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Portland cement structure and its major oxides and fineness

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Abstract. Predicting the compressive strength of concrete has been considered as the initial phase across the cement production processing. The current study has focused on the integration of the concrete compressive strength in 28 days with the mix of the major oxides and fine aggregates as an experimental formula through the use of two types of Portland cement resulting the compressive strength of the concrete highly dependent on time.

Keywords: Portland cement; major oxides; fine aggregates; compressive strength of concrete

1. Introduction

The compressive strength estimation of cement at any time especially across the 28 days has been highly regarded expressing the relationship of the cement compressive strength with its effective chemical and physical properties (Arabnejad Khanouki *et al.* 2010). The experimental relationships between the cement components and its fine aggregates with compressive strength have produced cement with high quality and low energy (Bao *et al.* 2016, Toghroli *et al.* 2017).

According to the literature, Bogue standard has been used to calculate the structural compositions of Portland cements to provide the compressive strength formula regarding any possible errors across the calculation. Portland cement has comprised the fine aggregate including the minerals with low crystallinity difficult to be defined in the recent methods (Kheyroddin 2008d, Sharbatdar 2008, Bazzaz 2018, Paknahad *et al*. 2018, Shariati *et al*. 2011, 2012, 2013, Sinaei *et al*. 2011).

Nevertheless, the science of materials in rigid structural systems has been progressed technologically due to the controlling mineralogical and microstructural mechanisms of the developed design systems. Considering the lack of any initial phase progressing for characterizing, the development of any qualified structural characteristic for any cement paste is impossible. Considering the constituent phases of cement processing, the composition and chemical contents of cement materials have been significantly involved regarding the unknown or esoteric essential factor to qualify the cement (Matschei *et al.* 2007, Gerami 2008, Kheyroddin 2008a, Kheyroddin 2008b, Kheyroddin 2008c, Kheyroddin 2008d, Sharbatdar 2008, Andalib *et al.* 2010, Armaghani *et al.* 2016, Safa *et al.* 2016, Stanojevic *et al.* 2017, Ismail *et al.* 2018, Ji *et al.* 2017, Hosseinpour *et al.* 2018).

Portland cement has been maintained by heating a mixture of limestone and clay or similar compositions with sufficient reactivity in 1450℃ (maximum). On Partial fusion occurring, the clinker micro structures have been produced including 67% (CaO=C), 22% (SiO_2=S), 5% (Al_2O_3=A), 3% (Fe_2O_3=F) and 3% other components with four major phases as 1) alite, 2) belite, 3) aluminate and 4) ferrite (Taylor 1997, Andalib *et al.* 2014, Mohammadhassani *et al.* 2014, Lee *et al.* 2016, Muhammad *et al.* 2016, Heydari and Shariati 2018, Paknahad *et al.* 2018). Alite as the most significant element of the normal Portland cement clinkers has constituted 50-70% tricalcium silicate or C_3S modified in the mortar and crystal structure by ionic substitutions reacting quickly in the presence of water, therefore, the normal Portland cement has achieved more essentiality to reinforce the compressive strength of concrete to gain the maximum possible value in 28 days (Petković *et al.* 2012, Nasrollahi *et al.* 2018). Belite with 15-30% of normal Portland cement clinkers has consisted of the dicalcium silicate or C_2S (Ca_2Sio_4) modified by ionic substitutions presented as β polymorph, which has reacted slowly with water providing lower compressive strength contribution in the first 28 days accompanied by substantial increasing. At the end of the first year, the compressive strengths obtained from pure alite are approximately the

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same under the controlled and comparable conditions. When aluminate has involved 5-10% of the ordinary Portland cement clinkers, tricalcium aluminate C_3A, ($Ca_3Al_2O_6$) has been modified in the mortar or in the structure by ionic substitutions reacting quickly with water and causing undesirable issues (False cement reaction), unless there would be an expert operator to control the unit or a gypsum has occasionally been added to the mortar.

