

A Study of the Pozzolanic Properties of Anfoega Kaolin

Tagbor, T. A., Boakye, K. A., Sarfo-Ansah J. and Atiemo, E.

CSIR-Building and Road Research Institute, Box UP 40, Kumasi, Ghana

Abstract

Kaolin, usually referred to as “hyire” in Ghana is commonly used for traditional religious ceremonies or medicinal purposes. However, there exist abundant kaolin deposits in Ghana whose potential is under-utilized. This work therefore has studied the potential use of kaolin from Anfoega, a small town in the Volta Region of Ghana as a mineral admixture in Portland cement. The raw kaolin was calcined at 700°C to produce metakaolin and milled to cement fineness. The physical, chemical as well as mineralogical properties were conducted on the raw and calcined kaolin. Compressive strength test on the mortars, prepared by replacing ordinary Portland cement with 5% to 40% metakaolin was also conducted. XRF analysis of the metakaolin identified SiO₂ and Al₂O₃ as the dominant oxides whilst the presence of pyrophyllites, magnetite and quartz were identified by XRD. Compressive strength decreased as metakaolin content in the cement increased. The optimum replacement was 25%, recording compressive strengths of 11.2MPa, 16.8MPa and 43.0MPa for 2, 7 and 28 days respectively. At 25%, water permeability was improved by 38.7%. Co-efficient of water permeability (K_α) decreased with increasing metakaolin content. This investigation has shown that Anfoega kaolin has good pozzolanic properties and can be used as a mineral admixture in Portland cement.

Keywords: Kaolin, metakaolin, compressive strength, mineralogical composition, permeability

I. Introduction

Kaolin is a commercial term used to describe white clay composed essentially of kaolinite, Al₄Si₄O₁₀(OH)₈, with admixtures from quartz and feldspar [1]. Although originally valued for use in the manufacture of white ware ceramics, the principal use of kaolin is now in the filling and coating of paper. It is also used to a lesser extent as filler in paint, rubber and plastics, as well as in a wide range of other applications [2]. Kaolins are distinguished from other clays by whiteness, and fine, controllable particle size [1].

It is generally necessary to process kaolin from the crude state in order to optimize these highly commercial properties. When calcined between temperatures of 500°C and 850°C, kaolin dehydroxylates into metakaolin [3]. Metakaolin reacts with Ca(OH)₂, produces Calcium Silicate Hydrates (CSH) gel at ambient temperature [4]. When mixed with Portland cement, the silica in the metakaolin combines with the free lime released during the hydration of cement [5]. The pozzolanic activity is due to the presence of fairly divided glassy silica and lime which produces calcium silicate hydrate (C-S-H), C₂ASH₈ (gehlenite hydrate) and C₄AH₁₃ (tetracalcium aluminate hydrate). The formation of secondary C-S-H by this reaction reduces total porosity and refines the pore structure, improving the strength and impermeability of the cementitious matrix [6, 7].

Pozzolanic materials including silica fumes, fly ash, slag, rice husk ash and metakaolin have been used in recent years as cement replacement for

developing high strength concrete with improved workability, strength and durability [8, 9]. Anfoega in the Volta Region of Ghana is well known for its vast kaolin deposit covering about 160,000 square meters. Kaolin, usually referred to as “hyire” or “white clay” in Ghana is commonly used for traditional religious ceremonies, medicinal purposes and sometimes eaten by pregnant women to control the urge for vomiting.

II. Materials and methods

Materials

The materials used for this work were Kaolin obtained from Anfoega, a small community about 250 km from Ho in the Volta Region of Ghana; Ordinary Portland cement (Class 42.5N) manufactured by Dangote Cement; Pit sand which satisfied BS 4550: Part 6 [10] requirements. The sand was sieved passing through 0.85 mm test sieve and retained on 0.60 mm test sieve. The physical and chemical properties of the kaolin, metakaolin, and OPC are presented in Table 1 and 2 respectively.

