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Quality Assessment of the Various Brands of Portland Cement Available in the Libyan Market

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Abstract
The quality of cement is one of the important factors related to the strength and durability of structural concrete. Different brands of Portland cement (PC) are used in Libya by local contractors for various construction works without prior knowledge of their performances. This study included the determination of the characteristics of different types of cement using chemical and physical tests and effecting a comparison between Libyan cement manufactured by the Libda, Al-Koms, Al-mergeb and Al- borg plants and some imported cements sold in the Libyan market. The percentages of the main compounds (C_3A, C_4AF, C_2S, C_3S) and free lime present in cement and the percentages of the chemical oxide loss on ignition (SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, SO_3) loss on ignition were evaluated. Also, the ratios of the alumina modulus (AM), silica modulus (SM) and lime saturation modulus have been calculated. The moduli of silica and lime saturation in all types of cement were found to correspond with the standard specification of the silica and lime saturation units in all types of cement and generally conform to the standard specifications .Cluster analysis showed four clusters of cement. It was observed that the sample (CE), an Egyptian cement, did not interact with the other types of cement.

1. Introduction
Portland cement is a hydraulic material composed primary of calcium silicates, aluminates, and ferrites. In a rotary kiln, at a temperature reaching 1450◦C, clinker nodules are produced from a finely ground, homogenized blend of limestone, shale and iron ore. The nodules are subsequently ground with gypsum, which serves to control the setting to a fine powder to produce the finished Portland cement. The resulting composition and texture (crystal size, abundance, and distribution) of the clinker phases depends upon the complex interactions of raw feed, the chemical and mineralogical composition, particle size distribution, feed homogenization, and the heating and cooling regime employed in the production [1,2]. In order to simplify these phenomena a standard approach has been proposed for the development of the clinker phases [3]. The ferric oxide (Fe_2O_3) reacts with aluminum oxide (Al_2O_3) and lime (CaO) to form the tetracalcium alumina ferrite (ferrite C_4AF or $Ca_4A_{l2}Fe_2O_{10}$). The remaining aluminum oxide reacts with lime to form the tricalcium aluminates (C_3A or $Ca_3Al_2O_6$). The lime reacts with the silicon oxide (SiO_2) to form two calcium silicate phases, namely dicalcium silicate (a, C_2S or Ca_2SiO_4) and tricalcium silicate (alite, C3S or Ca3SiO5). The important quality defining parameters of Portland cement are its chemical and phase composition. It is necessary to determine a complete mineralogy of clinker cement to correctly understand, interpret and predict the outcome of any plant production process [4,5], Every year a large amount of

Ordinary Portland Cement (PC) is produced and used for the construction of buildings, roads, highways and other local purposes . Libya produces about 7,530 tons of cement annually [6], however, this amount does not meet the growing demand for these materials and cement has been imported from various countries, such as Egypt and Turkey. These imported cements have not been evaluated according to scientific and economic standards dealing with production quality in order to assess their suitability for use in local conditions, which has led to a negative effect on the users, especially in the construction of buildings [7]. The use of poor quality cement in structural and constructional works may cause a disastrous loss of life and property destruction. Hence, the quality assurance of PC has become an important and critical factor. There are several brands of PC available on the market but their chemical compositions are apparently the same. Variations in physical properties occur due to changes in the proportional amounts of the chemical constituents. The chemical analysis of cement is carried out to check whether the supplied product conforms to the standard specifications or not. In the analysis, each oxide is usually expressed as a percentage. Wet chemistry is one of the methods that are employed in the composition determination of cement [8]. In addition to wet chemistry, there are various techniques that are utilized for the composition analysis of cement [9, 10]. As an example, the application of atomic absorption spectroscopy has been reported by a number of researchers for the analysis of cement [9, 11]. Another group has determined the aluminum oxide in Portland cement spectrophotometrically [10]. Among various techniques that are being used for the analysis of the components of Portland cement, the X-ray fluorescence (XRF) technique continues to have a wide popularity. The accuracy of XRF as well as the simplicity of the procedure used is the major reason stated for its use by several investigators [12, 13].