While ferrite has constructed 5-15% of ordinary Portland cement clinkers, tetra calcium aluminoferrite C_4AF, (Ca_2AlFeO_5) has been mainly modified in the mortar by changing the Al/Fe ratio and ionic substitutions. The rate of this reaction with water is not constant mightily due to the differences of the mortar or other characteristics resulting a high or low essentiality across the time.

European Bogue calculation (a standard method based on the elemental component materials of the clinker) has been used to determine the mentioned phases calculating the initial phase compositions by predicting the relative amount of the four major cement phases. While the method is based on the simple chemical assumptions, it has not been taken as an analytical method to measure the actual cement mortar phase compositions; thus it would provide inaccurate outcome due to the achieved equilibrium conditions across the mortar production.

Also the method has not contemplated the incorporation of foreign ions within the concrete structure or other different solid solutions. Therefore, according to a study by (Taylor 1997), the traditional clinker phases have not been defined as pure phases, but included some solid particles. Few studies have been performed by typical cement mortar composition (Crumbie *et al.* 2006) as follows:

- Alite and C_3S: [3(Ca 0.98 Mg 0.01 Al 0.067 Fe 0.0033)][(Si 0.97 Al 0.03)] O_5
- Belite and C_2S: [2(Ca 0.975 K 0.01 Na 0.05 Mg 0.01)][(Fe 0.02 Al 0.06 Si 0.9 P 0.01 S0.01)] $O_{3.9}$
- Aluminate and C_3A: [3(K 0.03 Na 0.06 Ca 2.76 Mg 0.08Ti 0.01)] [(Fe 0.22 Al 1.6 Si 0.18)]O6 Cubic [3(Na 0.292 Ca2.792)] [(Fe0.15 Al 1.725 Si 0.125)] O_6 Orthorhombic
- Ferrite and C_4AF: $Ca_2(AlXFe_2-X)_2O_5$—For example: Ca_2Al Fe 0.6 Mg 0.2 Si 0.15 Ti 0.05 O_5

The study of (Taylor 1997) has shown that the Bogue calculation generally considered with alite and belite has contented on the quantity comparing by graphs and optical microscopy techniques (Crumbie *et al.* 2006). Optical microscopy with graph drawing has been used to provide reliable phase content results for alite and belite (Taylor 1997), however, the aluminate qualification, the ferrite relations and the chemical molecule connection phases by graphing are highly complicated due to the small crystals of the phases within the microstructure.

Additionally, the chemical similarities between the two phases might raise problems when iron or alkali rich orthorhombic C_3A is active preventing an accurate distinguishing between the orthorhombic aluminates phases and ferrite since the crystal structure of ferrite is also orthorhombic and typically dendrite or prismatic in ordinary Portland cement clinkers (Crumbie *et al.* 2006).

Bogue's calculation time (To determine the compressive strength of the cement) has been refined but principally remained unaltered. Because of the extensive stability of the hydrated cement paste systems, using the same approach for pastes is impossible; however, the object has confined the validity. Thus, the quantitative phase composition has been estimated by using Bogue as follows:

- Assume that the compositions of the four major phases are C_3S, C_2S, C_3A and C4AF
- Assume that the Fe_2O_3 occurs as C_4AF
- Assume that the remaining Al_2O_3 occurs as C_3A
- C_3A, C_4AF and free lime deduction from CaO and solve two simultaneous equations to obtain the contents of C_3S and C_2S

This leads to the (1) equations in which CaO has been assumed for free lime

$$\begin{aligned} C_3S &= 4.07CaO-7.6SiO_2-6.72Al_2O_3-1.43Fe_2O_3 \\ C_2S &= 2.87SiO_2-0.75\,C_3S \\ C_3A &= 2.65Al_2O_3-1.69Fe_2O_3 \\ C_4AF &= 3.04\,Fe_2O_3 \end{aligned} \tag{1}$$

Additionally, the main parameters in the strength development of Portland cement are 1) phases, 2) fineness, 3) hydration time, 4) hydration temperature, 5) total porosity, and 6) pore structure, therefore, the degree of cement hydration has directly affected the porosity and strength. At ordinary temperatures (20-28 degree), rapid hardening Portland cement with higher fineness has been hydrated faster than other types (Janković *et al.* 2011). The previous study (Taylor 1997) has shown the effect of main four phases and fineness of cement based on the data obtained from the possibility of expressing the compressive strength by means of mathematical equations analysis. The strength development ratio and the ultimate strength have significant variation in diverse cement minerals. While the strength of C_2S has steadily progressed, the ultimate strength of this phase is like C_3S; in contrast, the strengths obtained by C_3A and C_4AF are significantly low even after a long hydration time.

