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Determination of Major Oxides Percentages in Portland Cement of Some Sudanese Cement Manufactories

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Abstract: In this quality control study, four samples of Portland cement from four Sudanese factories (Atbra, Barbar, Sakhar and Elshamal) were subjected aiming to assess the percentage of mainly oxides in the Portland cement. The study revealed that the percentages of minerals oxides; Calcium oxide, Silicon dioxide, Aluminum oxide, Ferric oxide and Magnesium oxide of the four factories were in the specified limit of quality control according to the American Society for Testing and Materials - Cement (ASTM C150).

Keywords: Portland Cement, Limestone, EDTA, Clinker

1. Introduction

Cement is a major industrial commodity that is manufactured commercially in over 120 countries. [1] Mixed with aggregates and water cement forms the ubiquitous concrete is used in the construction of buildings, road, bridges and other structure. In countries even where wood is in good supply concrete also features heavily in the construction of residential buildings. In fact twice as much concrete is used in construction around the world than the total of all other building materials. [2] There is huge requirement of cement in construction work all over the world. The quality of cement as per standard specification is very important for the cement manufacturing industries as well as for the civil construction industries for making solid and long life structure. The main constituent of the cement and clinker is calcium oxide (CaO) which is the major factor for cement quality. [3]

Cement is a defined chemical entity formed from predetermined ratios of reactants at a fairly precise temperature. Ordinary Portland cement results from the calcination of limestone and silica in the following reaction; [3]

$$5CaCO_3 + 2SiO_2 \longrightarrow (3CaO, SiO_2)(2CaO, SiO_2) + CO_2$$

Limestone Cement carbon dioxide

In addition to gypsum ($CaSO_4.2H_2O$), the cement composed of some minerals oxides; Lima (CaO), Silica (SiO_2), Alumina (Al_2O_3), Magnesium oxide (MgO) and ferric oxide (Fe_2O_3). Because of the complex chemical nature of these oxides in the cement industry, they have shorthand (C, S, A, M and F respectively) are used to denote the chemical compound. [4, 5]

Cements consist of different materials and are statistically homogeneous in composition resulting from quality assured production and material handling processes. Portland cement clinker is made by sintering a precisely specified mixture of raw materials (raw meal, paste or slurry) containing elements, usually expressed as oxides, CaO, SiO_2, Al_2O_3, Fe_2O_3 and small quantities of other materials. The raw meal, paste or slurry is finely divided, intimately mixed and therefore homogeneous. [6]

Ability to calculate compositions of cements in terms of the amounts of the main compounds present provided a valuable new tool for explaining, or predicting, differences in engineering performance among Portland cements. The ability to calculate the amounts of the major compounds in a clinker or cement had important implications. [7]

One of the analytical techniques to determine calcium oxide is complexometric titration with EDTA. The method uses a very large molecule called EDTA which forms a complex with calcium and magnesium ions. [3] A blue dye called Eriochrome Black T (EBT) is used as the indicator. This blue dye also forms a complex with the calcium and magnesium ions, changing colour from blue to pink in the process. The dye–metal ion complex is less stable than the EDTA–metal ion complex. For the titration, the sample solution containing the calcium and magnesium ion reacts with an excess of EDTA. The indicator is added and colour changes to blue as all the Ca^{2+} and Mg^{2+} ions present are complexed with the EDTA. The main reaction is:

$$Ca^{2+} + EDTA^{4-} \longrightarrow [Ca\text{-}EDTA]^{2-}$$

2. Materials and Methods

2.1. Sample

30g of Portland cement from each cement factory of study factories were obtained and subjected to studied and analyzed.

2.2. Chemicals and Reagents

- Concentrated hydrochloric acid.
- Ammonium nitrate, Ammonium chloride, Ammonium oxalate and Ammonia solution.
- Methyl red indicator.
- Buffer solution (pH =4) and Buffer solution (pH=10).
- Salicylic acid.
- Ammonia solution.
- EDTA.

2.3. Equipments and Apparatus

- Sensitive balance.
- Furnace.
- Beakers.
- Measuring cylinder.
- Volumetric flask.
- Ash less filter papers.
- Crucible.
- Burette.
- Funnel**.**

2.4. Determination Procedures of Oxides

2.4.1. Determination Procedure of Silicon Dioxide (Silica)

1g of cement for each sample was weighted in 100 ml beaker, 10 cm^3 of concentrated hydrochloric acid was added to it and then was heated on electric hotplate to dryness (in hood). Another 6 cm^3 of the acid and 30 cm^3 of distilled water were added respectively, the mixture was heated at boiling point.

