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POZZOLANIC CHARACTERISTICS OF A NATURAL RAW MATERIAL FOR USE IN BLENDED CEMENTS[*]

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Abstract– In this study, the potential use of a natural raw material in the manufacture of blended cements was investigated. Mineralogical, petrographic and chemical analyses of the samples showed that the natural raw material was a porphyritic volcanic rock close to trachyandesite composition with a SiO_2+Al_2O_3+Fe_2O_3 content of 79.86%. Further experiments were also designed to determine the physical properties and pozzolanic activity of the raw material. The mortar samples, made with a binder of ground trachyandesite and lime, developed compressive and flexural strengths of 2.5 and 3.3 times respectively higher than those required for a natural pozzolan. Further tests revealed that when the ground trachyandesite replaced 30% w/w of Portland cement, the blended cements produced had the desired physical and chemical characteristics with compressive strengths higher than 32.9 N/mm^2. These findings suggest that this material can be used in the production of blended cements.

Keywords– Pozzolan, blended cement, physical properties, mechanical properties

1. INTRODUCTION

Cement is a material that can bind solid particles e.g. gravel, sand, aggregate etc. within a compact structure. A variety of materials may exhibit cementitious properties. In the concrete industry, hydraulic cements such as Portland cement have the ability to set and harden in the presence of water. They are usually manufactured from calcareous raw materials containing silicates, aluminates and iron oxides [1]. Raw materials such as limestone and clay are heated in a kiln at 1400-1450°C to form predominantly clinker, which is then finely ground together with additives such as gypsum to obtain Portland cement [2]. Portland cement is the most common type of cement used in construction applications, but it is an expensive binder due to the high cost of production associated with the high energy requirements of the manufacturing process itself [1]. Other cheap inorganic materials with cementitious properties such as natural pozzolans e.g. volcanic tuff [3, 4] and clay [5], and waste products from industrial plants e.g. slag [6], fly ash [7, 8] and silica fume [9] can be used as a partial replacement for Portland cement i.e. blended cements [10]. In addition, to reduce the cost of binder, there are potential technological benefits from the use of pozzolanic materials as those blended with Portland cement in concrete applications. These include increased workability, decreased permeability [11], increased resistance to sulphate attack [12], improved resistance to thermal cracking and increased ultimate strength and durability of concrete [2, 13, 14].

Pozzolanic cement is a ground product of a mixture containing 20-40% natural pozzolan and 60-80% Portland cement clinker with the addition of a small amount of gypsum. Increase in the natural pozzolan content of cement would reduce the permeability of the paste with the implication of a high resistance to chemical attack, [15] i.e. increase in durability [16]. Furthermore, it was reported that despite the lower

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early concrete strengths, the addition of natural pozzolan (up to 20-30%) could also improve the compressive, splitting and flexural strengths of the concrete in the long term, for example, over a 365 day period [17]. According to TS EN 197-1 [18], minimal SO_3 (≤3.5%) and Cl (≤0.1%) contents, and loss-on-ignition (≤5%) are required for a blended cement. Mortar or concrete samples prepared from a blended cement must produce 7- and 28-day compressive strengths of higher than 16 and 32.5 N/mm^2, respectively.

Pozzolans (trass) are natural rocks (e.g. tuff) of volcanic origin and composed of silica and alumina oxides but almost no lime. Therefore, they cannot develop hydraulic properties in the absence of hydrated lime. Hydrated lime or material that can release it during its hydration (e.g. Portland cement) is then required to activate the natural pozzolans as a binding material [10]. The activity of a natural pozzolan, which is essentially determined by the reactive silica content, is also closely controlled by its specific surface area, chemical and mineralogical composition [14, 19, 20]. Reactive silica is readily dissolved in the matrix as $Ca(OH)_2$ becomes available during the hydration process. These pozzolanic reactions lead to the formation of additional C-S-H with binding properties [7].

Silicate minerals including feldspar, mica, hornblende, pyroxene and quartz or olivine present in volcanic rocks can easily undergo alteration to form secondary mineral phases such as clays, zeolites, calcite and various amphiboles [21]. The contribution of these secondary minerals to the pozzolanic activity of the natural pozzolans was demonstrated [3]. It is mainly accepted that the natural pozzolans that contain low quantities of clay minerals and high quantities of zeolite minerals show good pozzolanic activities [22, 23].