The main objective of the current study was to determine the chemical oxide (SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO and SO_3) compositions using a combination of X-ray Fluorescence Spectrometry (XRF) and classical methods of analysis, namely gravimetric and volumetric methods. The results were used to calculate the major compounds (C_3S, C_2S, C_3A and C_4AF) and the control ratios (LSF, SM and AM) in specimens of Portland cements to assess the quality of the various brands of Portland cement available on the Libyan market and to compare them with the standard specification of Portland cement.

2. Material and Methods

2.1. Sample Collection:

The samples were obtained from purchases in the local markets in the cities of Al-koms, Zlatin, Misurata and Benghazi as shown in table 1.

2.2 Sample Pre-treatment:

All the samples collected were ground and sieved through a 2mm mesh sieve. The product obtained after sieving was homogenized and kept in clean labelled polythene bags [14].

Table1: Samples under study

Sample	**code**
Zliten Cement Factory	CZ
Al-merqeb Cement Factory	CM
Lebda Cement Factory	CL
Souk Al-Khamis Cement Factory	CK
Al- borg Cement Factory	CB
Egyptian Cement Source	CE
Turkish Cement Source	CT

2.3 Chemical Tests

2.3.1 Gravimetric and Titrimetric Method (Classical Methods)

• **Loss on Ignition**(LOI) One gram (1g) of each sample was weighed into a cleaned weighed platinum crucible and placed in a muffle furnace at a temperature set between 900-1,000°C for one hour. After this time, the crucible was removed from the furnace and kept in a desiccator until it had completely cooled down. Then weighed and the weight noted down. The difference in the final weight and the initial weight of the sample gave the loss on ignition. This process was repeated for all the samples.

• **Silicon Dioxide** (SiO_2) A known sample weight was taken to which was added ammonium chloride, hydrochloric acid and nitric acid were added. The solution was vaporized and the silica was washed several times, filtered and the filtrate was saved for the alumina analysis. The filter paper containing the sediment was placed in a weighed platinum crucible and burned in a thermal oven at a temperature of 1100-1200 ° C for one hour then the crucible was weighed again after cooling [14, 15].

• **Aluminum Oxide** (Al_2O_3) Ammonium hydroxide was added to a portion of the filtrate acquired from the silica determination and this precipitated aluminum and iron as the hydroxides, which are filtered, burned and then weighed as the combined oxide [14-16]. The combined oxide is the combination of both iron oxide and aluminum oxide, combined oxide R_2O_3

$R_2O_3 = Fe_2O_3 + Al_2O_3$

$Al_2O_3 = R_2O_3 - Fe_2O_3$

• **Iron Oxide** (Fe_2O_3) is reduced to ferrous oxide using stannous chloride then titrated with a standard solution of potassium dichromate. The determination of iron oxide could then be applied to the combined oxide determination from the previous step to evaluate the aluminum oxide percentage (Al_2O_3) [14].

• **Calcium Oxide** (CaO) was determined by the addition of ammonium oxalate to a portion of the filtrate from the determination of silica. Calcium oxalate is deposited as a precipitate and can be acidified and titrated with potassium permanganate to give the oxalic acid determination and thence the calcium oxide percentage [14, 16]

Magnesium Oxide (MgO)
To the filtrate obtained from the deposition of calcium oxalate was added ammonium phosphate (dibasic) [$(NH_4)_2HPO_4$] and ammonium hydroxide. Magnesium is precipitated as magnesium phosphate, dried and then weighed in the form of ($Mg_2P_2O_7$) from which the percentage of magnesium oxide can be calculated [16].

• **Sulfur Trioxide (SO_3)**
Sulfate is precipitated by adding barium chloride to an acid solution of the cement. This is filtered and dried as barium sulfate ($BaSO_4$) from which the equivalent percentage SO_3 can be calculated [15].

2.3.2 X – Ray Fluorescence Spectrometry (XRF)
10g of each sample was weighed into a weighed boat to which four to five tablets of solidified ethylene glycol were added and then ground together with the aid of an automated milling machine. The ground mixture is further pelletized in a press with the aid of a pelletizing machine inside a ring after which the ring was examined by the XRF instrument [17].

