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Determination of Calcium Concentration in Different Brands of Portland Cements

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ABSTRACT

The strengths of cement are affected by calcium chloride percentage, especially when the cement composition within the range is encountered in commercial portland cement. The various types of cement are different in the amounts present of CaO, MgO, $Ca(OH)_2$, and $Mg(OH)_2$. Chemical compositions, among other factors are the most performance of producing a high quality of cement. This study aimed to determine the presence of calcium quantity in four different types of cement and study the calcium effect on its chemical constituents by the gravimetric method and titrimetric method using $KMnO_4$/ EDTA as per IS:4032. The results showed the DUNCAN has a higher quantity of calcium compares to other types of cement (RAASI, VISHU, MAHA GOLD) which showed less quantity of calcium exist. The high content of calcium present in cement led to rise in the heat and influences the setting time of alkali-activated. Dicalcium silicate and tetracalcium alumin are the main compounds of portland cement which are responsible for producing a higher heat of hydration, hardness rapidly, and initial set. The uniqueness of this method is a rapid method and gives accurate results in a short interval of time and also avoids the use of a high-temperature muffle furnace.

Keywords
Ordinary Portland cement, Properties, Good quality, Free lime content.

Introduction

Portland cement is the most widely used around the world in the construction industry as a basic ingredient of concrete, mortar, stucco, and non-specialty grout [1]. Portland cement is a complex product obtained mainly from four main constituents; silicon (SiO_2), oxides of calcium (CaO), iron (Fe_2O_3), aluminum (Al_2O_3), and other ingredients (magnesium oxide (MgO) and sodium oxide (Na_2O)). Also, the common materials used to manufacture cement are limestone, shells, and chalk or marl combined with shale, clay. Calcium and magnesium oxides are important constituents controlling the physical performance of portland cements their accurate estimation is very important to assess the quality of cement. Calcium chloride's role is to increase the heat of the cement. However, the part of the total heat developed by the tricalcium aluminate was decreased. It is used as an accelerator in the hydration process of cement, leading to a quick set of concrete and to get high initial strength concrete [2]. Calcium chloride ($CaCl_2$) dissolves easily in water and offers many advantages such as an increase in early strengths and it speeds the rate of the set by improves the workability and improves the strength of air-entrained concrete. Therefore, it is widely used as a concrete accelerator. Concrete acceleration with calcium chloride combines to produce better quality concrete which greatly facilitates completing jobs as quickly and economically as possible [3].

Aluminum chloride, calcium formate, sodium chloride, potassium carbonate, and calcium chloride are used as accelerators for concrete but the calcium chloride was the most widely used. An accelerator leads to increases in the rate of development of certain characteristic properties of cement and concrete. In cement, the strength contributed by two compound tricalcium aluminate and calcium chloride which are decreased the time of set and increased the strength of the

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resulting concrete. Besides, the physical properties of cement were affected by the addition of calcium chloride. However, the optimum amount to be added are different for each types of cement and at various curing temperatures [4].

Portland cement is essentially powdered calcium alumina silicate ($CaO.Al_2O_3.SiO_2$) which sets to a hard mass when treated with water and considered the popular type of cement. Portland cement is produced from a type of rock that includes chemical compounds called calcium carbonate [5]. It is made by pulverizing a mixture of limestone and clay and roasting the powder in a rotary kiln heated by gas or powered coal to a temperature of about 1500 ^oC. This treatment yield sintered lumps called clinker about the size of a marble.

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Cement is produced by applied high temperatures on the raw materials such as (shells, limestone, and marl or chalk combined with shale, clay, slate, blast furnace slag, and silica) treated and reacted at extreme conditions [7]. These processes are called pyro processing, (the raw materials are heated at severe temperatures for solid-state reactions to take place), which utilizes fuel sources such as coal, fuel oil, natural gas, and anything combustible [8].

The cement quality is the main vital factors for the cement manufacturing industries for making solid and long life structure which related to the strength and durability of structural concrete [9,10]. Portland cement is a hydraulic material composed primarily of calcium silicates, ferrites, and aluminates which is used as binding materials of concrete in civil construction industries. Clinker nodules are produced from a finely ground, homogenized blend of limestone, shale, and iron at a temperature reaching to 1500 ^oC, in a rotary kiln [11]. The cement is the main ingredient of concrete structure as a binder in construction work [12].

