ANALYSIS OF ORDINARY PORTLAND CEMENT (OPC)

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Abstract

A comprehensive analysis of Ordinary Portland Cement has been done. In this article, we report the outcomes of physical and chemical analysis of Ordinary Portland Cement with respect to various parameters such as setting time, compressive strength, fineness or surface area, specific gravity, loss on ignition etc. The results are obtained in good agreement with the standard value.

Keywords: *Ordinary Portland Cement, Clinker, Analysis, Setting Time, Strength*

Introduction

Ordinary portland cement signifies to the hydraulic binding material ground by mixing portland cement clinker, 6% ~ 15% blended materials, and apposite amount of gypsum.(Cement, 2011)

The highest amount of active blended materials mixed in cement should not be over 15% of the total mass. They are allowed to be replaced by kiln ash and inactive blended materials which should be no more than 5% and 10% of the cement mass respectively. The uttermost amount of inactive blended materials mixed in cement should not be greater than 10% of the total mass.

Chemically, ordinary portland cement represents minutely ground mixture of calcium aluminates and silicates of differing compositions, which cause to combine with water when mixed with it to formulate a firm solid framework with fine compressive strength.(Cement, 2011; Duda, 1985; Lea, 2019; Sharma, 2000)

Ordinary portland cement is a combination of the following compounds:

Formula	Abbreviation	Name	Composition (%)
$2CaO.SiO_2$	C_2S	Dicalcium silicate	20-40
$3CaO.SiO_2$	C_3S	Tricalcium silicate	30-50
$3CaO.Al_2O_3$	C_3A	Tricalcium aluminates	8-12
$4CaO.Al_2O_3.Fe_2O_3$	C_4AF	Tetracalciumalumino ferrite	6-10

Herein we have done a brief physical and chemical analysis of Ordinary Portland Cement.

Materials and Methods

Part A. Physical Analysis

Physical analysis includes: Setting time**,** Compressive strength**,** Fineness or surface area and Specific gravity etc(Bogue, 1947; Furman, 1962; Hall, 1952; Randolph Norris Shreve, 1984; Taylor, 1997).

1. Setting time

The setting of cement is administered by the generation of a network of partially hydrated cement particles associated by hydration products. The setting time will adjust how much time the contractor will have to get the concrete placed and finished. Hence, the most foremost properties of cement is setting time. Setting times was measured by Vicat Needle apparatus. These tests essentially measured when the hydrating cement paste grows

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some finite value of resistance to penetration. The determination of setting time is affected to a marked degree by the quality of water used in gauging, the temperature, hygrometric state of the atmosphere, and others factors.

For normal Portland cement the initial setting time shall not be less than 30 minutes and nor the final setting is more than 10 hours. The setting time of Portland cement usually decreases as the temperature is raised, but the rate of this change varies with different cements.

Preparation of Test Block

A cement paste was prepared with 400 gm cement sample and 100 ml of water to allow the paste of a paradigm consistency. When water was added to the cement sample, a stop watch was started. The Vicat mould was filled throughly with the cement paste prepared. The mould reluxing on a non-porous plate and smooth off the exterior of the paste making it level with the apex of the mould. The cement block thus made ready for use in the mould is the test block.

2. Compressive Strength

Strength tests take three different forms, the specimen being subjected to tension, compression or bending. When the specimen is subjected to compression while taking strength test, it is known as compressive strength.

The strength evolved by a cement is contingent on many factors, e. g., the grading of the sand or aggregate, the percentage of water used, the degree of mixing, and the temperature and humidity of the atmosphere in which it is regulated, the procedure by which the substance is placed in the moulds and the specimen made, the curing environments, the method of testing and the age at which experiments are accomplished.

Preparation of Test Specimen

About 600g sand and 200g Portland cement were weighed. Then 200 ml of water was added and mixed properly. Mixing should be uniform.

