$ in pozzolan-cement pastes [14]. Also, the ettringite formed at the early stage of hydration in pozzolan–cement pastes is increased with the addition of Na_2SO_4 since the SO_4^{2-} ions react with tricalcium aluminate, C_3A [30].

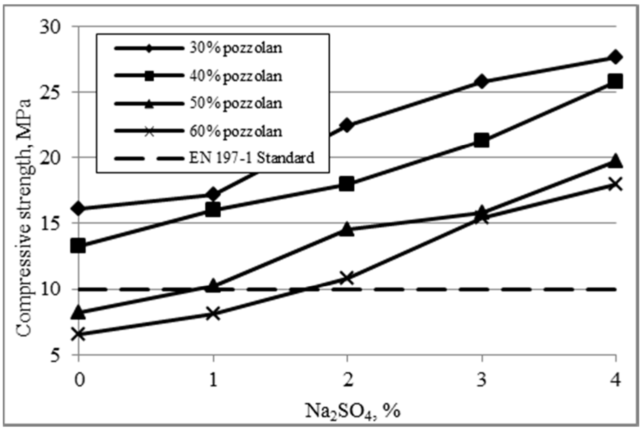

Figure 2. *2 days compressive strength of mortar cubes activated with different percentages of Na_2SO_4.*

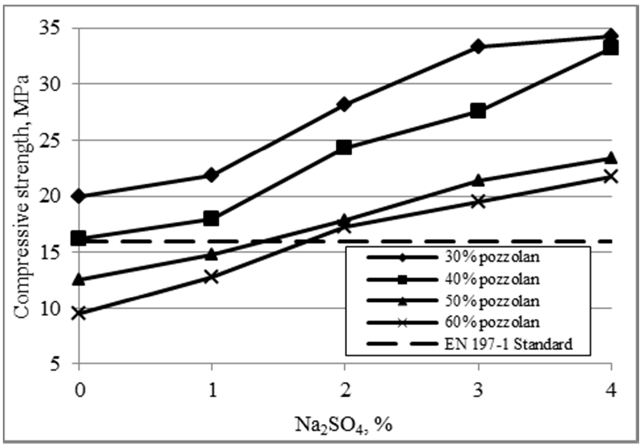

Figure 3. *7 days compressive strength of mortar cubes activated with different percentages of Na_2SO_4.*

Similarly, after curing for 7 days, compressive strength of activated mortar cubes increased with increasing Na_2SO_4 content. There was an 82.1% increase in strength when P_{60} was activated with 2% Na_2SO_4, exceeding the minimum EN 197-1 standard of 16MPa by 8.1%.

As curing period was extended to 28 days, apart from P_{30} which obtained a compressive strength of 39.0 MPa, all the other blended cements were short of the EN 197-1 minimum standard of 32.5 MPa. When activated with a minimum of 1% Na_2SO_4, P_{30} and P_{40} obtained strengths comparable to Class 42.5N and Class 32.5N cement respectively, whereas 2% Na_2SO_4 caused P_{50} and P_{60} to record strengths similar to Class 32.5N cement. The optimum dosage of Na_2SO_4 is 2%.

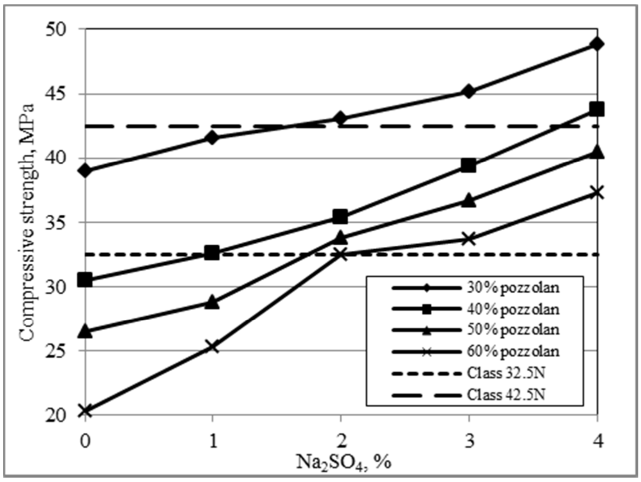

Figure 4. *28 days compressive strength of mortar cubes activated with different percentages of Na_2SO_4.*

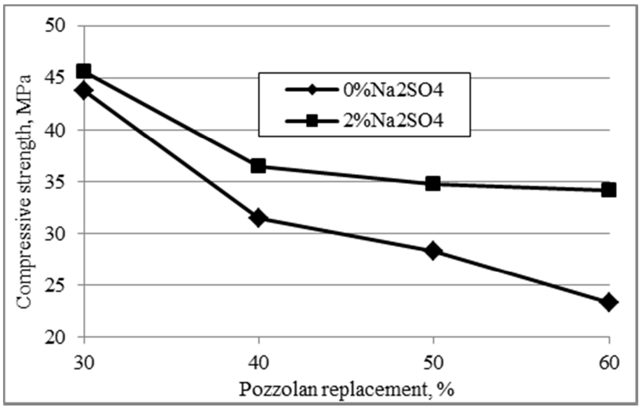

Figure 5. *180 days compressive strength of activated and unactivated mortar cubes.*