3. Results and discussion

Evaluation of the characteristics of the available brands of Portland cements is necessary in order to ascertain the product quality of the brands. The experimental results of the elemental oxide percentages are presented in Tables (2, 3)

Table2: Percentage composition of the major chemical constituents for the studied specimens of cement

Samples	LOI(%)	SO_3(%)	SiO_2(%)	Al_2O_3(%)	Fe_2O_3(%)	CaO(%)	MgO(%)
CZ	1.79±0.21	2.45±0.22	21.66±0.40	5.30±0.18	3.41±0.10	62.90±0.41	1.74±0.09
CM	2.14±0.27	2.60±0.28	21.09±0.41	4.73±0.36	4.23±0.31	63.63±0.41	1.19±0.11
CL	1.24±0.45	2.04±0.23	21.20±0.61	5.40±0.21	3.99±0.24	63.95±0.80	2.05±0.22
CK	1.58±0.25	2.22±0.33	20.86±0.47	5.93±0.21	2.66±0.19	62.94±0.49	2.35±0.31
CB	1.15±0.02	2.25±0.10	21.73±0.09	4.92±0.03	3.62±0.04	62.50±0.13	1.75±0.02
CE	10.61±0.01	2.91±0.01	16.76±0.05	4.91±0.02	3.14±0.01	63.95±0.01	1.64±0.03
CT	2.52±0.02	2.30±0.01	20.77±0.01	5.91±0.01	2.80±0.10	63.00±0.01	1.84±0.03
L.S.S 340-97	**3>**	**3>**	**20-24**	**4-6**	**2-4**	**62-69**	**1-3**
B.S 12-78	**5>**	**3.5>**	**21.19**	**-**	**3.27**	**64-66**	**-**
ASTM C150-07	**3>**	**2.90>**	**19-23**	**2.5-6.0**	**0-6**	**61-67**	**6>**

3.1 Percentage of Loss on Ignition

The percentage loss on ignition in the cement samples studied CT, CE, CB, CK, CL, CM and CZ are (2.52 ± 0.02, 10.61 ± 0.01, 1.15 ± 0.02, 1.58 ± 0.25, 1.24 ± 0.45, 2.14±0.27, and 1.79±0.21, respectively. All the samples were found to lie within the standard specification limits, except cement sample CE which was found to be above the standard specifications maximum limit as shown in Table (2) and Figure (1). The range obtained in this study conforms to the results obtained by Omoniyi and Okunola [18].

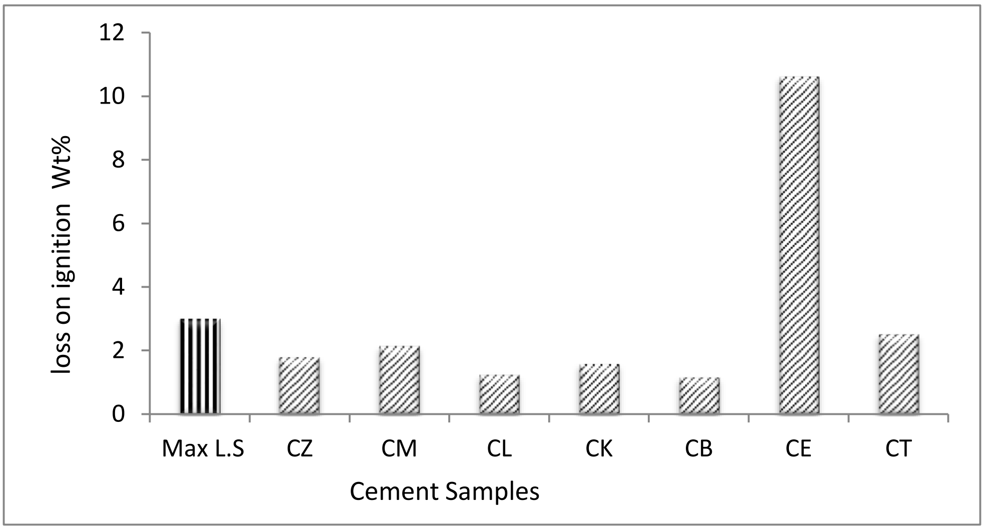

Figure 1: Loss of ignition for studied cements

3.2. Percentage of Oxides in Cement

The concentration of oxides in the different cement brands available in the Libyan market is presented in Table (2). The various percentage oxide compositions (CaO, Fe_2O_3, Al_2O_3, SiO_2, SO_3 and MgO) are presented graphically in Figure (2) followed by a detailed discussion of the results. Figure (2) shows the variation of chemical oxide compositions in different cement brands available in Libya compared to the Libyan Standard Specifications (L.S.S) univalent to the American (ASTM C150) and British (B.S12). The percentage of CaO samples studied were according to standard specification in ordinary Portland cement as shown in Table (2). The range obtained in this study conforms closely to the results obtained as reported by Al-Khatib [19].