Methods

The raw kaolin sample was prepared by taking out various impurities and then air dried to get an appreciable amount of moisture from the kaolin. It was milled in a jaw crusher to an average particle size of 150 μm. The milled kaolin sample was nodulized and calcined in a laboratory kiln at 700 °C for 2 hours with a temperature rise increment rate of 5°C/min to produce metakaolin. The calcined nodules were milled to cement fineness using laboratory type ball mill and sieved through 75μm standard sieve to

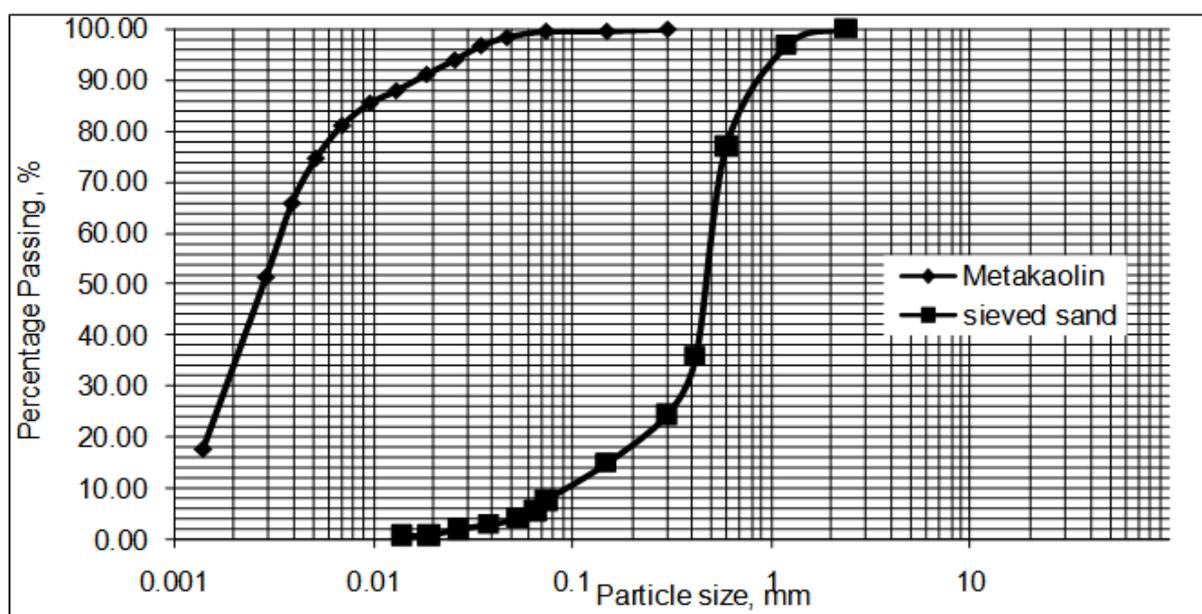
obtain powdered metakaolin (MK). The metakaolin was used to replace 5%, 10%, 15%, 20%, 25%, 30%, 35% and 40% of ordinary Portland cement (OPC) to produce blended cements. Physical properties such as particle size distribution, Blaine fineness, specific gravity and density were determined. Water demand, setting times and soundness were determined according to EN 196-3 [11]. The chemical and mineralogical properties were also determined using a Spectro Xlab X-Ray Fluorescence spectrophotometer and PHILIPS PW 1830 diffractometer respectively. 70 mm × 70 mm mortar cubes were molded according to procedures outlined by EN 196-1 [12] and cured in a moisture cabinet. The specimens were tested after 2, 7, 28 days. A water permeability study was conducted using methods prescribed by ASTM C642-06 [13].

III. Results and discussions

Physical properties

Figure 1 presents the particle size distribution graph of the milled metakaolin whereas Table 1 shows some physical properties of the OPC,

metakaolin and the blended cements used in this study. The particle size distribution, shown in Fig. 1 confirms that the sand used was very coarse and contained minimal amount of silt. The metakaolin was also very fine with 85% finer than 10 μm. The Blaine indices in Table 1 shows that the surface area of the blended cement increased as metakaolin content in the mix increased. Specific gravity on the other hand decreased with increasing metakaolin content. Water demand to form a workable paste increased with increase in the metakaolin content. This is because the smaller particle size of the metakaolin increased the surface area of the whole mix translating into higher water demand [14]. There was a progressive increase in both initial and final setting times as the admixture content increased. At 40%, the initial and final setting times had increased by 23.8% and 27% respectively. Setting time of cements changes with particle size, specific surface area and mineralogical structure of the cement mixtures [15]. The increase could be due to the decrease of tricalcium aluminate (C₃A) content of the OPC [16].



This results in slow hydration of the paste, thereby causing a delay in the setting of the paste. The soundness of the blended cements also increased as metakaolin replacement increased. However, the setting times and soundness obtained all fell within acceptable limits as prescribed by the EN 197-1 [17] standard.

