



Applicability of the standard specifications of ASTM C618 for evaluation of natural pozzolans

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ABSTRACT

Nowadays, the production of binary cements, containing pozzolans (including silica fume, fly ash, natural pozzolans, etc.), is a global practice. Many countries have ample resources of natural pozzolans, capable of being used in binary cements, which reduce environmental impacts while reaping greater economies of scale. The ASTM C618 standard provides one of the most applicable methods for evaluating natural pozzolans. Some research results show contradictions between performance of pozzolans in concrete and the specifications of ASTM C618, as pozzolans having a high pozzolanic activity according to this method do not always exhibit suitable performance in concrete, however, in the other researches ASTM C618 showed compatibility. This treatment analyses the chemical and physical properties of different natural pozzolans by means of ASTM C618 and complementary tests, viz. methods of EN 196-5, X-ray diffraction (XRD) and environmental scanning electron microscopy (ESEM) studies, insoluble residue content and thermo-gravimetric investigations. Measurements of mechanical and transport properties, for concretes containing various fractions of the pozzolans, were performed for further verification. The results illustrate that the pozzolanic properties, determined via the ASTM C618 standard, show some disparities with the performance of concretes, whereas the EN 196-5 standard agrees well with performance.

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1. Introduction

In recent decades, environmental considerations and energy efficiency requirements have motivated researchers to search for sustainable solutions in development. One of the most polluting industries is cement production, since the production of 1 metric-ton of cement results in the emission of about 1 metric-ton of CO₂ [1,2]. Therefore, the application of blended cements, in lieu of ordinary Portland cement, is rapidly increasing [3].

Contemporary surveys reveal that the majority of European cement production is allocated to blended cements [4]. EN 197-1 [5] designates 27 different cement types, from which 26 are categorized as blended cements. Blended cements consist of different supplementary cementitious materials (SCM), such as fly ash, silica fume, blast furnace slag, limestone and natural pozzolans. Natural pozzolans, owing to their abundance and relatively low costs, present considerable potential for employment in the cement and concrete industries. Additionally, their application generally results in decreases in pollutant emissions and increases in concrete durability properties [6–11]. For a long time the use of natural pozzolans has been mostly restricted to Italy, where considerable reserves of

natural pozzolans are found [12]. Nowadays, ample resources of natural pozzolans capable of being used in binary cements can be found in countries such as Italy, China, USA, Chile, Greece, Cameroon, Algeria, France, Turkey, Iran, Saudi Arabia, and Honduras [12–20].

Natural pozzolans are characterized by their pozzolanic activity and performance. Hitherto, several methods for assessment and classification of natural pozzolans have been presented, and debated [21–23]. From these methods, the requirements of ASTM C618-03 [22], for selecting pozzolans for use in concrete, and the methodology of EN 196-5 [23] for measuring pozzolanic activity of pozzolanic cements, have gained ample application in evaluating pozzolanic activity properties.

The outcomes of some research projects have exhibited some anomalies between these different approaches [24–26]. The authors investigated and found that ASTM C618 can have significant problems in characterizing natural pozzolans [26]. So, it was decided to make a comprehensive study on pozzolanic activity of different types of natural pozzolans with the different approaches that existed, such as ASTM C618 and the EN 196-5 method, X-ray diffraction (XRD) and environmental scanning electron microscopy (ESEM) studies, determination of insoluble residue content, and thermo-gravimetric investigations. Finally, since the performance of pozzolans in concrete mixtures (i.e. their effects on concrete

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properties), is ultimately the most representative judgment strategy for pozzolanic activity evaluation, the results were cross-checked with measurements of mechanical and transport properties of concretes with the same pozzolans.

2. Experimental program

2.1. Materials

2.1.1. Cement and pozzolans

A single cement type (Type II; ASTM C150 [27]) and four different natural pozzolans, viz. Tehran (T) of Trass type, Abyek (A) of Tuff type, Khash (K) and Pars Pumice (P) of Pumice type, have been incorporated in this research. Table 1 presents the chemical and physical properties of the cement and pozzolans.