The compressive strength of cement has depended on the cement fineness (Shariati *et al.* 2010, Shariati *et al.* 2011a, Shariati *et al.* 2011b, Shariati *et al.* 2011c, Shariati *et al.* 2012a, Shariati *et al.* 2012b, Shariati *et al.* 2013, Shariati *et al.* 2015, Shariati *et al.* 2016) providing the notion that the ultimate strength like the strength of the fully hydrated cement paste is independent from the original cement fineness adding that the cement fineness has determined the hydration development leading to the rate of strength development. The hydration and strength development, after short hydration times, have significantly been enhanced by the specific surface area growth. The grinding fineness of the cement (specific surface area) has been correlated effectively with the early age strength development due to the growing hydration ratio along with the cement fineness growth showing that the ultimate strength has also been raised by fine grinding (Hewlett 2003).

Few studies have investigated the relationship between the clinker phase composition and the strength after a hydration time by applying multiple linear regression analysis (R2). Thus, the relationships between the strength and cement composition have been expressed in an equation (type 2) (Safa *et al.* 2016, Tahmasbi *et al.* 2016, Toghroli *et al.* 2016, Khorami *et al.* 2017a, Khorami *et al.* 2017b, Khorramian *et al.* 2017, Mansouri *et al.* 2017, Sedghi *et al.* 2018, Shariati *et al.* 2018, Zandi *et al.* 2018)

$$\sigma_t=\alpha_0+\alpha_1C_1+\alpha_2C_2+\alpha_3C_3 \quad (2)$$

Where σ_t is the strength after the hydration time t, α_0, α_1, α_2, α_3 are constants and C_1, C_2, C_3 are the clinker phases contents. The relationship between the strength and cement composition on 30 Australian cements has been studied (Alexander 1972, Alexander and Ivanusec 1982) . Schramli has declared that C_3A has positive effects on the initial strength and moderate negative effects on the ultimate strength (Schrämli 1978). While the effect of C_3A and the amount of alkali has been studied later (Vonndop 1979), Aldridge has provided other equation (Aldridge 1980) (type 3)

$$\sigma_c=\alpha_0+\alpha_1C_3S+\alpha_2(C3S+C2S)+\alpha_3C_3A+ \alpha_4SA^* \quad (3)$$

* SA = specific surface area

The relationship of SO_3 with the strength has been studied by (Alexander and Ivanusec 1982). Aldridge has studied the strength of the cement composition (Aldridge 1980). Alexander has also studied the relationship of the strength, composition and cement fineness (Alexander 1972). The linear equations expressing the relationship between the compressive strength and clinker phase composition have been depicted in Eqs. (4) to (8), where k is constant and SA is surface area (cm^2/gr). The process has been analyzed by using the multiple regression analysis evaluating the experimental data of different cement sets.

While the Eqs. (4) and (5) have been offered by (Aldridge 1980), the Eqs. (6)-(8) have been provided by Alexander (1968,1972,1982).