3.4. Durability

Figure 5 is the 180 days compressive strength performance of the blended cements. At 180 days, all the blended cements improved in strength. The higher strength at later ages is as a result of the pozzolanic reactions increasing the amount of calcium silicate hydrates (C-S-H) while diminishing $Ca(OH)_2$ [31]. There was however a further increase in strength for all blended cements when activated with the optimum dosage.

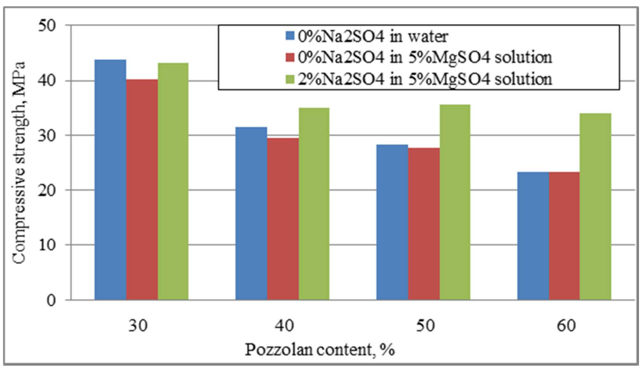

Figure 6. *180 days compressive strength of 2%Na_2SO_4 activated blended cements in aggressive media.*

Figure 6 is the compressive strength performance of activated and unactivated blended cements in an aggressive medium for 180 days. There were 8%, 6.3%, 2.5% and 1.3% respective reductions in strength when the unactivated blended cements were immersed in 5% $MgSO_4$ solution. There was however an improvement in compressive strengths when the blended cements were activated with the optimum dosage of Na_2SO_4. P_{50} and P_{60} were more resistant to the $MgSO_4$ solution than P_{30} and P_{40}. This could be due to the high fine pozzolan content [32].

4. Conclusions

Addition of Na_2SO_4 accelerated both initial and final setting times of the blended cement pastes. As pozzolan content in the mix increased, higher dosages of Na_2SO_4 was required to reduce the setting times.

Activation of high volume calcined clay pozzolan with 2%Na_2SO_4 improved 2 days compressive strength by 66.2% causing it to pass the minimum EN 197-1 standard by 8%. At 28 days, pozzolan cement containing 50% and 60% pozzolan obtained strengths comparable to Class 32.5N cements. Early strength and ultimate strength were significantly improved with the addition of Na_2SO_4. As Na_2SO_4 dosage increased, compressive strength also increased. The optimum Na_2SO_4 dosage was 2%.

Generally activated blended cements resisted $MgSO_4$ environment better than unactivated cements.

References

[1] Mehta PK. High-Performance, high-volume fly ash concrete for sustainable development, International workshop on sustainable development and concrete technology; 2006.

[2] Sobolev K., Mechano-chemical modification of cement with high volumes of blast furnace slag, Cement and Concrete Composites, 27 (2005) 7–8, 848–853.

[3] Donatello, A. Palomo, A. Fernández-Jiménez, Durability of very high volume fly ash cement pastes and mortars in aggressive solutions, Cement and Concrete Composites, 38 (2013), 12–20.

[4] Merida A. and Kharchi F. " Pozzolan concrete durability on sulphate attack," *1st International Conference on Structural Intergrity,* Vol. 114, pp. 832-837, 2015.

[5] Wankhede P. R. and Fulari V. A. "Effect of fly ash on properties of concrete," *International Journal of Emerging Technology and Advanced Engineering*, V. 4 pp. 1-6, 2014.

[6] Sarath C. and Saha P., "Contribution of Fly Ash to the Properties of Mortar and Concrete‖," *International Journal on Earth Science & Engineering*, ISSN 0974-5904, Volume 04, No 06 SPL, pp. 1017-1023, October 2011.

[7] Momade, Z. and Atiemo, E. "Evaluation of pozzolanic activity of some clays In Ghana," *Journal of Science and Technology*, Vol. 24 pp. 76-83, 2004.

[8] Atiemo, E., "Clay as pozzolana for building purposes," *Journal of Building and Road Research Inst*., Vol. 2, pp. 16-24, 1998.

[9] Atiemo, E., “Production of pozzolana from some local clays: prospects for application in housing construction.,” *Biennial Journal of Building and Road Research Institute*, Vol. 9, pp. 34-41, 2005.