2.1.2. Aggregates

The physical and mechanical properties of siliceous coarse and fine aggregates are reported in Table 2.

2.2. Evaluation of pozzolans by ASTM C618

ASTM C618 presents chemical and physical requirements and specifications for fly ash and natural pozzolans for cement replacement (see Table 3), where the standard test procedures of ASTM C311 [28] are incorporated. ASTM C618 was published in 1968 to combine and replace ASTM C350 on fly ash and ASTM C402 on other pozzolans [29].

Briefly speaking, in this method, the foremost important criteria for pozzolanic activity are: (1) the sum of chemical components, that is $\text{SiO}_2 + \text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$, and (2) the strength activity index, defined as the ratio of the compressive strength for a mortar with 20% pozzolan replacement for cement by mass to the compressive strength of a control mortar. For the control mortar, the water-to-binder ratio by mass (w/b) equals 0.485, and water in the test mortar is regulated to give the same consistency (flow) as the control mortar. Note that, w/b stands for water/binder, where binder

consists of cement plus cement replacement material (pozzolan, if any).

According to Table 3, the pozzolans fulfill the requirements of ASTM C618. It should be noted that the strength activity index of pozzolans is determined based on ASTM C311. It can be inferred that pozzolans A and T have favorable activities and pozzolan P has the lowest activity.

2.3. Complementary tests

2.3.1. Evaluation of pozzolanic activity by EN 196-5

In EN 196-5, pozzolanic activity is assessed based on the concentration of calcium ion, Ca^{2+} (expressed as calcium oxide or CaO), present in the aqueous solution in contact with hydrated blended cement after a fixed period. As seen in Fig. 1, in the “diagram for assessing pozzolanic activity” (a plot of CaO versus OH), a unique predefined curve demarcates pozzolanic and non-pozzolanic areas. The blended cement is satisfactory, if the concentration of CaO in the solution falls below the curve.

In order to define pozzolanic activity for the natural pozzolans, test method EN 196-5 was conducted on four binary cements, designated as follows:

BCT: 80% Portland cement plus 20% T.
 BCA: 80% Portland cement plus 20% A.
 BCK: 80% Portland cement plus 20% K.
 BCP: 80% Portland cement plus 20% P.

Table 2
Aggregate physical properties.

Aggregate type	Physical properties			
	Specific gravity	Absorption (%)	Fineness modulus	% <75 μm
Fine	2.53	2.60	3.2	1.1
Coarse	2.56	1.46	–	0.4

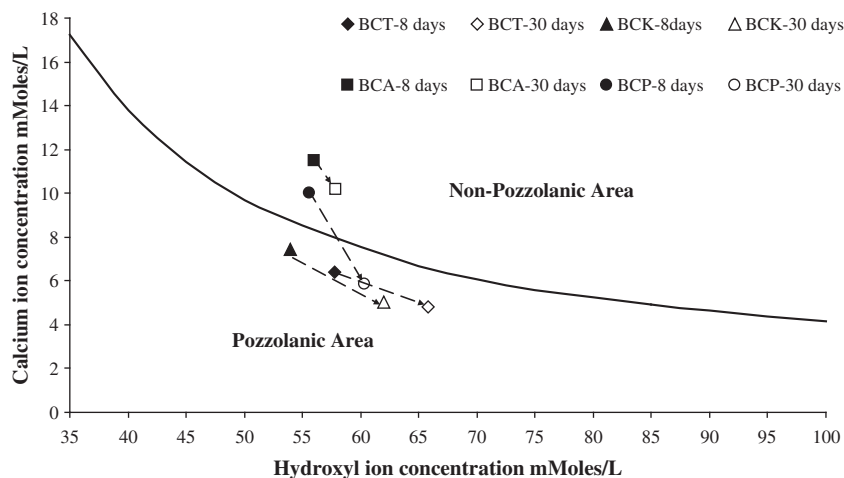
Table 1
Physico-chemical and other properties of cement and pozzolans.