Chemical and mineralogical analysis

The results of the chemical analysis of OPC, kaolin and metakaolin as determined by X-ray fluorescence (XRF) have been presented in Table 2. For OPC, the sum of the CaO and SiO₂ content gave 78.52% and loss on ignition of 1.2% which satisfies the standard limit of EN 197-1[17].

Table 2: Chemical analysis of OPC, kaolin and metakaolin

Composition, %	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	L.O.I
OPC	18.88	3.57	3.36	59.64	1.89	4.93	1.2
Kaolin	51.52	36.9	0.96	0.58	0.08	0.22	13
Metakaolin	57.5	40.18	1.23	0.45	0.12	0.61	0.65

Chemically, the OPC could be considered as good cement. Also, the SiO₂ content of 51.52% and loss of ignition of 0.65% of the metakaolin passed the requirements specified by the EN 197-1 [17]. The sum of SiO₂, Fe₂O₃ and Al₂O₃ content of the metakaolin exceeded the minimum value of 70% required by ASTM C618 [18]. Chemically, the metakaolin could also be considered as a good pozzolanic material. The XRD analysis in Fig. 2 shows that the kaolinite phase [Al₂SiO₅(OH)₄.2H₂O] was dominant with traces of quartz (SiO₂). After calcination (Fig. 3), more of the SiO₂ phase was formed with Al₂O₃.4SiO₂.H₂O and Fe₃O₄ also present.