$$\sigma_{28}(MPa)=2.5+0.45C_3S+1.14C_3A+0.045SA \quad (4)$$

$$\sigma_{28}(MPa)= -83+0.32C_3S+1.2(C_3S+C_2S)+1.14C_3A+0.49SA \quad (5)$$

$$\sigma_{28}(psi)=200C_3S+90C_2S+405C_3A, \quad (w/c=0.35)$$
$$\sigma_{28}(psi)=90C_3S+0C_2S+405C_3A, \quad (w/c=0.50) \quad (6)$$
$$\sigma_{28}(psi)=40C_3S+0C_2S+80C_3A,$$

$$\sigma_{28}(MPa)=5.14+0.386C_3S-0.087C_2S+2.980C_3A+0.64C_4AF+0.0617SA \quad (7)$$

$$\sigma_{28}= 1.97+0.188C_3S+0.279C_3A+0.0385SA \quad (8)$$

Various units and methods have been applied in data comparing of the strength. Phatak and Deshpande have studied the prediction of 28 days compressive strength of 53 grade cements by dimensional analysis (Phatak and Deshpande 2005) resulting the dimensional analysis method adequately efficient to formulate the mathematical models regarding various materials in two experimental results. Additionally, in-depth analysis has been required to validate the mathematical model. The influence of Portland cement characteristics on the compressive strength up to 28 days applying partial least square (PLS) analysis has investigated by (Svinning *et al.* 2008) presenting the origin of the parameters as mineralogy and superficial microstructure. Meanwhile, Wronged Bogue has been commonly used in cement phases.

2. Experimental study

2.1 Materials

Portland cement types II, V of Tehran and Sofiyan cement plants have been applied in the current study.

2.2 Test procedure

The chemical analysis of the samples has been examined by X-ray and standard ASTM methods. While the major oxides of the samples including CaO, SiO_2, Al_2O_3, Fe_2O_3, have been measured without using European Bogue method, the sample Fineness has been measured by Blain method, then ASTM C150 has been used to measure the compressive strength of the samples of 28 days with standard materials and conditions (Standard 2009).

"The use of the standard Bogue calculation to predict the phase composition of Portland cement clinkers can give serious errors" (Crumbie *et al.* 2006). Additionally, based on Taylor opinion, C_3S, C_2S, C_3A, C_4AF are not the same phases proposed by Bogue. Thus, according to the writer of this study, estimating the 28 days compressive strength of cement is the use of the amount of major oxides (instead of phases) (Taylor 1997).

3. Results and discussion

Conducting a test (X-Ray Diffraction), the relationship between cement major oxides and its fineness with compressive strength cement has been produced by high quality and low energy. Also, the integration of compressive strength at 28 days with major oxides is not linear, and then the effect of CaO and SiO_2 is greater than other oxides adding that Al_2O_3, Fe_2O_3 are effective as well.

The main objective of the study is to propose a formula on the major oxides and fineness with compressive strength (Eqs. (9)).

$78\leq k\leq 82$ and C=CaO%, S=SiO_2%, A=Al_2O_3%, F=Fe_2O_3% and B=Blain (cm^2/gr) (2)

The chemical effect (First term)
Physical effect (second term)

The formula has been formed on the statistical studies and essential coding of a 28-day characteristic resistance. Regarding the variables on the cement phases of the previous research (Bogue's method), the results have not shown a satisfactory result and the difference is more than