Compound/property	Cement	Pozzolans			
	Type II	T (Trass)	A (Tuff)	K (Pumice)	P (Pumice)
<i>Chemical analysis, %</i>					
Calcium oxide (CaO)	64.45	4.50	1.95	7.16	4.54
Silica (SiO_2)	20.24	65.69	73.36	58.84	64.73
Alumina (Al_2O_3)	4.38	12.04	12.08	17.13	16.48
Iron oxide (Fe_2O_3)	3.66	2.94	1.53	5.10	3.99
Manganese oxide (MnO)	0.09	0.09	0.10	0.09	0.05
Magnesium oxide (MgO)	2.94	1.29	0.55	2.37	1.39
Sodium oxide (Na_2O)	0.20	1.97	3.19	3.66	3.69
Potassium oxide (K_2O)	0.68	2.64	3.22	2.01	1.78
Titanium dioxide (TiO_2)	0.31	0.31	0.21	0.67	0.30
Sulfur trioxide (SO_3)	1.49	<0.10	<0.10	<0.10	0.56
<i>Bogue potential compound composition, %</i>					
Tri calcium silicate (C_3S)	52.74	–	–	–	–
Di calcium silicate (C_2S)	20.31	–	–	–	–
Tri calcium aluminate (C_3A)	3.35	–	–	–	–
<i>Other properties</i>					
3 days compressive strength, MPa	22.3	–	–	–	–
7 days compressive strength, MPa	30.6	–	–	–	–
28 days compressive strength, MPa	41.4	–	–	–	–
Initial setting time, min	150	–	–	–	–
Final setting time, min	190	–	–	–	–
Specific surface, m^2/kg	329	–	–	–	–
Specific gravity	3.15	2.46	2.64	2.65	2.58
Loss on ignition (975 °C)	2.64	8.49	2.92	1.88	2.59
Loss on drying (105 °C)	0.2	2.25	0.85	0.3	0.6

Table 3

Chemical and physical properties of pozzolans according to ASTM C618.

Requirements	Pozzolans				
	Class N, ASTM C618	T	A	K	P
Chemical requirements					
SiO ₂ + Al ₂ O ₃ + Fe ₂ O ₃ , %	Min, 70.0	80.88	86.97	81.07	85.20
Sulfur trioxide (SO ₃), %	Max, 4.0	<0.10	<0.10	<0.10	0.56
Moisture content, %	Max, 3.0	2.48	0.52	0.18	0.85
Loss on ignition, %	Max, 10.0	8.49	2.92	1.88	2.59
Physical requirements					
Amount retained when wet-sieved on 45 µm sieve, %	Max, 34	4	2	7	5
Strength activity index, at 7 days, percent of control	Min, 75	110	105	89	80
Strength activity index, at 28 days, percent of control	Min, 75	118	116	91.5	82
Water requirement, percent of control	Max, 115	106	94	100	104
Autoclave expansion or contraction, %	Max, 0.8	0.05	0.10	0.09	0.09

**Fig. 1.** Treatment results for pozzolan cement pastes at 8 and 30 days at 40 °C (acc. EN 196-5).

The results of EN 196-5 (cf. Fig. 1) imply that:

- After 8 days, BCT fulfills the pozzolanic activity test requirements. By further increasing the reaction time to 30 days, higher alkalinity yet lower CaO concentrations are observed. Hence, chemical reaction is presumably controlled by zeolitic-dominated alkali–calcium-exchange, and therefore, pozzolan T satisfies pozzolanic activity.
- For BCA, after both 8 and 30 days, non-pozzolanic activity is observed. Therefore, pozzolan A may be considered as a non-reactive mortar or concrete filler.
- BCK exhibits mediocre pozzolanic activity, that is to say, after 8 days of reaction the reading is below the curve (cf. Fig. 1), whereas at 30 days the reading shifts to higher alkalinity but lower CaO concentrations (see Fig. 1). Therefore, pozzolan K has pozzolanic activity.
- After 8 days, BCP shows no pozzolanic activity. By increasing the reaction time to 30 days, pozzolanic activity is observed; that is, pozzolan P is slowly reacting and needs more time for alkaline activation.