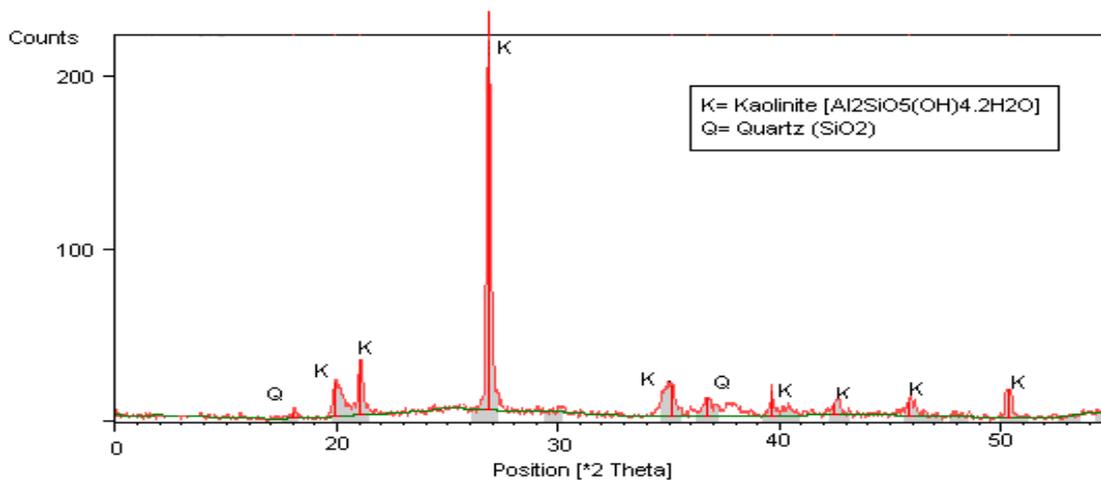


Figure 2: XRD of kaolin

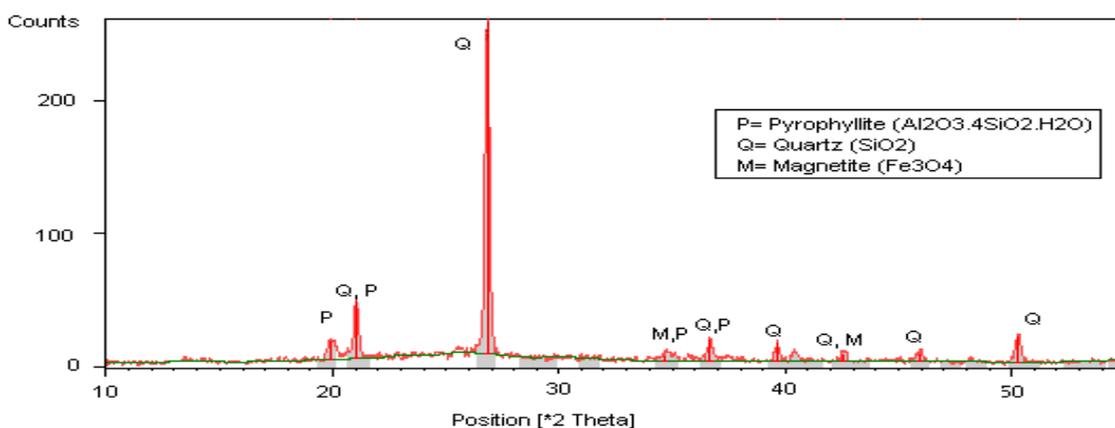


Fig. 3: XRD of metakaolin

X-ray diffractograph obtained for the 28 days hydrated cement paste containing 25% metakaolin is shown in Fig. 4. The main hydration products obtained are calcium disilicate [CaSi_2O_5], Portlandite [$\text{Ca}(\text{OH})_2$], Tobermorite [$\text{Ca}_5\text{Si}_6\text{O}_{16}(\text{OH})_2$] and sodium disilicate gel [$\text{Na}_2\text{Si}_2\text{O}_5$].

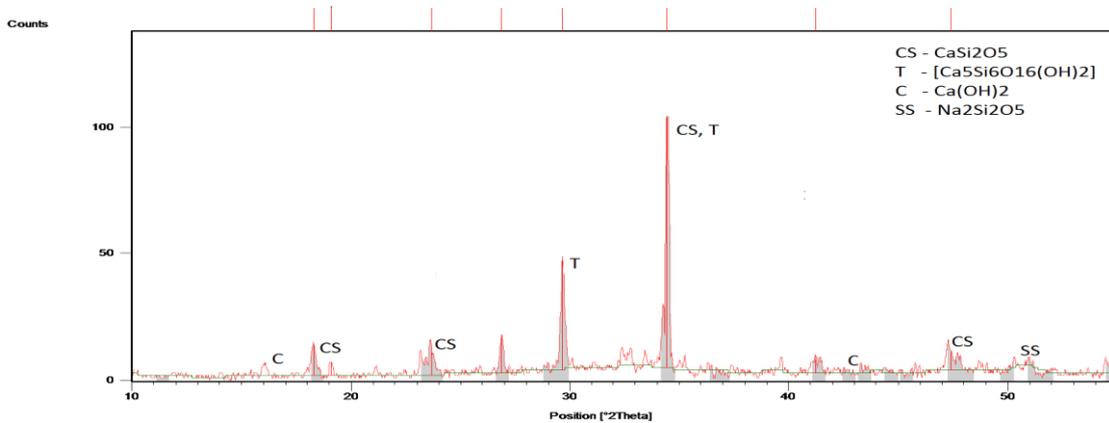


Fig. 4: XRD of cement containing 25% metakaolin hydrated for 28 days

Compressive strength

The 2, 7 and 28 days compressive strength results of reference and blended cement shown in Fig. 5 indicates that all the OPC obtained a 2 and 7 days strengths of 22.5 MPa and 31.7 MPa respectively. These strength values consistently reduced as the metakaolin replacement increased. The addition of 25% metakaolin decreased strengths to 11.2 MPa and 16.8 MPa respectively, but still satisfied EN 197-1 [17] standard for early strength. When the replacement was increased beyond 25%, the compressive strength consequently decreased, satisfying the standard at 2 days but trailing it by 0.6MPa at 7 days. Beyond 30%, the mortar cubes recorded compressive strengths lower than the EN 197-1 [17] standard for 7 days strength.