Table 1 Experimental and theoretical results for Tehran cement –Type II

TYPE-II (Tehran)	SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	CaO (%)	B (cm^2/gr)	R_{28} (exp.) MPa	R_{28} (formula) MPa	Difference (%)
T2	21.37	4.66	3.97	61.95	2841	42.8	41.4	-3
T2	21.43	4.66	3.88	62.14	2962	43.8	42.0	-4
T2	22.13	4.88	4.07	62.51	2874	41.5	41.9	1
T2	21.25	4.82	4.02	61.93	2811	42.0	41.2	-2
T2	21.91	4.72	3.80	62.88	3021	43.7	42.9	-2
T2	21.06	4.60	3.71	61.86	3165	42.5	42.3	-1
T2	21.06	4.60	3.71	61.86	3021	41.0	41.9	1
T2	21.95	4.84	3.85	62.72	2962	40.7	42.5	4
T2	21.51	4.69	3.81	62.30	3021	42.1	42.3	0
T2	21.95	4.84	3.85	62.06	2933	43.3	41.6	-4
T2	21.51	4.69	3.78	62.39	3230	43.1	42.9	0
T2	21.80	4.75	3.76	62.20	2933	42.5	41.9	-1
T2	21.83	4.64	3.76	61.87	3109	41.8	42.0	0
T2	21.83	4.64	3.75	62.66	2874	41.0	42.4	3
T2	22.21	4.77	3.76	62.81	3021	40.6	42.8	5
T2	21.91	4.87	3.72	62.84	2811	41.2	42.3	3
T2	21.85	4.88	3.73	62.71	3080	42.7	42.8	0
T2	22.07	4.89	3.79	62.80	2765	42.5	42.1	-1
T2	21.68	4.81	3.78	62.57	2765	41.8	41.9	0
T2	21.65	5.01	3.89	62.51	2811	41.2	41.9	2
T2	21.31	4.62	3.86	62.34	2874	42.5	42.0	-1
T2	21.65	5.01	3.89	62.51	2904	42.1	42.1	0
T2	22.09	4.74	3.84	62.87	2715	41.2	42.1	2
Average	21.70	4.77	3.83	62.40	2935	42.1	42.1	0
St.dev.	0.33	0.12	0.10	0.36	134	1.0	0.0	2
Var.	0.11	0.02	0.01	0.13	17980	1.0	0.0	6

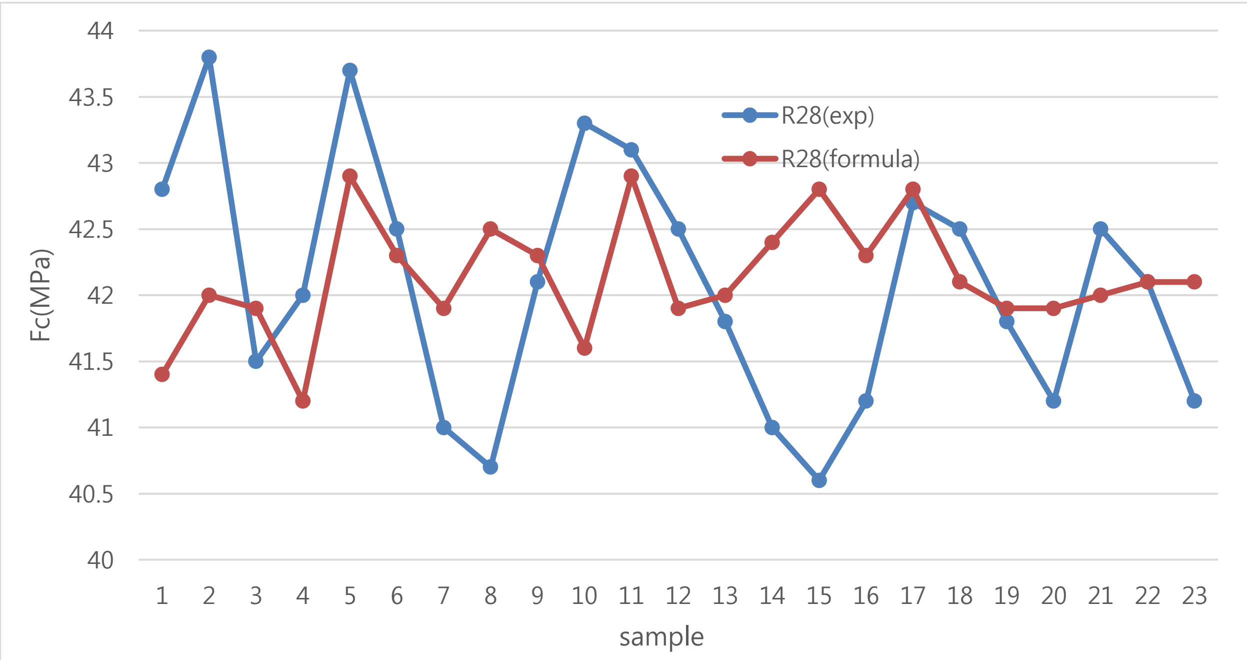

Fig. 1 Comparing the theoretical and experimental results for Tehran cement –Type II

30%. Considering the whole relationship including the samples' major oxides with CaO, SiO2, Al2O3, Fe2O, 3 have been measured and the phases calculation has shown various resistance ratio (over 22%). Finally, regarding the notion of the resistance gap reduction and the easy access of all cement analysis, the focus has been on the major oxides. Taking the variations, the most crucial parameter is the Blain variation of cement leading to the 6 relationships after 6 months resulting the parameter k.