The hot solution was filtered through ash less filter paper, the precipitate was washed with 30 cm^3 of hot distilled water (the filtrate was kept to estimate the iron and aluminum). The precipitate and filter paper was conveyed to clean and weighted crucible. The crucible and its contents were burned to 800°C for 50 minutes and the crucible was leaved to cool in the dryer and then was weighted it.

Calculation:

$$\text{Percentage of silicon oxide} = \frac{Weight of si_2o}{Weight of sample} \times \%100$$

2.4.2. Determination of the Combined Oxides (Al_2O_3 & Fe_2O_3)

The filtrate remaining after precipitation of silica was diluted to about 200ml in a beaker; 2g of ammonium chloride was added and drops of methyl red indicator, was heated to boiling. Ammonia solution was added gradually until the color turns to yellow. The beaker was leaved for 10 minutes. The solution was filtered through ash less filter paper, the precipitate and filter paper was washed with ammonium nitrate solution 2% (*the filtrate was kept to estimate the calcium ions*). The precipitate and filter paper was conveyed to clean and weighted crucible. The crucible and its contents were burned at 800°C for 50 minutes, the Crucible was leaved to cool in the dryer and then was weighted to determine of combined oxides.

Ferric oxide percentage:

1g of cement was weighted in a 100 ml beaker, 5 cm^3 of concentrated hydrochloric acid was added to it, was steered well until disappearing of the green color then 10ml of distilled water, the solution was transferred quantitatively to 100 ml volumetric flask, then was purred in a beaker and leaved to settle down. 20ml from the clear solution was pipette in volumetric flask; 5ml buffer solution and 1ml of salicylic acid was added. The mixture was titration against standard EDTA solution (0.01M) until the end point. The titration was repeated until two consecutive reading that equal. From titration, number of moles and weight in g were calculated and then the percentage was calculated.

Aluminum oxide percentage:

Aluminum oxide percentage = combined oxides percentage –ferric oxide percentage.

2.4.3. Determination of the Calcium Oxide Percentage

The filtrate remaining after precipitation of Iron III and Aluminum III was acidified, and heated to boiling. By measuring cylinder 40ml of hot ammonium oxalate 4% was added and ammonia solution was added gradually until the color turns to yellow. The beaker was leaved for 10 minutes. Then the solution was filtered through ash less filter paper; the precipitate and filter paper was washed with ammonium oxalate solution 0.1%. The precipitate and filter paper was conveyed to clean and weighted crucible, the crucible and its contents was burned at a temperature of 550°C for 50 minutes. Then The Crucible was leaved to cool in a dryer and then was weighted.

Calculation:

$$\text{Percentage of calcium Oxide} = \frac{Weight of si_2o}{Weight of sample} \times \%100$$

2.4.4. Determination of Magnesium Oxide

50 ml was pipette from the filtrate remaining after precipitation of CaO to conical flask and drops of indicator was added and 10 ml of buffer solution was added then 10 ml of ammonia solution was added after that the mixture was titrated against standard EDTA solution (0.01M) until the endpoint. The titration was reading three times or until two

consecutive reading that equal or agree within 0.1 cm difference. From titration, number of moles and weight in g were calculated and then the percentage was calculated.

3. Results

Four samples of portal cement were collected from four Sudanese factories and then were subjected to analysis processes and the oxides percents were determined. The Silicon dioxide, and Calcium, Aluminum, Magnesium and Ferric oxide percentage and the limits of quality control standard were illustrated in table 1.

Table 1. *The percentages of four oxides detected in study samples.*

Factory	SiO_2	CaO	Al_2O_3	MgO	Fe_3O_2
Atbara	20.54%	62.73%	4.44%	2.94%	3.68%
Barbar	21.15%	63.78%	5.00%	2.16%	3.24%
Elshamal	21.65%	65.02%	5.11%	1.87%	3.01%
Sakhr	20.11%	60.44%	4.93	2.8%	3.85%
ASTM C150	19 – 23%	61 – 67%	2.5 – 6.0	0 – 5.0%	0 – 6.0%
P value	0.442	0.263	0.077	0.092	0.659

P =probability value

The variation of different brands of five oxides which were determined in four samples from Sudanese factories were represented in figures 1, 2, 3, 4 and 5.