The percentage of SiO_2 in the cement samples CT, CE, CB, CK, CL, CM, CZ, where the values ranged from 16.76 % to 21.73 % , which is within the specification limits, with the exception of the CE cement , are fall below the range as shown in Table (2). The results of this study on SiO_2 were in agreement with the previous work of Omoniyi and Okunola [18].

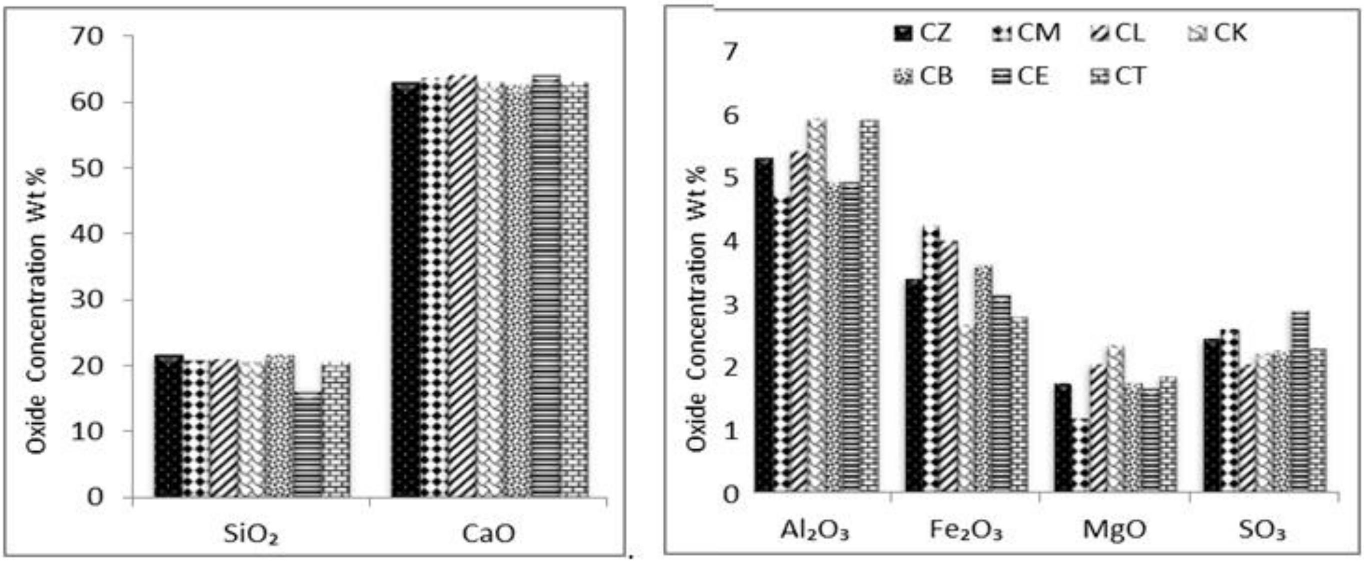

Figure 2: Variation of chemical oxides composition in studied cement

The percentage of magnesium oxide (MgO) is lower than that of other major standard specifications, as shown in Table (2). From these results the percentage of Al_2O_3 in the cement samples CT, CE,CB ,CK,CL, CM,CZ are 5.91±0.01, 4.91±0.02, 4.92±0.03, 5.93±0.21, 5.40±0.21, 4.73±0.36, 5.30±0.18, respectively, All the values fall within the Libyan (L.S.S) and American (ASTM C150) Standard Specifications Table(2). The result is similar to those reported by Fares et al., [20]. The percentage SO_3 in the cement samples studied range in value from 2.04 % to2.91 %, which is within the specification limits as shown in Table (2). From the results the Fe_2O_3 percentage of all samples fall within the American (ASTM C150) Standard Specification, however, CM above the range (L.S.S) Standard Specifications in ordinary Portland cement as shown in Table (2), The result is similar to those reported by Elbagermi *et al.*, [21] for oxides available in cement where ranged from 1.19 % - 2.35%. All the samples conform to the Libyan (L.S.S) and American (ASTM C150) Standard.