Natural pozzolans with a high content of SiO_2 + Al_2O_3 (≥80%) but a low content of MgO and SO_3 generally exhibit a high pozzolanic activity [19, 24]. However, every natural pozzolan with a strong acidic character does not show pozzolanic activity [14], and hence the assessment of pozzolanic activity of a given natural pozzolan is a prerequisite for its use in the cement industry.

In this paper, the potential use of a natural raw material in the production of blended cement was investigated. The chemical, mineralogical, petrographic, mechanical and pozzolanic characteristics of the samples were examined to correlate the performance of the blended cements produced. Furthermore, the optimum amount of natural raw material to produce a blended cement of desired specifications was determined using a commercially produced clinker.

2. MATERIALS AND METHODS

In this study, samples obtained from a deposit located in Taşhane, the Samsun-Terme district of Turkey, were used. The deposit can be characterised by the presence of massive and widely jointed andesitic tuff layers that contain augite and biotite, and are pale and greenish gray in colour. Representative samples amounting to a total of 100 kg were collected from the site and homogenized prior to use in the chemical, mineralogical and petrographic analyses, and in physical and pozzolanic activity tests.

A number of thin sections were prepared and examined under a Nikon Polarized Light Microscope (Eclipse LV100Pol) to complete the petrographic analysis of the samples. Mineral phases present in the material were also identified using a Rigaku (Geigerflex, D/Max-IIIC) X-ray diffractometer (CuK_α =1.54 Å, Ni filter, 35 KV, 15 mA, 2º/min, 2θ=3-70º). Table 1 presents the chemical composition of the raw material.

Blended cements were prepared by fine inter-grinding (for 30 min) of raw material (20-35% by weight), clinker manufactured in Trabzon Cement Co. and gypsum (4%) in a laboratory mill (30x36 cm (ØxL), 60 rpm, Ø70-30 mm balls). Chemical composition of the clinker and blended cements produced were determined using wet chemical methods by following TS EN 196-2 and 21 [25, 26] as shown in Table 2. The analytical procedure involved fusion of a known amount of the sample with sodium peroxide (at 500±10°C) followed the acid (HCl) digestion of the melt. The solution was then used to determine the major oxides (SiO_2, Al_2O_3, Fe_2O_3, CaO and MgO) and SO_3 via gravimetric and volumetric finishes [25]. Flame photometry (Jenway) was used for the determination of alkali oxides (K_2O and Na_2O) [26]. Loss of ignition (LOI) was analysed by heating the sample in a furnace (Protherm) for 15 min at 975°C.

Table 1. Chemical composition of the natural raw material determined by wet chemical analysis following TS EN 196-2 [25]

Composition	Content (%)	TS 25 [29]
SiO_2	58.46	≥70
Al_2O_3	16.01	
Fe_2O_3	5.39	
CaO	8.77	
MgO	2.60	≤5
K_2O	1.34	
Na_2O	4.44	
SO_3	0.41	≤3
Cl	0.001	
LOI	2.24	≤5
Total	99.66	

Table 2. Chemical composition of clinker (C) and blended cements produced from the natural raw material (NRM) determined by wet chemical analysis following TS EN 196-2 [25]

Composition	Clinker	20% NRM	25% NRM	30% NRM	35% NRM
Undissolved residue	---	17.64	22.76	27.13	31.18
Total SiO_2	20.89	26.17	28.25	31.29	33.29
Al_2O_3	4.89	6.63	7.52	8.08	8.71
Fe_2O_3	4.40	4.47	4.32	4.35	4.29
CaO	64.97	43.46	48.94	48.67	43.60
MgO	0.85	1.61	1.39	1.03	1.19
SO_3	0.21	1.93	1.99	1.95	1.93
Loss of ignition	1.84	2.58	2.85	2.81	2.74
Na_2O	0.27	0.98	1.20	1.34	1.76
K_2O	1.15	1.12	1.10	1.10	1.15
Total	99.43	98.95	97.56	100.62	98.66
Free CaO	2.76	1.92	1.84	1.58	1.41
Lime saturation	92.53	---	50.35	45.74	38.43
Total additive	---	22.50	28.76	34.00	36.31
CO_2	---	0.75	0.88	0.86	0.82
Cl^-	0.014	0.014	0.013	0.013	0.013