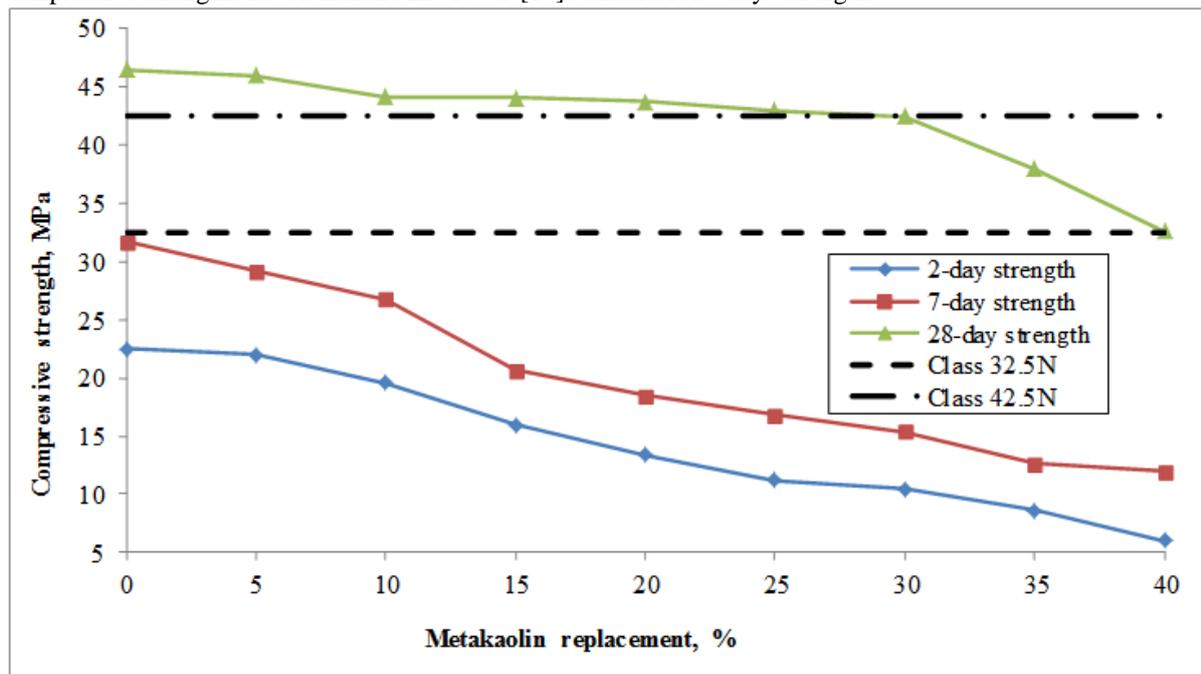


Figure 5: Compressive strength of OPC with different metakaolin contents

The reduction in strength is due to the slow reaction rate of the active metakaolin constituents with the liberated $\text{Ca}(\text{OH})_2$ from the Portland cement [19]. When the curing period was increased to 28 days, the reference cement sample recorded a compressive strength of 46.5 MPa. The strength values recorded for all blended at 28 days were high, as even mortar cubes containing 40% metakaolin cement obtained compressive strength that passed the ASTM C595 [20] standard. Thus 30% metakaolin cement satisfied requirements for

Class 42.5N cement whereas 40% metakaolin content performed as Class 32.5N cement. However, the optimum replacement was 25% metakaolin, considering both early and ultimate strength.

Water permeability

The 28 days water permeability studies conducted on the reference and blended cements are presented in Table 3. Co-efficient of water permeability, K_a , decreased as metakaolin content increased. The reference cement obtained a co-efficient of water permeability of $5.32 \times 10^{-10} \text{ m}^2/\text{s}$ which was improved by about 38.7% when the OPC was substituted with 25% metakaolin. At 40% metakaolin substitution, co-efficient of permeability had decreased by 48.9%. Metakaolin acts as a filler and seals the pores in the mortar due to its high degree of fineness [21]. It significantly influences the pore structure in mortars and produces substantial pore refinement leading to significant modifications to the water transport properties [22].

Table 3: Water permeability studies on OPC and blended cements

Sample	OPC	5%	10%	15%	20%	25%	30%	35%	40%
$K_a \times 10^{-10}, \text{m}^2/\text{s}$	5.32	5.12	4.83	4.52	4.01	3.26	3.13	3.01	2.72

K_a – Co-efficient of water permeability

IV. Conclusion

This study has shown that kaolin from Anfoega has the relevant chemical content and when calcined at 700°C for 2 hours could be converted to metakaolin. The metakaolin had a good chemical composition satisfying the standard requirements of a pozzolan. The mineralogical analysis of the metakaolin showed the presence of more SiO_2 phase and traces of $\text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2 \cdot \text{H}_2\text{O}$ and Fe_3O_4 . The hydrated cement containing metakaolin showed a greater consumption of $\text{Ca}(\text{OH})_2$ than in the reference cement with traces of unreacted Portlandite which could be consumed if curing period is extended. Compressive strength of blended cements increased with increasing metakaolin content. 30% metakaolin cement performed as Class 42.5N cement whereas 40% satisfied requirements of Class 32.5N cement. The optimum replacement was 25% metakaolin, considering both early and ultimate strength. Co-efficient of water permeability also decreased with increasing metakaolin content. This investigation has shown that Anfoega kaolin has good pozzolanic properties and can be used as a mineral admixture in Portland cement.