Table 3: Chemical composition of Portland cement using classical and XRF methods

X- Ray Fluorescence (XRF)			Classical Method			Parameter (%)
CT	CE	CB	CT	CE	CB	
2.53±0.02	2.91±0.01	2.25±0.01	2.30±0.01	2.90±0.03	2.37±0.04	SO_3
5.91±0.09	4.91±0.02	4.92±0.03	5.90±0.01	4.51±0.08	5.63±0.04	Al_2O_3
2.80±0.1	3.14±0.01	3.62±0.04	2.80±0.02	3.14±0.04	3.55±0.05	Fe_2O_3
1.48±0.03	1.64±0.03	1.75±0.02	1.85±0.03	1.67±0.04	1.60±0.02	MgO
20.77±0.01	16.76±0.05	21.73±0.09	20.76±0.04	16.72±0.03	21.76±0.03	SiO_2
63.00±0.02	63.95±0.03	62.5±0.02	63.01±0.01	63.62±0.03	63.10±0.02	CaO

The results of statistical analysis (T-Test) showed that there were significant differences between the percentage of chemical oxides obtained using XRF method and a classical method of the same samples of the studied cement, except the percentage of iron oxide (Fe_2O_3) for samples CT and CE which no significant differences were observed as shown in table 3.

3.3 Quality of Cement

The major constituents of Portland cement are tricalcium silicate [$3CaO·SiO_2$], dicalcium silicate [$2CaO·SiO_2$], tricalcium aluminate [$3CaO·Al_2O_3$] and tetracalcium aluminoferrate [$4CaO·Al_2O_3·Fe_2O_3$], the shorthand notation for these compounds in the global cement industry is C_3S, C_2S, C_3A and C_4AF respectively [22]. The compositions of the constituents are used to determine the quality of the Portland cement.

Table 4: Analysis on variance (Anova) on mineral composition and the quality factors of the cement samples studied according to standard specification notation in ordinary Portland cement as shown in Table1.

Samples	C_3S(%)	C_2S(%)	C_3A(%)	C_4AF(%)	LSF	AM	SM
CZ	43.8±4.52 ^{a}	28.60±4.21^{a}	8.29±0.49^{a}	10.32±0.31^{a}	0.88±0.02 ^{a}	1.56±0.07^{a}	2.49± 0.07^{a}
CM	53.55±3.99 ^{b}	20.07±3.97^{b}	5.36±0.89^{b}	12.88±0.93^{b}	0.91±0.02^{b}	1.12±0.09^{b}	2.36 ± 0.16^{b}
CL	51.37±2.46^{bc}	22.10±5.69^{bc}	7.58±0.52^{c}	12.12±0.74^{c}	0.91±0.03^{bc}	1.36±0.07^{c}	2.26 ± 0.15^{c}
CK	47.62±5.08^{d}	23.94±5.04^{cd}	11.16±0.69^{d}	8.10±0.57^{d}	0.91±0.02^{bcd}	2.23±0.17^{d}	2.43± 0.09^{ab}
CB	43.57±0.71^{ae}	29.43±0. 70^{ae}	6.92±1.55^{e}	11.02±0.12^{e}	0.88±0.00^{ae}	1.36±0.02^{ce}	2. 55 ±0.01^{ae}
CE	74..22±0.53^{f}	8.02±0.02^{f}	6.64±0.24^{cef}	9.58±0.29^{f}	1.03±0.02^{f}	1.42±0.02^{cef}	2.16 ± 0.02^{cf}
CT	48.26±0.63^{acd}	23.12±0.19^{bcd}	10.92±0.22^{d}	8.54±0.22^{d}	0.92±0.02^{bcd}	2.11±0.03^{g}	2.36±0.02^{bcd}
LSS 340-97	60>	10-30	6 <	13>	0.66-1.02	1.2-2.2	2-3.2
BS 12-78	40 – 80	0 – 30	7 – 15	4 – 15	0.66-1.02	1.4-3.5	1.5-4.0
ASTM C150-07	45-65	7-32	8-12	8-11	-	-	-

Figures in the same column having the same superscript are not significantly different (P> 0.05). Figures in the same column having different superscripts are significantly different
SM: Silica Models, AM: Alumina Models, LSF: Lime saturation Factor.