The mixture was tamped by tamping rod into a mould. Tamping was done 24 times in each portion of mould while it is half filled. Again, tamping was done 24 times in each portion of mould while it is filled completely. The resulting cement, sand and water mixture is called mortar. The mortar as demonstrated in the mould is the test specimen.

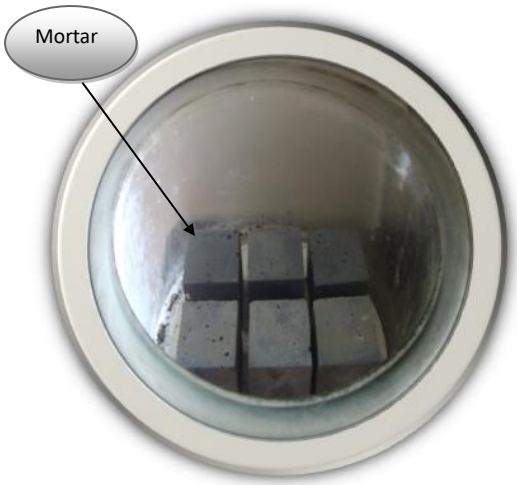

Fig. 1: Preparation of mortar

3. Fineness or Surface Area

Surface area represents the fineness of cement. The more finely a cement, greater is the strength and particularly the early strength it yields. Fineness is measured by Blain air permeability apparatus. More is the fineness, less the airflow. So the cement reacts readily.

4. Specific Gravity

Archimedes figured out that the weight of body in air minus its weight in water is equivalent to the weight of the water displaced by the body.

Specific gravity is specified as "The weight of a body estimated with the weight of an equal amount of pure water at 4°C. When a body is positioned in water, the volume of water replaced is equal to the volume of the body. When the body is accommodated in water that it undergoes a noticeable loss of weight. This loss of weight is equal to the weight of the water displaced. There are many ways to express the equation for the specific gravity (SG). Here is the most familiar one:

$$SG = \frac{\text{(Weight in Air)} \times \text{(SG of Fluid)}}{\text{Loss of Weight in Fluid}}$$

Part B: Chemical Analysis

The cement sample was an inorganic compound. It contains mainly silica, aluminum oxide, iron oxide, and little amount of calcium oxide, magnesium oxide and sulfur oxide.(Ali et al., 2008; Bogue, 1947; Duda, 1985; Furman, 1962; Hall, 1952; Randolph Norris Shreve, 1984)

1. Loss on Ignition.

Procedure: About 1.0 g of precisely ground cement sample was taken in a formerly weighed little porcelain crucible and heat it in a high temperature burner (high temperature was attained by flowing air and gas simultaneously through two separate pipe) at 800-900˚C for 20-30 minutes. Then it was cooled and measured in desiccators.

The experiment was performed again for five minute intervals till persistent weight was achieved. The difference in the weight gave the loss on ignition. Loss on ignition was then calculated by using the relation,

Loss on ignition = [(X × 100)/Sample weight] %

Where,

X = weight of the sample before ignition – weight of the sample after ignition.

2. Silica (SiO_2)

Procedure: About 0.5 g cement sample and 0.5 g solid ammonium chloride, NH_4Cl were taken in a porcelain dish. The two content were mixed properly with glass rod. The dish was covered with lid and 5.0 ml of concentrated HCl was added with glass rod (slowly) to the sample. The porcelain dish was heated in a water bath until the content in the dish was become fully dried. Then it was kept in an oven at 110˚C for one hour for further dry up. Then the dish was cooled and 5ml of concentrated HCl was added to moisten the sample. 20ml of hot water was added and afterwards the content in the dish was digested in a water bath for 30 minutes. The digested content was filtered through an 11 cm Whatmann No. 41 filter paper. Hot distilled water was used to wash the precipitate till the washings were lacking chloride. Then the filtrate was taken to a volumetric flask of 250 ml and filled up to the mark. This stock solution was used for determining sesquioxide (Fe_2O_3+Al_2O_3). The filter paper and the precipitate

was situated in a measured porcelain crucible, enveloped with the stopper, warmed mildly to char the paper unaccompanied by inflaming and ignited at 800-900˚C in a high temperature burner for 45 minutes.