[10] Siddique R., and Khan M. I., “Supplementary Cementing Materials,” Springer-Verlag Berlin Heidelberg, pp. 179, 2011.

[11] Singh, M. and Garg, M., “Reactive pozzolana from India clays-their use in cement mortars,” *Cement and Concrete Research*, Vol. 36, pp. 1903-1907, October 2006.

[12] Lea, F. M, “The Chemistry of Cement and Concrete,” 3rd ed., pp. 235-245. Edward Arnold (Publishers) Ltd., Glasgow, 1970.

[13] Boakye K. A., Atiemo E. Sarfo-Ansah J., Osei-Frimpong A. and Adjaottor A. A. “Improvement of setting times and early strength development of clay pozzolana cement through chemical activation,” *Journal of Chemical Engineering and Chemistry Research*, Vol. 1, No. 2, pp. 77-83, 2014.

[14] Shi, C. and Day, R. L., “Pozzolanic reaction in the presence of chemical activators: Part I. Reaction kinetics,” *Cement and Concrete Research*, Vol. 30, pp. 51– 58, 2000.

[15] Shi, C. and Day, R. L., “Comparison of Different Methods for Enhancing Reactivity of Pozzolans,” *Cement and Concrete Research*, Vol. 31, pp. 813-818, 2001.

[16] Owens, K. J., Bai, Y., Cleland, D., Basheer, P. A. M., Kwasny, J., Sonebi, M., Taylor, S., and Gupta, A., “Activation of high volume fly as paste using chemical activation,” 2nd International Conference on Sustainable Construction Materials and Technoligies, Universita Politecnica Delle Marche, Ancona, Italy, pp. 1759-1770, 28-30 June 2010.

[17] Angel P. and Ana F. “Alkaline activation, procedure for transforming fly ash into new materials. Part I: Applications,” World of Coal Ash (WOCA) Conference, May 9-12, 2011 in Denver, USA.

[18] Xu, A., Sarkar, and S. L., “Microstructural study of gypsum activated fly ash hydration in cement paste,” *Cement and Concrete Research,* Vol. 21, pp. 1137-1147, 1991.

[19] Shi, C. J., “Early microstructure development of activated lime-fly ash pastes,” *Cement and Concrete Research*, Vol. 26, pp. 1351-1359, 1996.

[20] Shi, C. and Day, R. L., “Pozzolanic reaction in the presence of chemical activators, part II: Reaction products and mechanism,” *Cement and Concrete Research*, Vol. 30, pp. 607-613, 2000.

[21] American Society for Testing and Materials, Annual book of ASTM standards: Part 14-C 618; Standard specification for fly ash and raw or calcined natural pozzolana for use as a mineral admixture in Portland cement concrete, ASTM, Philadelphia-2000.

[22] European Committee for Standardization, Composition, specifications and conformity criteria for common cements, The European Standard, EN 197-1, CENBrussels-2000.

[23] British Standards Institution, Standard methods of test for soils for civil engineering purposes, BS 1377, BSI, London-1990.

[24] American Society for Testing and Materials, 2000 annual book of ASTM standards: Part 13-C 204; Standard test method for fineness of Portland cement by air permeability method, pp. 195-201, 2000.

[25] European Committee for Standardization, Methods of testing of cement - Part 3. Determination of setting time and soundness, EN 196-3, The European Standard CEN, Brussels-2000.

[26] British Standards BS 4550-6, Methods of testing cement. Standard sand for mortar cubes, BSI, London, 1978.

[27] European Committee for Standardization, Methods of testing of cement - Part 1. Determination of strength, EN 196-1, The European Standard CEN, Brussels-2000.

[28] Memon, S. A., Arsalan, R., Khan, S., Lo, T. Y., “Utilization of Pakistani bentonite as partial replacement of cement in concrete,” *Construction and Building Materials, Vol.* 30, pp. 237-242, 2012.

[29] Sitthisak P., Samunya S., Supaporn W., and Thammarat P. “Effects of ash content and curing time on compressive strength of cement paste with rice husk ash,” *Journal of Science and Technology*, Vol. 17, pp. 293-302, 2010.

[30] Lee, C. Y., Lee, H. K., and Lee, K. M., “Strength and microstructural characteristics of chemically activated fly-ash-cement systems,” Cement and Concrete Research, Vol. 33, pp. 425-431, 2001.

[31] Chindaprasirt, P., Homwuttiwong, S., and Sirivivatna, V., “Influence of fly ash fineness on strength, drying shrinkage and sulfate resistance of blended cement mortar, *Cement and Concrete Research*, Vol. 34, pp. 1087-1092, 2004.

[32] Kaid, N., Cyr, M. Julien, S. and Khelafi, H. “Durability of concrete containing a natural pozzolan as defined by performance-based approach,” *Construction and Building Materials,* Vol. 23, pp. 3457-3467.