The quality of the cement products were calculated from the oxide concentrations of the cement using the Bogue formulae [1, 3, 5]. The formula is as follows: [23].

C3S = $[(4.071 \cdot CaO) - (7.6 \cdot SiO_2) - (6.718 \cdot Al2O3) - (1.43 \cdot Fe_2O_3) - (2.852 \cdot SO_3)]$…...eq.1

C2S = $[(2.867 \cdot SiO_2) - (0.7544 \cdot C_3S)]$………………………………….………… eq.2

C3A = $[(2.650 \cdot Al_2O_3) - (1.692 \cdot Fe_2O_3)]$……………………………………………. eq.3

C4AF = $[3.043 \cdot Fe2O3]$……………………………………………………….. eq.4

The Bogue calculation is a means of estimating the quality of the cement based on its oxide composition. Notwithstanding, the knowledge of the potential phase compositions is also important in assessing the properties of a cement [24] Based on the concentrations of the chemical oxides Table (2) and on the results of the Bogue calculation, the percentage proportions of C_3S, C_2S, C_3A and C_4AF in CT, CE,CB,CK,CL,CM,CZ were calculated. The results are presented in Table (3) and Figure (3). The percentage of C_3S in cement samples studied were found to lie within the specification limits, except cement sample CE which was found to be above the ASTM150 maximum limit of 45-65% and above the Libyan Standard Specifications maximum limit of 60%.The percentages of the C_2S component for the cement types considered in the present study are given in Figure (3). There were some differences to be noted in the C_2S ratio, where the CE specimen shows a slightly lower value for this C_2S ratio in ordinary Portland cement according to the specification limits, due to the high ratio of CaO of the samples CM, CL and CT illustrated some similarity in the C_2S ratio but the CB, CZ specimens exhibited the highest values. All the samples, with the exception of CE cement, are within the Libyan (L.S.S) and American (ASTM C150) Standard Specifications, however, CE fell below the range as shown in Table(3) . Figure (3) presents the C_3A ratio in the cement samples studied ,where the values ranged from 5.36% to 11.16% , which is within the specification limits in ordinary Portland cement , except that the cement sample CM fell below the range as shown in Table (4). For C_4AF, all the cement brands are within the Standard Specification limits.

Variation in the chemical constituents of cement affects the hardening/hydration, setting time, corrosion resistance and the colour of the cement [2,25] Concrete made with cement having a relatively high C_3S content will tend to gain more strength and produce more heat of hydration at the earlier stages, usually within the first week of placement. Concrete made with cement having a relatively high C_2S content tends to gain more of its strength at later stages, perhaps up to four weeks after placement. Cements with a relatively high C_3S content will have a relatively low corresponding C_2S content. Concrete made with cement having a relatively high C_3A content tends to exhibit faster setting times; more heat generation occurs in the initial few hours after the concrete is poured [26]. Both C_3A and C_4AF components make little contribution to the strength of the cement [27,28].