2.3.2. Mineralogical analysis by XRD and determination of insoluble residue

For the natural pozzolans, crystal minerals are determined via the (XRD) method and results are presented in Table 4.

Insoluble residue (IR) is measured according to EN 196-2 [30] using two methods: in the first (here named as method I), hydrochloric acid and sodium carbonate are used, while the second (here

termed as method II) utilizes hydrochloric acid and potassium hydroxide. The results are presented in Table 5.

The crystal minerals and insoluble residue analyses reveal the following.

Pozzolan T contains about 30% pozzolanic reactive matter, consisting of 8% Calcite CaCO₃, about 17% Zeolite (minerals of the Clinoptilolith–Heulandite group), and about 5% glass phases. The quantitative amount of IR = 70% agrees well with the sum of 25% soluble Calcite and Zeolite, and about 5% soluble glass phases. Other crystalline mineral components, viz. Quartz, Plagioclase (Sodium–Feldspar), Sanidine (Potassium–Feldspar), Mica, Cristobalite, Hornblende and Clay minerals, are considered to be nearly insoluble under the chemical disintegration conditions of method I. Under stronger chemical attacks of method II, IR is drastically reduced to 30%, probably resulting from Feldspar and Mica dissolution. The remaining residues (ca. 30%) predominately consist of Quartz and Cristobalite.

Pozzolan A includes a relatively small amount, about 8% pozzolanic reactive matter, consisting of 4% Calcite CaCO₃ and minor glassy phases. There are also about 90% relatively insoluble crystalline mineral components, e.g. Quartz, Plagioclase (Sodium–Feldspar), Sanidine (Potassium–Feldspar), Mica, Cristobalite, Hornblende and Clay minerals. These confirm the 92% insoluble residue amount.

Pozzolan K contains about 13% pozzolanic reactive matter, including 1% Calcite CaCO₃, traces of Zeolite (from the Morde-nite-group) and small amounts of glassy phases.

Pozzolan P has 13% pozzolanic reactive matter, consisting of 1% Calcite CaCO₃ and about 1% Zeolite, and traces of Clay minerals,

Table 4

XRD results for semi-quantitative crystalline mineral contents of the pozzolans.

Minerals	Pozzolan			
	T	A	K	P
Quartz, %	18	48	1	6
Plagioclase (Albit), %	18	33	68	60
K–Feldspar (Sanidine), %	9	7	–	–
Mica, %	17	7	6	3
Cristobalite, %	12	–	13	18
Calcite, %	8	4	1	1
Hornblende, %	(Traces)	1	11	8
Clay minerals, %	Traces (Smectite)	Traces (Smectite)	–	–
Zeolite minerals, %	17 (Clinoptilolite)	–	Traces (Mordenite)	1 (Smectite, Kaolinite)
Amorphous phases, %	Clear	–	Traces	–

Table 5

Results for determination of insoluble residue (IR).

Pozzolan	IR (%)	
	Method I	Method II
T	71.54	29.30
A	91.98	80.70
K	86.71	75.60
P	87.31	60.80

soluble in method I. Also, there is about 11% glassy phases. Since pozzolan P has a higher soluble amount in method II, it speculatively has a better performance at longer ages.

2.3.3. Particle shape imaging by electron microscopy, ESEM method

For the pozzolans, particle shape analysis is performed using an environmental scanning electron microscope (ESEM). Fig. 2 depicts some sample images.