In the final formula with one coefficient, k has been used while its magnitude has depended on the cement type (78-82). Taking the major oxides' percentage and the

Table 2 Experimental and theoretical results for Sofiyan cement –Type II

TYPY II (Sofiyan)	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	CaO %	B (cm^2/gr)	R_{28} (exp.) MPa	R_{28} (formula) MPa	Difference %
T2	23.10	4.23	3.86	63.62	2872	42.7	43.4	2
T2	22.00	4.43	4.85	62.91	2903	42.9	42.4	-1
T2	22.60	4.17	3.92	63.01	3128	43.6	43.4	0
T2	21.70	4.08	3.81	63.25	3167	45.4	44.0	-3
T2	22.70	4.16	4.07	63.33	3048	44.0	43.5	-1
T2	22.60	4.07	3.93	63.13	3157	42.2	43.7	3
T2	21.80	4.69	3.74	63.13	3196	43.2	43.7	1
T2	21.50	4.44	3.46	63.06	3473	45.7	44.6	-2
T2	22.22	4.30	3.80	63.50	3291	44.4	44.5	0
T2	22.20	4.30	3.80	63.30	2960	42.4	43.4	2
T2	22.90	4.07	3.65	63.93	2839	42.1	43.9	4
T2	22.90	4.22	3.55	63.98	2784	42.7	43.8	3
T2	21.60	4.52	3.50	62.91	2817	44.8	42.7	-5
T2	22.70	4.42	3.33	63.43	2966	44.2	43.6	-1
T2	21.80	4.23	3.40	63.68	3356	45.1	45.1	0
T2	21.80	4.20	3.52	63.42	3186	44.8	44.3	-1
T2	21.80	4.44	3.93	63.55	3058	43.9	43.9	0
T2	21.90	4.31	3.84	63.65	3058	44.3	44.1	0
T2	22.00	4.18	3.74	63.82	3167	44.6	44.7	0
T2	22.20	4.11	3.80	63.95	3068	43.3	44.6	3
T2	22.20	4.35	3.84	64.14	2960	43.8	44.4	1
T2	22.20	4.32	3.92	64.48	3310	44.7	45.7	2
T2	21.70	4.63	3.85	63.51	2882	43.3	43.4	0
T2	21.80	4.75	3.85	63.42	2839	43.6	43.1	-1
T2	22.00	4.70	4.00	64.24	2946	44.2	44.0	0
T2	23.00	4.60	3.87	63.79	2966	42.2	43.8	4
T2	22.60	4.97	3.80	63.75	2806	42.7	43.3	1
T2	22.80	4.63	3.71	64.32	2935	42.6	44.4	4
Average	22.23	4.38	3.80	63.58	3041	43.7	43.9	1
St.dev.	0.48	0.24	0.28	0.43	182	1.0	0.7	2
Var.	0.23	0.06	0.08	0.18	32980	1.1	0.5	5