Although the chemical composition of a natural pozzolan is significant for its qualification as a potential admixture, unequivocally important is its pozzolanic activity, since some natural pozzolans may fail to show this activity [14]. Strength development is often used to determine pozzolanic properties of a material i.e., the ability to react with lime and form cementitious products [24]. Tests were carried out to determine the physical characteristics of the material using the procedures outlined in TS EN 450-1 [27] and TS 24 [28]. Pozzolanic activity of the raw material was determined as prescribed by TS 25 [29]. Mortar samples were prepared in triplicate by mixing the appropriate amounts of the natural raw material, standard sand, lime and water (Table 3). These were then sealed to prevent evaporation and allowed to cure in a moist environment initially for 24 h (at 23±2°C) and then for a further 6 days at 55±2°C prior to the determination of compressive and flexural strengths (Table 4) by the "Rilem-Cembureau Method" [30]**.** The flexural strength of the samples was tested on bending equipment (RMU 24100, 10 kN) at a loading rate of 50 N/s. Reactive silica content of the material was also determined as the difference between the total silica and the silica present in the insoluble residue of the natural material [25].

Table 3. Admixture proportions in the mortars prepared to determine the pozzolanic activity following TS 25 [29]

Component	Amount (g)
Lime – $Ca(OH)_2$	150
Pozzolan (P)	$2 \times 150 \times \dfrac{\text{SG of Pozzolan}}{\text{SG of Lime}}$
Standard sand [13]	1350
Water	0.5x(150+P)

*SG: Specific Gravity

Table 4. Physical properties and pozzolanic activity test results compared with TS 25 [29]

Property	Sample	TS 25 [29]
Compressive strength, N/mm^2	10.19	>4
Flexural strength, N/mm^2	3.34	>1
Specific gravity (SG), g/cm^3	2.50	---
Specific surface area (Blaine), cm^2/g	4928	>3000
Retained on 200 μm sieve, %	3.30	<0.6
Retained on 90 μm sieve, %	4.40	<8
Saturated surface-dry SG, g/cm^3	2.105	---
Dry density, g/cm^3	1.909	---
Apparent SG, g/cm^3	2.373	---
Resistance to freezing and thawing (Na_2SO_4), %	2.930	---
Relative absorption, %	13.25	---
Bulk density, g/cm^3	1.910	---
Abrasion resistance, %	44	---

The blended cement samples were subjected to the physical characterization prior to the mechanical tests using the procedures outlined in TS EN 196-1, 3 and 6 [30-32]. Specific surface area of the clinker and blended cement samples was measured using an Astek Blaine device and the fineness as the fraction of a sample retained on 90 μm and 200 μm sieves was determined using an air-jet Alpine sieve test device and sieves according to TS EN 196-6 [32]. Specific gravity (SG) of the samples was measured by Le Chatelier pycnometer using kerosene according to TS 639 [33]. The blended cements were used to prepare test specimens (six specimens for each test) with dimensions of 40x40x160 mm [30-32]. Cement (450 g), sand (1350 g) and water (225 ml) were well-mixed in a Seger mortar mixer and the mixture was put into the molds. Over 2, 7 and 28-day curing periods under suitable conditions, compressive strength measurements were performed using a compression tool (Atom Teknik, 10 kN) operating at a loading rate of 2400 N/s [30]. The mean values obtained from six replicate specimens were presented in the results. The initial and final setting times of the mixtures were also determined according to TS EN 196-3 [31] using a Vicat apparatus at room temperature. The Le Chatelier apparatus was used to determine the soundness (volume expansion) of the mixtures, which involved initial curing for 24 h in an oven (Astek) maintained at 21 ± 1^oC and 98% humidity followed by boiling in a water heater (Atom Teknik) for 3 hours prior to the measurement of the distance between the indicators [31].

3. RESULTS AND DISCUSSION

a) Chemical and mineralogical characteristics of the natural raw material

According to TS 25 [29] Turkish Standard, a natural pozzolan is required to contain a minimum of 70% $SiO_2+Al_2O_3+ Fe_2O_3$. The results of chemical analysis (Table 1) show that the raw material fulfils the specifications of TS 25 [13] with a SiO_2+Al_2O_3+Fe_2O_3 content of 79.86% [34]. A relatively high content of active phases (SiO_2+Al_2O_3) with 74.5% could also indicate the acidic character of the material. Furthermore, reactive silica content of the sample was determined to be %33.62, indicating the pozzolanic character for the raw material. The relative degree of pozzolanic activity of natural pozzolans was reported to be intimately associated with their reactive silica content [35]. Papadakis et al. [36] also demonstrated the correlation of pozzolanic activity and compressive strength of a natural pozzolan with its reactive SiO_2. Furthermore, Çavdar and Yetgin [15] reported that an increase in Al_2O_3, Fe_2O_3, MgO and K_2O content of natural pozzolans adversely affected the pozzolanic activity.