The ESEM images provide insight to the particulate properties of the pozzolans.

Pozzolan T has roundish grains with rough surfaces capable of absorbing a considerable amount of water in cementitious pastes. Additionally, existing Zeolite minerals (see Table 4), intercalate water molecules to their internal pore structure.

Pozzolan A is characterized by both roundish and edge-rich grain habitus with a relatively large amount of coarse grains. The grains' surfaces are smoother than pozzolan T leading to lower water imbibition.

Pozzolan K encompasses sharp-edged and split-like grains. Particle surfaces seem to be very even and dense, compared to pozzolans T and A, resulting in lower water demand for cementitious pastes.

Pozzolan P is characterized by sharp-edged and split-like grains, originating from relatively hard and brittle-breaking natural stones. Particle surfaces are glass-like, very even, and dense, as compared to pozzolans T and A; therefore, pozzolan P should not increase water demand.

2.3.4. Thermo-gravimetric investigation

The pozzolanic activity (lime binding capacity) of pozzolans was performed by thermo-gravimetric investigation. This method

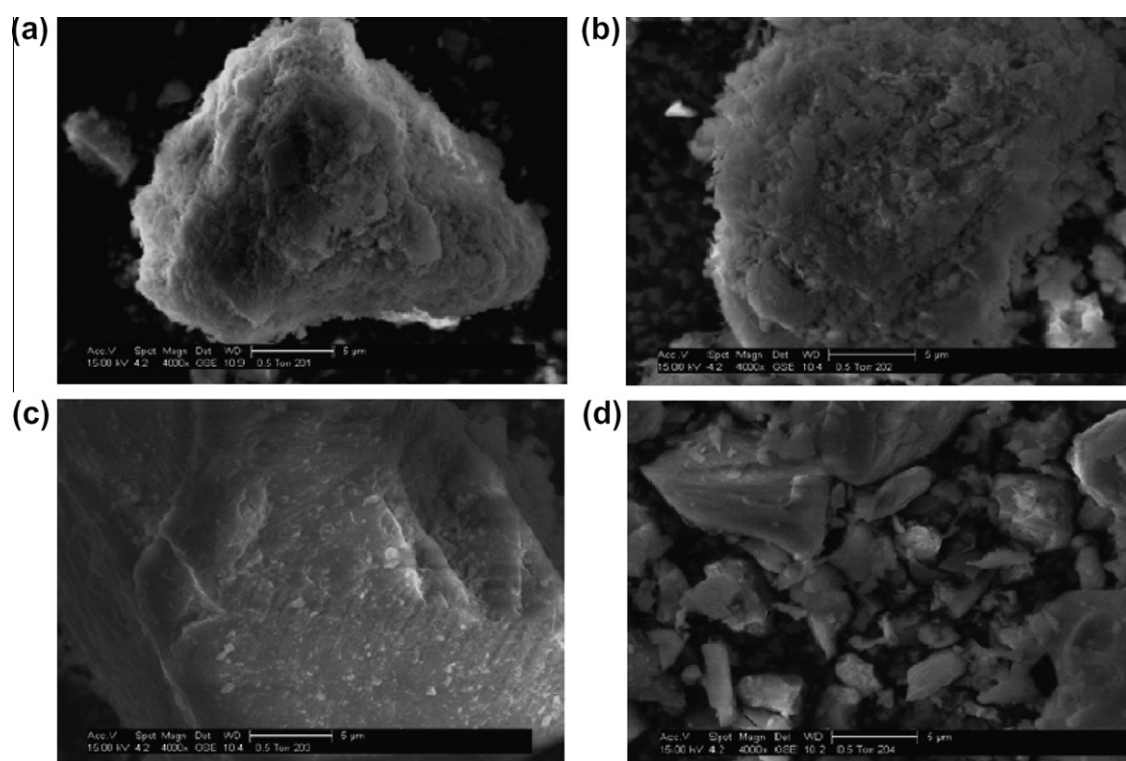
**Fig. 2.** ESEM images of: (a) pozzolan T, (b) pozzolan A, (c) pozzolan K, and (d) pozzolan P.