In general, the portland cement is the most important which is soft kind and form a hard mass when mixed with water due to the chemical combination of the cement compounds with water that yields submicroscopic crystals or a gel-like material with a high surface area [13,14]. However, the hydration properties, constructional cement will lead to set and harden underwater, which is often called hydraulic cement.

The binding materials (bond stones in masonry and tie beams in timber framing) are used as adhesive substances in building and construction [15]. Calcium carbonate is heated sufficiently, it undergoes a decomposition which yields the calcium oxide and carbon dioxide. Pure calcium oxide is a white amorphous substance that emits an intense light called limelight when heated to a high temperature [16]. Calcium oxide is used as a drying agent which can easily react with water. Calcium oxide is known by various names such as lime, quick lime, and slaked lime for the product formed from its reaction with water. Because of the high heat of reaction of quick lime with water, it should always be stored where it cannot come in contact with water, for the slacking reaction generates enough heat to ignite paper or wood [17].

$$CaO + H_2O \longrightarrow Ca(OH)_2$$

$$Ca(OH)_2 \longrightarrow Ca^{2+} + 2OH^-$$

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Usually, the calcium determination take place by treatment with sulphuric and hydrofluoric acids to remove silica then precipitated with Oxalat. Finally, the precipitation treatment with ammonia solution to remove other interfering oxides [18].

Portland cement strength depends on the chemical composition and the fineness to which it is ground. However, the strength developed in the first week of hardening related to the tricalcium silicate C_3S while, the di-calcium silicate C_2S for the subsequent increase in strength. Both chemical compounds (C_3S, C_2S) help to determine the cements setting and its properties [19]. However, the main reason behind the hardening, or setting, of cement is the hydration of the aluminate, calcium silicate, and alumino ferrite minerals. The alumina and iron are created a little direct contribution to strength [20].

Therefore, this study aimed to determine and evaluate the effects of calcium quantity present in four different type of cement and study the effect of calcium content due to its importance in the production of high-quality cement which helps to increase the early strength and high rate of hydration which harden the concrete quicker. This study also compared between the selected type of cements and their chemical composition at pH-10.

Literature Review

Portland cement is produced by grinding and burning a limestone mixture, clay, and shale at approximately 1500°C. Strength of portland cement depending on the conditions of the product curing and the mixture used. Usually, a typical concrete sets in about 6 hours and the strength keep on risen slowly as long as the water is available for continued hydration to dry out then, the strength of concrete growth stop. The C_3S (tricalcium silicate) and the C_2S (dicalcium silicate) are mainly responsible for the strength developed in the first week of hardening and the subsequent increase in strength respectively. The other compounds are present only in little amount which will be create a little direct contribution to strength [21]. Calcium silicate cement was recently used as restorative dentistry and gradually making their way through the various materials while it is true that they have long been used in endodontic [22].

Table 1. Main constituents in a typical portland cement

Compound Name	Formula	Percent%
Tricalcium Silicate	$3CaO \times SiO_2$	50
Dicalcium Silicate	$2CaO \times SiO_2$	25
Tricalcium Aluminate	$3CaO \times Al2O_3$	12
Tetracalcium Aluminoferrite	$4CaO \times Al_2O_3 \times Fe_2O_3$	8.0
Gypsum	$CaSO_4 \times H_2O$	3.5

Portland cement harden comes from series of chemical reactions. These chemical reactions include basic chemical compounds which are listed in Table 1. The reaction proceeds with the addition of water as this process are called hydration. Depending on these reactions which occur

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at different times, quantity, and rate, result in how the portland cement hardens and gains strength [23].

a) Tricalcium silicate (C_3S). considered the main component of portland cement which is responsible for initial set, early strength, hydrates to produce crystalline calcium hydroxide, and calcium-silicate-hydrates. Higher percentages of C_3S in portland cements led to gain early strength.

b) Dicalcium silicate (C_2S). Is an important constituent of portland cement clinker. Dicalcium silicate contributes to late strength by hydrates and hardens slowly beyond one week due to its lower reactivity.

c) Tricalcium aluminate (C_3A). is considered the most reactive part of portland cement and it is mostly related to the heat of hydration. C_3A is contributed the initial hydration process of the cement when mixed with calcium silicate. It reduces the durability of concrete and releases a large amount of heat during the first week of hardening.

d) Tetracalciumaluminoferrite (C_4AF). Is considered more resistant to reactions with the sulfate salt while reacts with water rapidly and then, the setting occurs within minutesto produce the grey colour. Tetracalciumaluminoferrite acts as a filler contributes and used to assist in the manufacture of portland cement by allowing lower clinkering temperature.