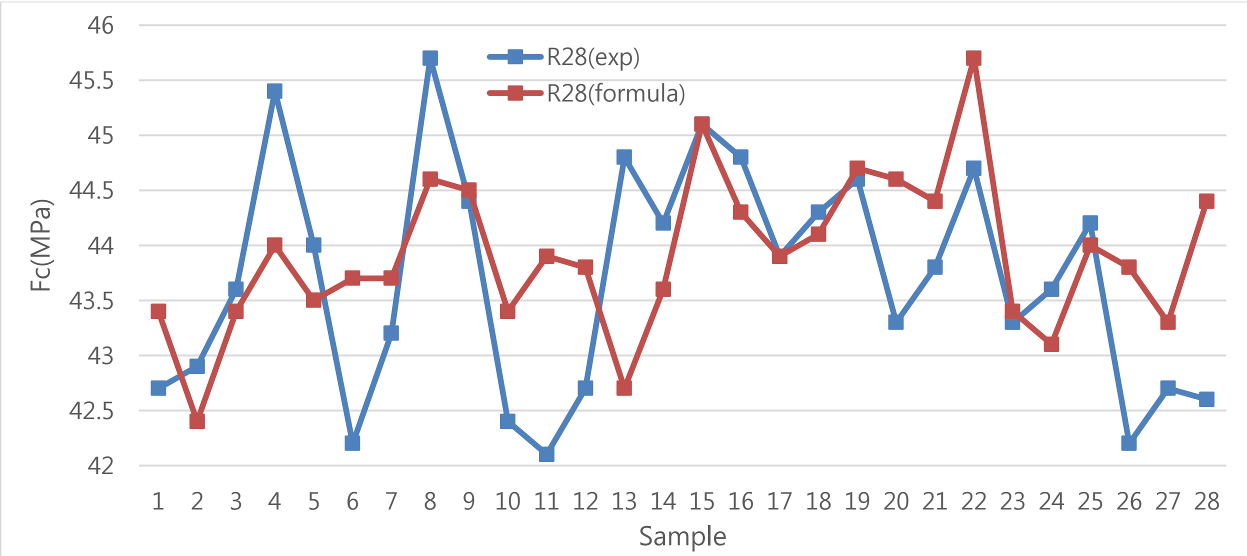

Fig. 2 Comparing the theoretical and experimental results for Sofiyan cement –Type II

fineness (Blain, cm2/gr), the compressive strength (MPa) at 28 days would be accurately estimated. Around 77 experiments from previous tests on the cements Tehran type II, Tehran type V, and Sofiyan type II have been used to verify the proposed formula for the prediction of 28 days compressive strength (Table 1-3) indicating the effectuality of the prediction capability with a gap less than 5% (between two strengths). The current study has proposed the use of major oxides (instead of phases) for estimating the 28 days compressive cement strength.

Table 3 Experimental and theoretical results for Tehran cement- Type V

TYPE V (Tehran)	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	CaO %	B (cm^2/gr)	R_{28} (exp.) (MPa)	R_{28} (formula) (MPa)	Difference %
T5	22.29	4.29	4.98	62.66	2698	41.1	41.5	1
T5	21.30	4.26	4.82	62.35	2749	42.7	41.5	-3
T5	21.98	4.15	4.76	63.15	2904	42.0	42.8	2
T5	22.09	4.00	4.57	63.02	2615	41.0	42.0	2
T5	22.23	3.27	5.14	63.39	2841	42.1	43.1	2
T5	21.92	3.87	4.90	63.46	2904	43.1	43.3	0
T5	22.61	4.00	4.45	63.69	2927	41.2	43.6	6
T5	22.00	3.38	4.87	63.39	2811	41.3	43.1	4
T5	21.19	3.64	4.93	62.85	2749	44.3	42.3	-5
T5	21.95	3.60	4.52	63.47	2615	42.3	42.8	1
T5	22.00	4.11	4.97	63.50	2815	42.5	43.0	1
T5	22.40	3.71	4.41	64.10	2615	43.0	43.5	1
T5	21.31	4.25	4.60	62.86	2904	43.7	42.6	-3
T5	22.69	4.39	4.86	62.69	3021	38.4	42.3	2
T5	22.08	3.85	4.83	63.27	2926	42.0	43.1	3
T5	21.94	3.97	4.67	63.07	3109	41.8	43.3	4
T5	21.37	3.73	4.85	62.53	2443	43.3	41.1	-5
T5	21.59	3.79	4.74	62.66	2824	42.2	42.2	0
T5	21.51	3.83	4.76	62.60	2615	43.2	41.6	-4
T5	21.68	3.76	4.43	62.27	2648	41.8	41.4	-1
T5	21.86	3.72	4.92	62.40	2778	41.2	41.7	1
T5	21.84	3.63	4.86	63.16	2480	42.3	42.0	-1
T5	22.00	3.59	4.92	63.09	2928	42.0	42.9	2
T5	21.99	3.81	4.80	63.33	2962	43.0	43.3	1
T5	21.44	4.00	4.80	62.49	2841	43.6	42.0	-4
T5	22.22	3.86	4.73	63.00	2904	42.4	42.7	1
T5	22.09	3.59	4.62	63.56	2715	42.8	43.1	1
T5	22.40	4.03	4.92	62.98	2841	41.2	42.4	3
T5	22.22	3.97	4.96	63.32	2750	40.9	42.6	4
Average	21.94	3.86	4.78	63.05	2791	42.2	42.5	1
St.dev.	0.39	0.27	0.18	0.45	156	1.0	1.0	3
Var.	0.15	0.07	0.03	0.20	24335	1.0	0.0	7