References

- [1] Bloodworth, A. J., Highley, D. E. and Mitchell, C. J. (1993) Industrial Minerals Laboratory Manual, Kaolin. A Report prepared for the Overseas Development Administration under the ODA/BGS Technology Development and Research Programme, Project 9 1/1, p.1.
- [2] Talabi, A. O., Ademilua, O. L., and Akinola, O. O. (2012) Compositional features and industrial application of Ikere kaolinite, Southwestern Nigeria, *Research Journal in Engineering and Applied Sciences*, 1(5) 327-333.
- [3] Patil S. N., Gupta and A. K., Deshpande, S. S., (2012): Metakaolin-Pozzolanic Material for Cement in High Strength Concrete, *Journal of Mechanical and Civil Engineering (IOSR-JMCE)*, 46-49.
- [4] Poon, C. S., Lam, L., Kou, S. C., Wong, Y. L., and Wong, R. (2001) Rate of pozzolanic reaction in high-performance cement pastes, *Cement and Concrete Research*, 31:1301-1306.
- [5] Dojkov, I., Stoyanov, S., Ninov J. and Petrov B. (2013) On the consumption of lime by metakaolin, fly ash and kaolin in model systems. *Journal of Chemical Technology and Metallurgy*, 48 (1) 54-60.
- [6] Siddique R. and Khan M. I. (2011), *Supplementary Cementing Materials*. Springer-Verlag Berlin Heidelberg, 179.
- [7] Singh, M. and Garg, M. (2006), Reactive pozzolana from India clays-their use in cement mortars, *Cement and concrete research* 36 (10) 1903-1907.
- [8] Khater, H. M. (2010) Influence of metakaolin on resistivity of cement mortar to magnesium chloride solution. *Ceramic – Silicaty*, 54 (4) 325-333.
- [9] Kim H.S., Lee S.H. and Moon H.Y. (2007) Strength properties and durability aspects of high strength concrete using Korean metakaolin. *Construction and Building Materials*, 21 (6) 1229-1237.
- [10] British Standards Institution (1978). Part 6: Methods of testing cement: standard sand for mortar cubes, p.23. BSI, London.
- [11] European Standard EN 196-3 (2000), Methods of testing cement: Determination of setting times and soundness, p: 1-18. European Committee for Standardization, Brussels.

- [12] European Standard EN 196-1 (2000), Methods of testing cement: Determination of strength, p: 1-36. European Committee for Standardization, Brussels.
- [13] American Standard for Testing and Materials ASTM C642-06: Standard Test Method for Density, Absorption, and Voids in Hardened Concrete, ASTM, Philadelphia, 2006, p. 3.
- [14] Memon, S. A., Arsalan, R., Khan, S., Lo, T. Y. (2012) Utilization of Pakistani bentonite as partial replacement of cement in concrete, *Construction and Building Materials*, 30, 237-242
- [15] Lea FM. The chemistry of cement and concrete. 3rd ed. London: Edward Arnold, Ltd.; Reprinted in 1976.
- [16] Ambroise, J., Maximilien, S., Pera, J. (1994), Properties of metakaolin blended cements. *Ad. Cem. Based Matter*, 1(4) 161-168.
- [17] European Standard EN 197-1 (2000), Composition, specifications and conformity criteria for common cements, p: 15. European Committee for Standardization, Brussels.
- [18] American Society for Testing and Materials (2000), 2000 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, p.355-358. ASTM, Philadelphia.
- [19] Lea, F.M. (1970a), The chemistry of cement and concrete, 3rd ed., p: 235-245. Edward Arnold (Publishers) Ltd., Glasgow.
- [20] American Society for Testing and Materials (2000) 2000 annual book of ASTM standards: Part 14-C 595; Specification for blended hydraulic cement, ASTM, Philadelphia, 337-345.
- [21] Guneyisi, E., Gesoglu, M., & Mermerdas, K. (2008) Improving strength, drying shrinkage, and pore structure of concrete using metakaolin. *Materials and Structures*, 41, 937-949.
- [22] Sabir, B. B., Wild, S., & Bai, J. (2001). Metakaolin and calcined clays as pozzolans for concrete: A review. *Cement & Concrete Composites*, 23, 441-454.