XRD analysis of the samples revealed that plagioclase, biotite, and hornblende were the most abundant mineral phases identified (Fig. 1). Microscopic examination of the thin sections showed that the raw material could be generally described as trachyandesite with a hyalo microlitic porphyric texture (Fig. 2). The material was composed mainly of glassy groundmass, plagioclase (Pl), hornblende (Hb) and biotite (Bi) within a decreasing order of their abundance. Augite and magnetite as opaque phases were also identified. Plagioclase appeared to occur mainly as phenocrysts, some of which were zoned with a visible complicated twinning present (Fig. 3a). Some sieve-textured plagioclase phenocrysts were also present (Fig. 3b). Phenocrysts of hornblende were observed to be subhedral showing a typical amphibolic cleavage. The glassy groundmass was noted to be comprised of the microlites of plagioclase and sanidine, and the microphenocrysts of hornblende and biotite.

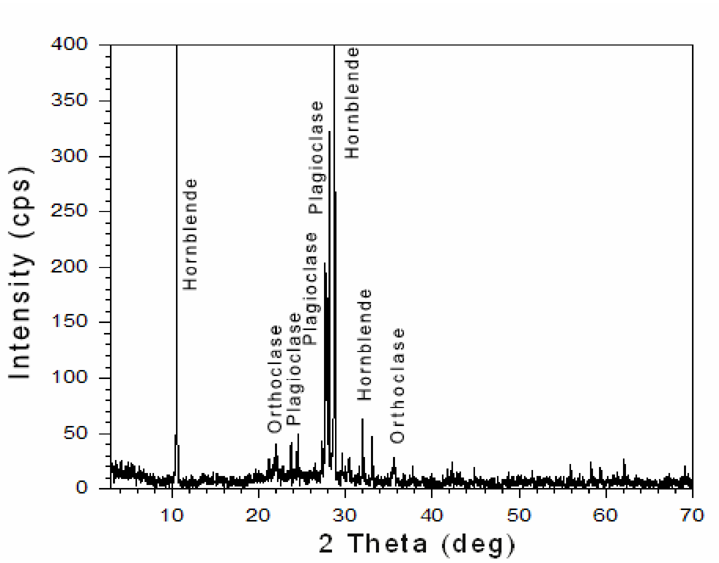

Fig. 1. XRD profile of the natural raw material sample and mineral phases identified

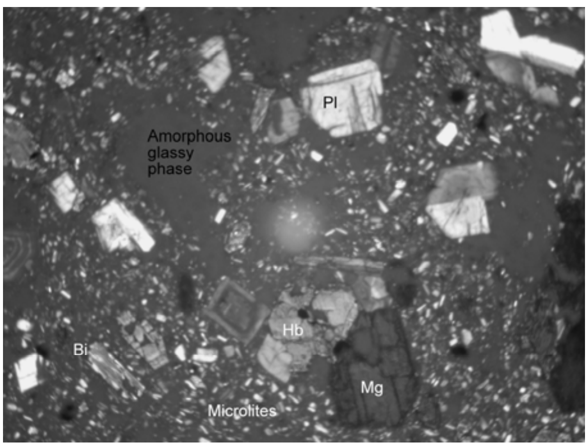

Fig. 2. Microscopic observations under polarized light (x4, XPL). Hyalo microlitic porphyric texture with phenocrysts/microphenocrysts of plagioclase (Pl), hornblende (Hb), biotite (Bi) and magnetite (Mg) as an opaque phase in a fine-grained glassy groundmass