Table 6
Results of thermo-gravimetric measurements.

Pozzolan	Pozzolanic activity, Ca(OH) ₂ consumption (%)	
	8 days	30 days
T	34	40.5
A	22	21.5
K	36	38.5
P	21.5	28.5

is based on the thermal decomposition of crystalline calcium hydroxide in a temperature range of 400–500 °C to calcium oxide and water. The pozzolanic activity is measured by combining 50% natural pozzolan and 50% Ca(OH)₂ powder in presence of enough water for pozzolanic reaction and measuring the Ca(OH)₂ consumption. The results of this test which are presented in Table 6 for 8 and 30 days, show the arrangement of pozzolanic activity as follows:

- A has the least activity and T has the most activity.
- K has more activity than P.

The increase in pozzolanic activity from 8 days to 30 days of pozzolan P is more visible than the other pozzolans.

3. Evaluating pozzolan effects in concrete mixtures

In order to (1) investigate the effects of pozzolans on concrete properties, and (2) verify the preceding findings, concrete mixtures with 15% and 30% pozzolan as cement replacement by mass were compared with control concrete through several tests. Table 7 presents the concretes' mixture proportions.

Table 7
Mixture proportions of concretes containing pozzolan.

Mixture	Type of pozzolan, cement replacement (%)	Cement (kg/m ³)	Pozzolan (kg/m ³)
C	–	380	–
T-15	Tehran, 15	323	57
T-30	Tehran, 30	266	114
A-15	Abyek, 15	323	57
A-30	Abyek, 30	266	114
K-15	Khash, 15	323	57
K-30	Khash, 30	266	114
P-15	Pars pumice, 15	323	57
P-30	Pars pumice, 30	266	114

Note: The followings are valid for all mixtures: w/b = 0.5, water = 190 kg/m³, fine aggregate = 870 kg/m³ and coarse aggregate = 870 kg/m³.

3.1. Compressive strength

The measured compressive strengths (according to EN 12390-3 [31]) for 150 mm cubic specimens at different ages of 7, 28, and 90 days are presented in Fig. 3. As observed, at 28 and 90 days, concrete mixtures of pozzolans T and A have the highest and lowest compressive strength, respectively. Concrete mixtures of pozzolan P have the lowest compressive strength at 7 days, but their compressive strengths are more than concrete mixtures of pozzolans K and A at 90 days. It can be concluded that pozzolan A has the lowest pozzolanic activity. Additionally, pozzolan P shows better activity at longer ages.

3.2. Depth of water penetration

Fig. 4 presents the depth of water penetration under pressure, determined for 150 mm cubes at the ages of 28 and 90 days according to EN 12390-8 [32]. Here, concrete mixtures of pozzolans T and A have the lowest and highest permeability, respectively. Additionally, concrete mixtures of pozzolan P have a better performance than concrete mixtures of pozzolan K at 90 days.

3.3. Rapid chloride penetration

Fig. 5 illustrates the rapid chloride permeability of specimens, measured for cylindrical specimens (100 mm diameter and 50 mm height) at 28 days, according to ASTM C1202 [33]. The highest and lowest chloride ion permeability is observed in concrete mixtures of pozzolans A and T, respectively, consistent with the water penetration results.

4. Discussion

Based on the ASTM C618 specifications, the pozzolanic activity index is the foremost important criterion for determination of pozzolanic performance. According to Table 3, all the studied natural pozzolans fulfill the requirement of ASTM C618. The observed pozzolanic activity decreases for pozzolans T, A, K and P, inclusively. Besides, the summation of SiO₂, Al₂O₃ and Fe₂O₃ is more than 80% in all pozzolans, with pozzolan A having the highest amount (86.97%). Predicated on these results, it can be postulated that pozzolans A and T would have the best pozzolanic performances, while pozzolan P would have the worst performance.