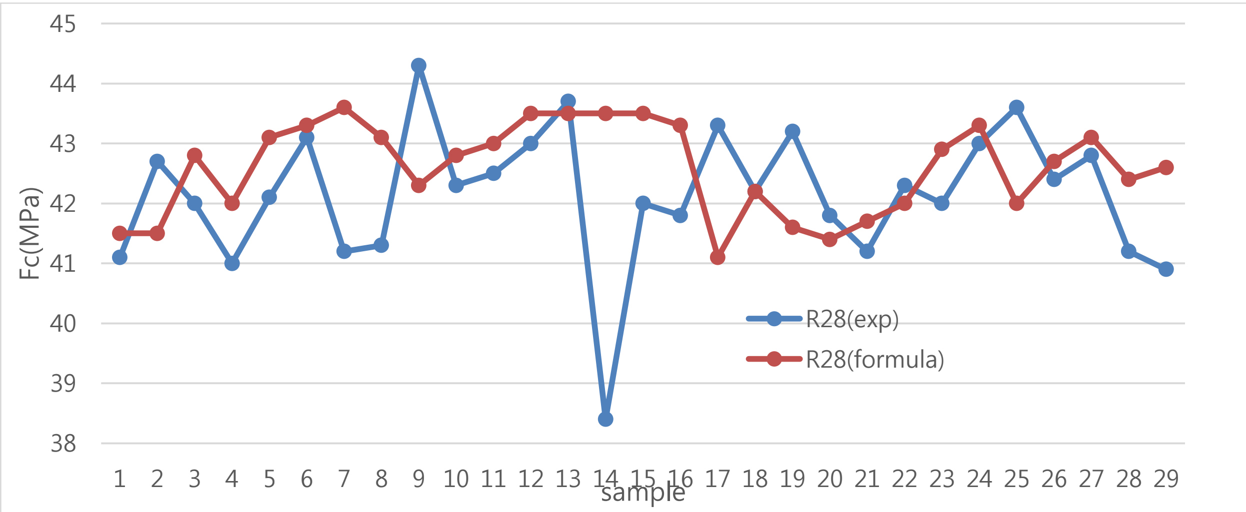

Fig. 3 Comparing the theoretical and experimental results for Tehran cement- Type V

Cement fineness is an important factor of the compressive strength. Then in the proposed formula, the coefficient of fineness is 0.0025 or 1/400 providing around 7 MPa strength, i.e., about 18% of the total 28 day compressive strength.

4. Conclusions

Using the proposed experimental relationship between the cement major oxides and its fineness with compressive strength, cement would be produced in high quality, low energy and low cost. The proposed experimental formula has shown the effect of CaO and SiO_2 greater than the other oxides. Considering the major oxides value and cement fineness, the compressive strength has been gained with acceptable precision. Also, the cement compressive strength has been achieved in the major oxides and fineness function, adding that the effect of other chemical and physical parameters is about 5%. There have been low accuracy and few errors in the previous proposed experimental formulas for estimating the 28 days cement compressive strength since they have been designed by the cement phases and calculated by Bogue method providing not actual phases; consequently, any relationship between the compressive strength and Bogue cement phases has made errors and not recommended.

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