2.1. Cement production: Cement is produced from a mixture of clay, limestone, and sand in a rotating kiln by heating to temperatures reaching 1500ºC. Calcium carbonate is the raw material used for cement production is ($CaCO_3$), which is derived from chalk, crushed limestone, and shells. Cement production processes depending on the handling of raw material before being fed to the rotary kiln which categorized as dry, semi-dry, semi-wet, and wet processes [24]. During these process of production, small amounts of shale, clay, or ash were to be added to provide controlled quantities of aluminum, iron, and silicon [25].The cement produced from the kiln to be cooled, and then finely ground to produce the cement powder [26].

2.2.Crushing: The raw material limestone (5 parts) and clay (1 part) are crushed to a fine powder separately in suitable machines then mixed in proper proportions and finely ground (pulverization). The pulverized mass (dry in the form of a slurry) is introduced into a rotator kiln with the help of a screw conveyer [27,28].

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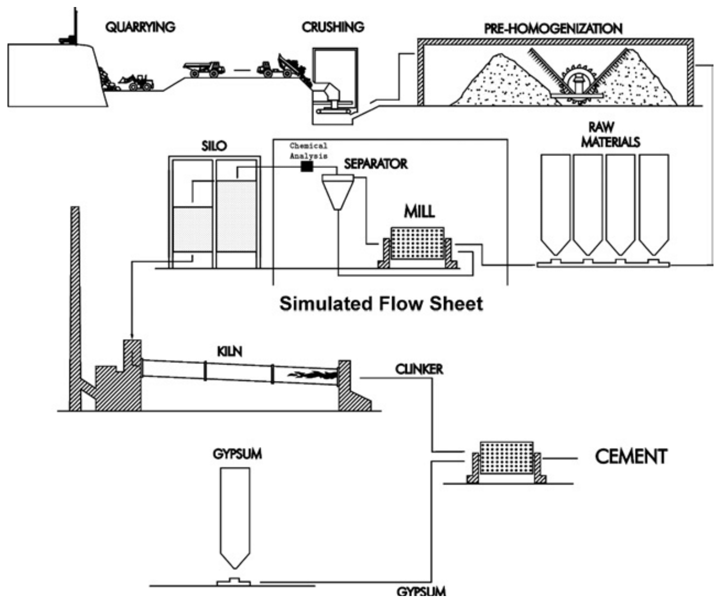

Figure 1. Cement production

The rotator kiln is a cylinder made of steel 150 M long 4 meters in diameter lined inside with bricks. It rotates on its axis at a rate of 30-60 revolutions per minute. Due to the charge slowly moves down the kiln. The charge takes 2-3 hours to travel from one end to the other end.

2.3.Calcinations: A blast of burning coal dust is introduced into the kiln from the lower end. The charge is heated with the help of the burning coal and is exposed to different temperature zones and chemical reactions take place leading to the formation of cement [29].

The temperature in the upper part of the kiln is around 1023 kana, the material loses water here. In the middle part of the kiln where the temperature is 1273 k, limestone ($CaCO_3$) decomposes and gives lime (CaO) (Singh, 2007).

$$CaCO_3 \xrightarrow{1273\ K} CaO + CO_2$$

The temperature is maximum in the lower part of the kiln, it is around 1770-1870 k. The oxides reacts with silica and form calcium silicate and aluminum silicate. The product is called clinker and has a grey color [30].

$$3CaO + SiO_2 \longrightarrow 3CaO.SiO_2$$

$$3CaO + Al_2O_3 \longrightarrow 3CaO.Al_2O_3$$

2.3.Powdering and Packing: The product (clinker) is cooled, and then 2-3% gypsum is added. The resulting powder is packed in bags and sold as cement [31]. Gypsum (Calcium sulfate dihydrate ($CaSO_4$. $2H_2O$) increases the setting time of cement [32]. Cement exhibits characteristics properties of setting and hardening when mixed to a paste with water. This makes them capable of join ingrigidmasses like bricks, stones tiles, etc. into a coherent structure [33]. Cement has the property of setting and hardening underwater by some chemical reactions with it and is called hydraulic cement.