Considering assessments via EN 196-5 (cf. Fig. 1), BCA has no pozzolanic activity at 8 and 30 days, hence, pozzolan A does not satisfy pozzolanic activity requirements. On the other hand, BCP shows pozzolanic activity after 30 days, suggesting that pozzolan P has superior pozzolanic activity when compared to pozzolan A.

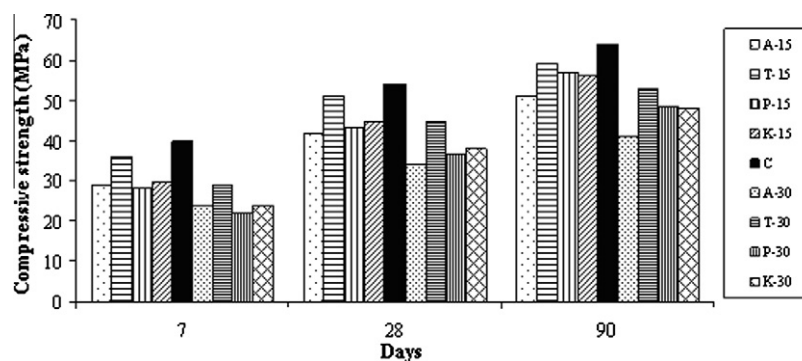


Fig. 3. Compressive strengths of concretes versus age.

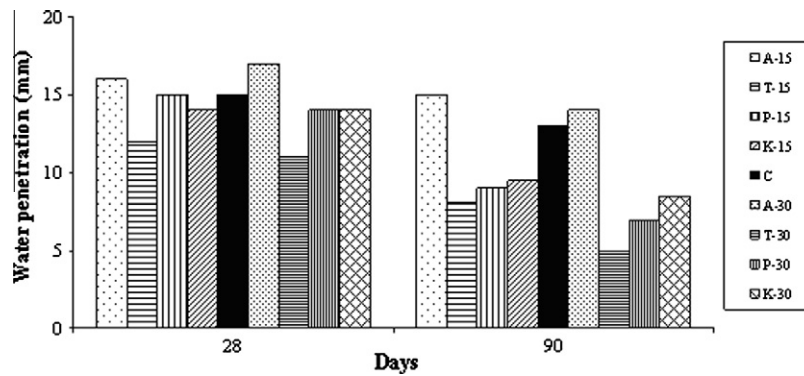


Fig. 4. Depths of water penetration for concretes versus age.

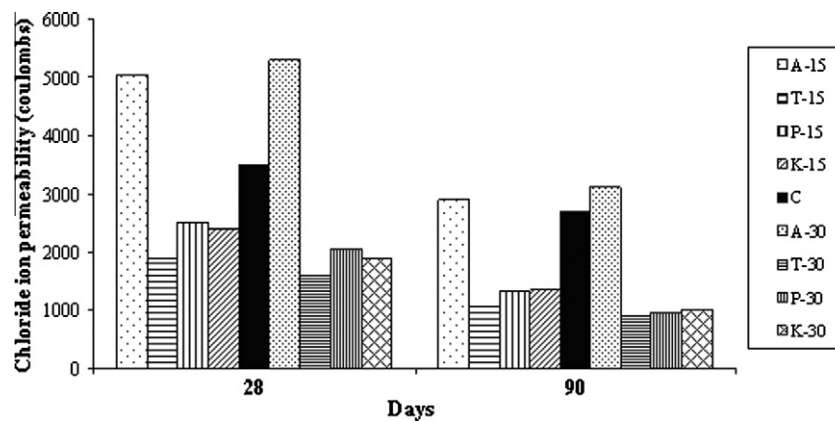


Fig. 5. Rapid chloride permeability test results for concretes versus age.