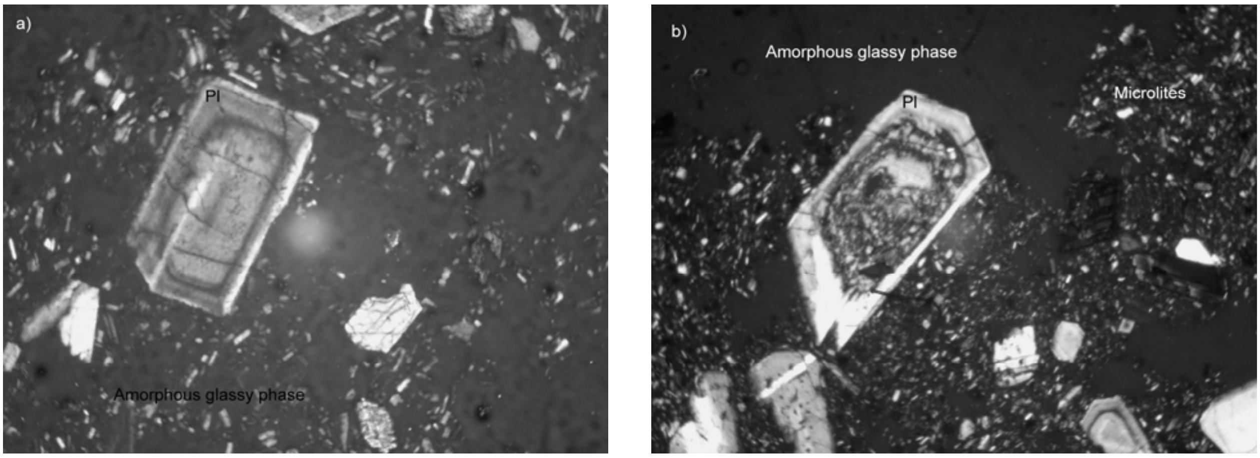

Fig. 3. Microscopic observations under polarized light (x10, XPL). a) A phenocryst of zoned plagioclase and b) a phenocryst of plagioclase (Pl) with a sieve texture

b) Physical properties and pozzolanic activity of the natural raw material

The physical properties of the raw material and the results of pozzolanic activity tests are shown in Table 4 where the standard values [29] are also presented for comparison. The compressive and flexural strengths of the mortar samples produced from the raw material were found to be 2.5 and 3.3 times higher respectively than these specified in TS 25 [29]. Relatively high surface area of the material could have contributed to the strength development, which generally tends to increase with surface area [24].

Rodriguez-Camacho and Uribe-Afif [14] examined the performance of nine natural pozzolans of Mexican origin. They found that the pozzolans showing pozzolanic activity at ≥5.4 N/mm^2 with an alumina content of 11.6-14.7% were highly resistant to sulphate attack compared with those which contain ≥16% Al_2O_3. In this regard, the raw material used in this study could be expected to produce a moderate resistance to sulphate attack when used as an admixture in the cement, but further tests should be performed to verify this assumption.

c) Physical characterisation and mechanical performance of blended cements

The effect of the addition of the raw material (20-35%) on the physical properties and mechanical performance of the blended cements is shown in Table 5 and Fig. 4. The setting times of pastes appeared to increase with the addition of the raw material up to 25%. Thereafter, a reverse trend of decrease was observed, probably linked to the high surface area of the blended cements at these replacement levels (Table 5) as suggested by Targan et al. [37]. A similar trend in setting times was also observed. However, the increase in setting time with the addition of the natural pozzolan could be attributed to the increase in water-to-Portland cement ratio [38]. The decrease in the value of volume expansion (soundness) of pastes with increasing the replacement level (Table 5) appeared to be consistent with the corresponding decrease in the free CaO content of the blended cements (Table 2). Furthermore, free lime could be consumed via pozzolanic reactions in the presence of the natural pozzolan [1, 10]. The beneficial effect of pozzolanic materials on soundness has been reported [39].

Mechanical performance of blended cements in concrete mortars was closely controlled by the level of replacement. A consistent reduction in the compressive strength and rate of strength development of mortars was observed as the amount of the raw material in the blended cement increased (Table 5 and Fig. 4). However a 28-day strength activity index of blended cement mortars over 75% is generally specified for the determination of the optimum substitution level [14], and blended cement mortars are required to develop 7-day and 28-day strengths of ≥16 and ≥32.5 N/mm^2 respectively, as indicated by TS EN 197-1 Turkish standard [18]. Accordingly, the raw material can be added up to 30% w/w to meet the physical, chemical and mechanical requirements for blended cements (Table 6). It should be noted that the low strength of blended cement could be ascribed to the slow development of the pozzolanic reaction, and we can assume that measurements at 90 days will probably show higher strengths.