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Table 2. Chemical composition of ordinary portland cement

S.No.	Constituents	Range%
1	Silica (SiO_2)	17-25
2	Calcium (CaO)	60-67
3	Alumina (Al_2O_3)	03-08
4	Iron (Fe_2O_3)	0.5-06
5	Magnesia (MgO)	0.5-4.0
6	Oxides of alkalis (Na_2O & K_2O)	0.3-1.2
7	Sulphuric Anhydride (SO_3)	2.0 -3.5

3. Cements Classification

3.1 Natural Cement

This type of cement is produced by calcining and grinding of the rocks, this naturally occurring to give up to 25% clayey (argillaceous) material of limestone which is also known as Roman Cement [34]. The calcining temperature for sintering and little tricalcium silicate C_3S produced is about (600 to 1220 ^oC). Therefore, strength development is slow [35]. However, the dicalcium silicate C_2S is considerable, the initial set and hardening are rapid in comparison with portland cement [36]. Natural cement is showed properties between that of Portland cement and hydraulic lime without any additional constituents are added to the production process.

3.2 Pozzolana Cement

Pozzolana cement is a materials which form hydraulic cementing materials by mixing with lime without the use of heat. These are made by simply mixing and grinding natural pozzolana (deposits of volcanic ash) and slaked lime used for making the construction walls and domes [37,38].

3.3 Slag Cement

Slag cement is made from blast furnace slag and hydrated lime which is containing up to 65 percent slag of mixture between portland cement and granulated slag. This cement set more slowly than portland cement used to a limited extent for making concrete in bulk construction where strength is relatively unimportant [39].

3.4 Portland Cement

Portland can be defined as an extremely finely ground product obtained by calcining together argillaceous (clay, containing) and calcareous (lime, containing) raw materials at about 1500 ^oC used for construction works [40].

Materials and Methods

Chemicals and Reagents

500 ml beaker, pipette, standard flask, burette, glassrod, measuring jar. Oxalic acid**,** $KMnO_4$ solution, Cement solution, water, EDTA, magnesium indicator, NH_4OH solution, dil. H_2SO_2.N/30 oxalic acid was prepared by dissolving 0.21 g/100 mL ($H_2C_2O_4 . 2H_2O$). N/30

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$KMnO_4$ the solution was prepared by dissolving approximately 0.265 g of $KMnO_4$ in 1 liter of water. Potassium permanganate acts as a self-indicator. However, the reaction end point was observed with the color change from colorless to pale pink

Material

Calcium, Copper, lead and, zinc was used as metals that are soluble in oxalates. Dilute sulphuric acid and standard potassium permanganate were used to determine the calcium in cement by titration with the oxalic acid with standard potassium permanganate solution

Experiential Section

1. Determination of Calcium by $KMnO_4$

In a small beaker 0.2 g of cement and 20 mL of HCl (1.1). The solution was heated over asbestos for 30 min, till cement is decomposed [Ca^{+2}] and converted into calcium chloride [$CaCl_2$], then 2–3 drops of methyl red indicator (color of the solution is red in acidic medium) was added. After that, 15 mL of ammonium oxalate solution and 10% NH_4OH were added slowly to the mixture till the color disappeared and calcium oxalate precipitated. The precipitate was filtered, washed by distilled water and, dissolved in 2 mL H_2SO_4. The total volume is made up to 250 mL with distilled water in a flask. By pipette out, 2 mL of Ca^{+2} solution of cement sample into a conical flask and 10 mL of standard $MgSO_4$ solution then add 50 mL of distilled water and add 3 mL of buffer solution pH-10 (Table 3). The solution was heated to 40 ^oC. The indicator was added and titrate against EDTA till the color changes from red to blue.

Principle

The determination of calcium is carried out by converting it into oxalate, which is estimated by titrating against standard $KMnO_4$ solution, $KMnO_4$ solution is standardized by using a standard oxalic acid solution. In an acidic medium $KMnO_4$ oxalate ion into CO_2 gets reduced to Mn (II) ion. The rate of the reaction is increased as shown on heating (60-70^oC) the oxidation is qualitative.