The insoluble residue (IR) content via EN 196-2 reveals that pozzolan A has the highest amount of insoluble residue. For pozzolan P however, IR is high in hydrochloric acid and sodium carbonate and significantly declines in hydrochloric acid and potassium hydroxide. Thus, pozzolan P encompasses minerals which are respectively insoluble in dilute, but soluble in stronger acidic and basic solutions. This is the main clue to the better performance of pozzolan P in the EN 196-5 test at later ages. Results of thermo-gravimetric analysis confirm the above mentioned results.

Regarding concrete properties (see Section 3), mixtures with pozzolan A have the worst performance (i.e. lowest mechanical and highest transport properties) at different ages. Mixtures with pozzolan P are unsatisfactory (in comparison to other mixtures) at early ages, however, at longer ages, mixtures of pozzolan P turn out to be better than mixtures of pozzolans A and K. Based on the forgoing statements, there is some anomaly between the judgments of ASTM C618 and the real performance of pozzolans in concrete.

The results attest that ASTM C618 is less accurate in defining the pozzolanic activity of natural pozzolans. In ASTM C618, the pozzolanic activity index is defined from ASTM C311, where the ratio of the compressive strength for a cement mortar with 20% pozzolan replacement to the strength of a control mortar, given that both mortars have identical consistency, is measured. For the control mortar, w/b is 0.485, whereas for the mortar with pozzolan replacement, the water content is adjusted to result in the same consistency (flow) as the control mortar. Hence, particular facets of the pozzolan, depending on a host of parameters such as grains' shapes, sizes, and surface texture, govern the required water amount to result in a consistency identical to that of the control

mortar. Thus, the differentiation of w/b may effect the compressive strength, and in turn, the pozzolanic activity index. Because the pozzolan replacement for cement is performed on a mass basis and the pozzolan and cement may have substantially different densities, the difference in the volume fraction of water-filled space in the two mixtures may be further increased.

As seen in Fig. 2, for pozzolan T, the grains have rough and porous surfaces, leading to high water absorption. This pozzolan (T) needs the highest amount of water to reach the consistency of the control mortar (106%). However, owing to the appropriate pozzolanic properties of this pozzolan, the increased water demand does not negatively effect (i.e. decrease) the strength activity index. For pozzolan A, the water demand is lower and the strength activity index is satisfactory based on ASTM C618, yet pozzolan A does not have a good performance in concrete. This outcome may be attributed to the low water demand which produces an artificially satisfactory pozzolanic activity index. Akin to this notion, studies on fly ash, silica fume, and rice husk ash have revealed that pozzolans which satisfy the ASTM C311 requirements may practically have low pozzolanic activities [24–26].

5. Conclusions

The investigation presented in this paper manifests the following major conclusions:

1. The results of determining pozzolanic activity of pozzolans showed that the ASTM C618 standard does not match suitably with the real performance in concrete, perhaps mainly due to

its allowing a variable w/b for the control and pozzolanic mortars.

2. The strength activity index defined by ASTM C311 is not a suitable indicator for pozzolanic activity. Regarding ASTM C311, the authors recommend that the ratio of compressive strength for the control mortar, and the mortar with 20% pozzolan replacement should be compared at identical w/b .
3. The performance of natural pozzolans for replacement in cement is amenable to the crystalline/non-crystalline properties of grains. The total amount of SiO_2 , Al_2O_3 and Fe_2O_3 can not present pozzolanic activity, yet the summation of non-crystalline minerals is important.
4. The EN 196-5 method can accurately define pozzolanic activity in binary Portland cement mixtures. The judgments of this method agreed well with tests results of concrete mixtures in the present study.
5. The authors' perception is that: a combination of chemical and physical methods can authentically define the pozzolanic performance of natural pozzolans for application in binary cements.

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