Table 5. Physical characteristics and mechanical performance of blended cements containing the natural raw material following TS EN 196-1, 3, 6 [30-32]

Properties	Clinker	20% NRM	25% NRM	30% NRM	35% NRM
+200 µm sieve, %	0.6	0.2	0.2	0.1	0.1
+90 µm sieve, %	5.9	4.8	4.3	3.6	2.2
Spec. surface area (Blaine), cm^2/g	3102	2709	2890	3321	3980
Specific gravity, g/cm^3	3.07	2.88	2.88	2.78	2.78
Volume expansion, mm	23	6	4	3	3
Setting time Start.-End, h	2.00-3.20	2.40-3.45	3.10-4.10	2.30-4.05	2.20-3.15
2-day strength, N/mm^2	16.3	14.0	12.0	10.2	9.1
7-day strength, N/mm^2	28.7	23.5	21.0	19.9	18.9
28-day strength, N/mm^2	42.5	36.2	33.9	32.9	30.0
Strength Activity Index (28-day)	100	85.2	79.8	77.4	70.6

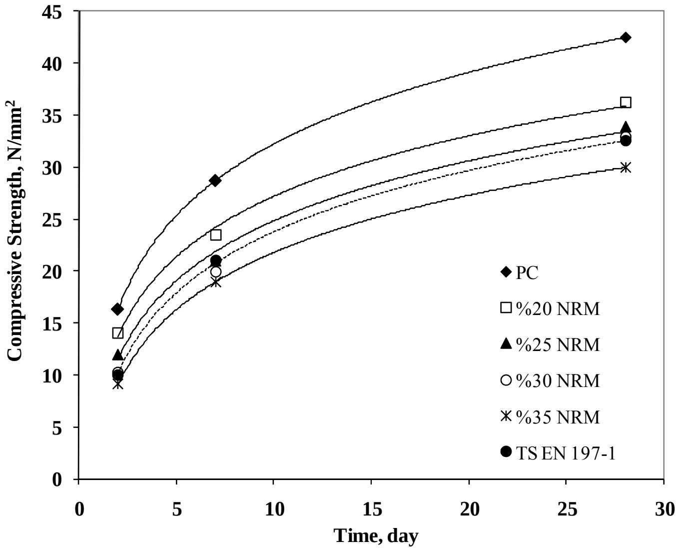

Fig. 4. Development of compressive strength of the mortar samples produced from blended cements containing up to 35% w/w natural raw material over a curing period of 28 days

Table 6. Physical, chemical and mechanical characteristics of different blended cements following TS EN 197-1 [18]

Strength class	Compressive strength, N/mm^2		Setting start (min.)	Volume expansion (mm)	SO_3 (%)	Cl^- (%)	Cement class
	7 days	28 days					21-35% Natural pozzolan
32.5	≥16	32.5-52.5	≥75	≤10	≤3.5	≤0.1	CEM II/B, CEM IV/A, CEM V/A

4. CONCLUSION

Natural pozzolans of pyroclastic origin can be used as an admixture to manufacture blended cements in order to lower the cost of binder and improve the strength and durability in concrete applications. In this study, a natural raw material of volcanic origin was studied for its potential exploitation as a pozzolanic material in the production of blended cement. Mineralogical and petrographic evaluations of the samples have shown that the raw material can be characterised as porphyritic trachyandesite consisting predominantly of plagioclase, hornblende and biotite mainly as phenocrysts within a fine-grained glassy groundmass. Physical, chemical and pozzolanic activity tests on the samples indicated that the raw material meets the requirements for a natural pozzolan for use in the production of blended cements. The raw material, acidic in character with a SiO_2+Al_2O_3+Fe_2O_3 content of 79.86% and a reactive silica content of 33.62% was found to exhibit high pozzolanic activity in compressive and flexural strengths of 2.5 and 3.3 times higher, respectively, than those indicated in TS 25 [30]. These findings have indicated that the material is a suitable material for use in the manufacture of blended cements.

Evaluation of physical and chemical characteristics, and mechanical performances of blended cements produced from raw materials have revealed that the rate of strength development and ultimate compressive strength at a particular curing time decreases with increasing the pozzolan content (20-35% w/w) of blended cements. The setting time tends to increase with the natural pozzolan replacement level of up to 25% w/w, while the addition of natural pozzolan lowers the volume expansion of pastes by 74-87%. It can be deduced that the raw material up to 30% w/w can be added to produce blended cements of the desired quality.

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