Standardization of $KMnO_4$

The solution of oxalic acid and $KMnO_4$ was prepared, $KMnO_4$ solution is standardized by titrating against the standard oxalic solution. In a flask, oxalic acid solution 25 mL and 20 mL of dil. H_2SO_2 (about 4N) then the solution was diluted.

Table 3. Amount of calcium present in different brands of cement

S.No.	Samples	Weight g/250 mL
1.	DUNCAN	0.132
2.	RAASI	0.012

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3.	VISHU	0.023
4.	MAHA GOLD	0.028

2. Determination of Calcium by Substitution Method

Principle

Calcium ions are titrated with EDTA a relatively stable calcium complexes is formed

$$Ca^{+2} + H_2Y^{2-} \longrightarrow CaY^{2-} + 2H^+$$

The Ca^{+2} ion are alone no sharp endpoint can be obtained with eriochromeblak–T and the transition from red to blue.

The magnesium indicator complexes are much more stable than the calcium indicator complex but less stable than the EDTA complex. Consequently during titration of the solution in the presence of eriochromebalck–T, the EDTA reacts first with free Ca^{+2} ion then with free Mg^{+2} ions, and finally the complex wine-red color of solution changes from wine-red to blue at the endpoint.

Preparation of standard $MgSO_4$

1.54 g of $MgSO_4$ in a 250 mL standard flask was dissolved in distilled water and makeup solution up to the mark.

Standardization of EDTA

Place instantaneously titration should be done slowly at the endpoint. Calculate the pipette out 20 ml of $MgSO_4$ solution into a 100 mL conical flask and add 5 mL of buffer solution of pH-10 then heat the solution to 40 degrees. Add 10 mL of E.B.T. indicator. After that, The prepared solution titrated with the EDTA till the blue color. However, the reddish color should be disappeared at the endpoint and the blue color appeared.

Table 4. Amount of calcium present in different brands of cement

S.No.	Samples	Weight g/250 mL
1.	DUNCAN	0.122
2.	RAASI	0.014
3.	VISHU	0.022
4.	MAHAGOLD	0.025

Results and Discussion

Calcium was determined by titrating with complexing agents such as ethylenediamine tetraacetic acid (EDTA) using visual and photometric techniques. They showed that all the selected cement

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were contained the calcium oxide (CaO) content within the standard value. Among the four selected cement samples, Duncan cement was found to contain the highest percent content of CaO while Raasi and Vishu cement showed the least percent content of calcium oxide (CaO) in both methods.

The major factor and main component of cement quality is calcium oxide (CaO). As shown in Table 5, the calcium content present in the four selected cement which determined by two methods. It is determined by severe analytical techniques. Complexometric titration with EDTA is one of the analytical techniques to determine calcium oxide and it is used usually to find the total magnesium and calcium content of milk, seawater, and various solid materials. This technique may apply to find out the total hardness of freshwater provided to the solution. EDTA was used in this method which forms a complex with calcium and magnesium ions and the indicator was a blue dye Eriochrome Black T (EBT), which is in charge of changing colour to pink during the titration. The EDTA–metal ion complex showed high stability than the dye-metal ion complex. Therefore, the solution containing the calcium ion reacts with an excess of EDTA to form a complex with the EDTA. The indicator was responsible for the colour changes from red to blue.

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

The interferences are mainly caused by cations of the calcium, aluminum, manganese, and iron, with EDTA to form the complex. Apart from these, the metals also react irreversibly with indicators. The secations also give rise to color change in the indicator, making it difficult to detect the endpoint. However, the calcium oxide determined in a different types of cement and compared between them (Table 5).

Table 5. Amount of calcium present in $KMnO_4$ and substitution method for the same brand

Samples	**$KMnO_4$**	**Substitution**
DUNCAN	0.132g	0.122 g
RAASI	0.02g	0.014 g
VISHU	0.023g	0.022 g
MAHAGOLD	0.028g	0.021 g

The amount of calcium present is different for the same sample that's lead to prove the estimation by $KMnO_4$ is more accurate than another method (Table 3). Therefore, the quality of cement is one of the important factors which is depending on the amount of calcium to produce, the strength, and the durability of structural concrete.

Conclusion

The level of calcium oxide (CaO) in the selected cement samples gave a positive result, especially DUNCAN cement demonstrated the highest percentage of calcium content among others. However, the determination of calcium by $KmnO_4$ method was more accurate than the substitution method.

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