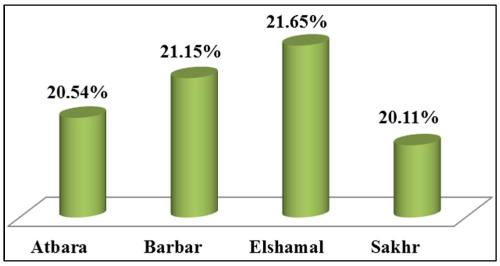

Figure 1. *The percentages of silicon dioxide in four samples.*

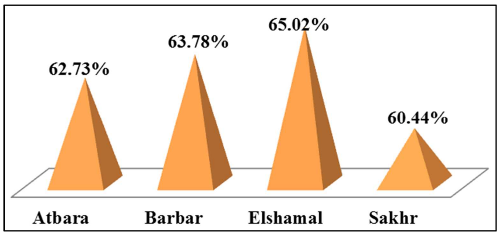

Figure 2. *The percentages of calcium oxide of five factories.*

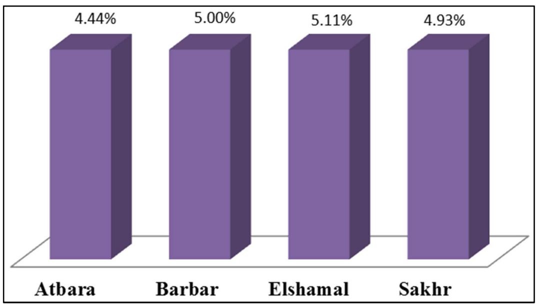

Figure 3. *The percentage of aluminum oxide of study samples.*

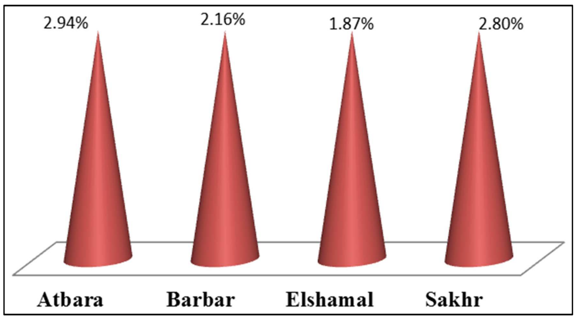

Figure 4. *The magnesium oxide percentage of five factories.*

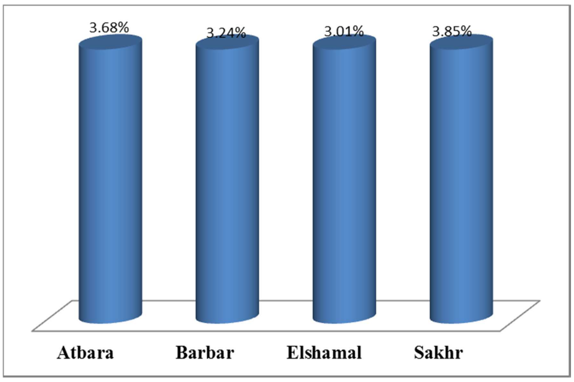

Figure 5. *The percentage of ferric oxide of four factories.*

4. Discussion

The proper lime content is limited due to the lower early strength produced when lime content of ordinary Portland cement is too low and unsoundness when it is too high. [8] our study revealed that the calcium oxide percentage in Sakhar factory was slightly low comparison with other factories, however all percentages of calcium oxide in four samples were in the normal range according to the American standard of Portland cement (p = 0.263). High lime content is associated with early strength whereas slightly lower content of lime favors ultimate strength which develops gradually over a long period time.

By using the American standard limit of silicon dioxide in Portland cement as cut off point, the present study showed that all the factories samples with in specified limit and there is no variation in the different brands.

The study was represented the aluminum oxide of Atbra, Barbar, Elshamal and Sakharas 4,44%, 5.00%, 5.11 and 4.93%respectively and all these values were in the standard limit of ASTM C150.

Due to high magnesia in cement may be detrimental to the firmness of the portal cement, so that amount of magnesia must not exceed 2% especially at late ages. [9] The study showed that the magnesium oxide of four samples within the specified limit (0 – 5.0%) of the ASTM C150 and there was no significant difference between percentages of all samples (p = 0.0658).

Ferric oxide amount was found in specified limit of ferric oxide acceding to the united state standard for Portland cement. Variations in chemical constituents affect the cement properties like, hardening, setting time, corrosion resistance color, etc. [10] Possible and potential source of error in testing might be grade of chemicals, and preparation of reagents and accuracy of method performance which depends on the investigator proficiency.

5. Conclusion

The study compares the quality of different oxides of Sudanese Portland cement factories. The percentage of Silicon dioxide, Calciumoxide, Aluminum oxide, Ferric oxide and Magnesium oxide were calculated in according with united state standard. All the values were found in the specified limit of the standard.

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