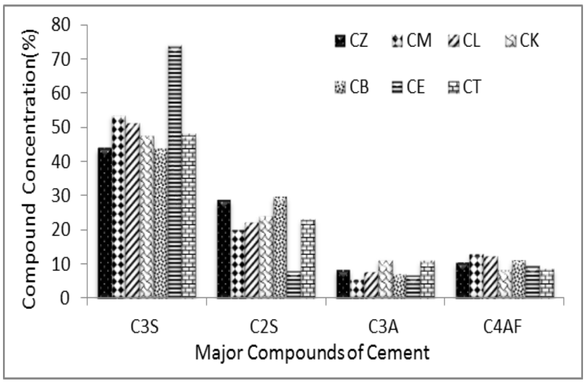

Figure 3: Variation of cement potential in different brands of cement

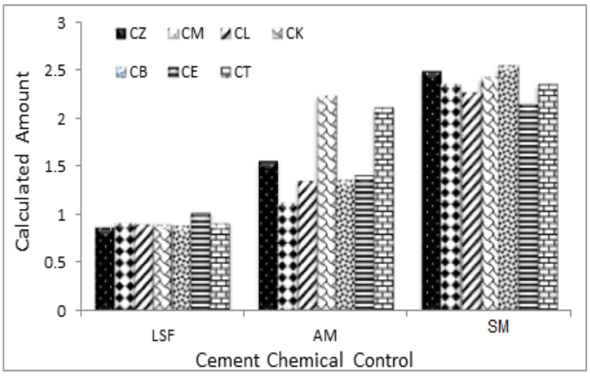

Figure4: Variation of cement ratio in different cement brands

The lime saturation factor, the silica and aluminum ratios (formulae stated below) are important factors for chemical control in cement setting [26]. Based on the formulae, the lime saturation factor, silica and aluminum ratios were calculated from the percentage oxide compositions. The results obtained are presented in Figure (4). The formula for this calculation is as follows:[26].

Lime Saturation Factor (LSF) = $\{(CaO)/ [(2.8SiO_2) + (1.2Al_2O_3) + (0.65Fe_2O_3)]\}$ ……eq.5

Aluminum Model (AM) = $[(Al_2O_3)/ (Fe_2O_3)]$….. eq.6

Silica Model (SM) = $[(SiO_2)/ (Al_2O_3 + Fe_2O_3)]$……………………………………....eq.7

From the calculated results all cement samples studied have LSF, AM,SM values which lie within the range of the Standard Specifications, except LSF for the CE cement which lies above the range of the Standard Specifications Table(4), Figure(4).

3.4 Results of cluster analysis of cement properties studied

After applying the cluster analysis method to the studied cement data, several conclusions can be made.The data from the samples of cement studied using the process of clustering method of single link showed the existence of four groups (clusters) of the cement. According to this method, the first group included cements CZ and CB. The second group included cements CM and CL, the third group included cements CK and CT, while the fourth group consisted of cement CE. It is possible that the quantitative ratios of raw materials are different for the CE cement than for the other types. The chemical properties of cements CZ, CB are very similar and similar to the cement properties of CM, CL. We notice that the CZ cement plant and the CB plant are located within one geographical area in the Zliten region whereas the CM and CL plants are located in a different geographical area (Al-Komsthe). With similar properties of the cements CT, CK we can predict that the raw materials have a similar geological structure and composition and that the surrounding environmental conditions are very similar.

The most important thing to emerge is the cement specificity of CE, where it was observed that it did not interact with any other type of cement under study as shown in Figure (5), this may be due to the different geographical nature of the region providing the raw material for this type of cement.

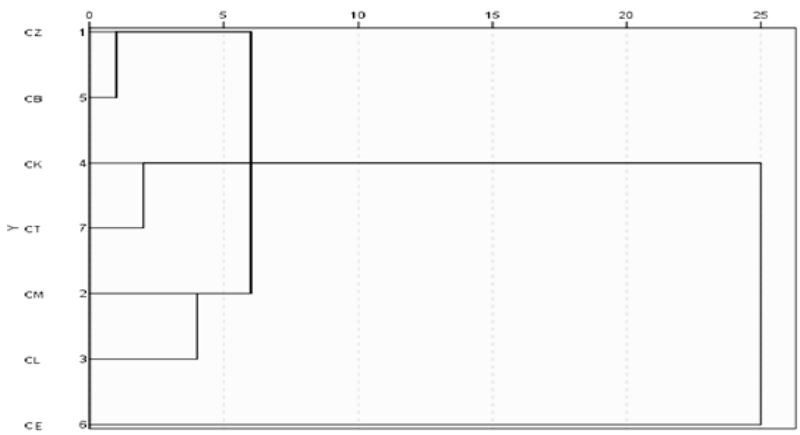

Figure 5: Results of the cluster analysis of the studied cement

Conclusion

The result of the analysis indicates that cement samples under study are generally good for concrete work especially where no other special properties are required. Cluster analysis showed four clusters of cement. It was observed that the sample (CE), an Egyptian cement, did not interact with the other types of cement.

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