The porcelain crucible was cooled in desiccators and weighed. The procedure was performed again till steady weight was secured. The difference in the weight gave the silica content of the cement sample. The percentage of the silica was then determined.

3. Sesquioxide (R_2O_3)

Procedure: 100 ml of stock solution was grabbed in a 600 ml beaker and 5 ml of conc. HNO_3 was added. The solution was boiled on the hot plate for 1-2 minutes. Then 2-3 ml of methyl red indicator was mixed and the hydrated 'sesquioxides' was precipitated by adding 1:1 ammonia solution to the boiled stock solution, prior to the color changes from orange to yellow. The solution was permitted to settle for a few minutes and filtered by an 11 cm Whatmann No. 41 filter paper.

The precipitate was cleaned with very warm 2% ammonium nitrate solution. The filter paper was situated in an already measured porcelain crucible and ignited at 800-900˚C for 45 minutes. Then it was cooled in desiccators and weighed. The difference in the weight gave the sesquioxide content of the cement sample. The percentage of the sesquioxide was then determined.

4. Lime (CaO)

Procedure: The filtrate obtained from the sesquioxide determination was boiled on the hot plate. Then concentrated HCl was added to acidify the solution until the colorless solution turned into orange red color. 10 ml of 5% ammonium oxalate solution was mixed to the hot solution. The solution was neutralized with 1:1 NH_3 solution and cloudy white solution was then appeared. The solution was permitted to stand 3 hours and then filtered through Whatmann No. 42 filter paper. The precipitate was rinsed with hot 0.1% (1 g per liter) ammonium oxalate solution. Then the filter paper was kept in a previously measured porcelain crucible and charred and then ignited in a high temperature burner at a Temperature of 800-900˚C for 45 minutes. Then it was cooled in desiccators and weighed. The difference in the weight gave the CaO content of the cement sample. Then the percentage of lime was determined.

Chemical Reaction:

$$Ca^{++} + C_2O_4^{--} + H_2O \longrightarrow CaC_2O_4.H_2O$$

$$CaC_2O_4.H_2O \xrightarrow{100\text{-}105^{\circ}C} CaC_2O_4 + H_2O$$

$$CaC_2O_4 \xrightarrow{475\text{-}525^{\circ}C} CaCO_3 + CO$$

$$CaCO_3 \xrightarrow{1200^{\circ}C} CaO + CO_2$$

5. Magnesium Oxide (MgO)

Procedure: The filtrate obtained from lime determination was used for the determination of MgO. Concentrated HCl was added to the hot filtrate to make it acidic, while orange red color appeared. If not, little drops of methyl red indicator were mixed. Then 10 ml of saturated solution of di-ammonium hydrogen phosphate and 25 ml of conc. NH_3 solution was appended with relentless stirring. Subsequently, the solution was chilled to room temperature and twitched for 20-30 minutes until a cloudy solution appears. If not, a few drops of NH_3 solution were added. The solution was then kept aside for 2-3 hours and filtered through Whatmann No.44 filter paper. The precipitate was splashed with 2.5% ammonia solution. The filter paper was allocated in a formerly measured porcelain crucible, charred and ignited in a high temperature burner at a temperature of 800-900˚C. The crucible

was taken out, cooled in desiccators and measured. The difference of the weight gave the MgO content of the cement. The percentage of the MgO was then determined.

Chemical Reaction:

$$Mg^{++} + HPO_4^{--} + NH_4^{+} + OH^{-} \longrightarrow MgNH_4PO_4 + H_2O$$

$$2MgNH_4PO_4 \longrightarrow Mg_2P_2O_7 + 2NH_3 + H_2O$$

6. Iron oxide (Fe_2O_3)

Procedure: About 10 ml HCl was added with 10 ml solution from the mixed oxide & heat it to boil covering with watch glass. Then $SnCl_2$ solution was added by drop wise until the solution was decolorized & then it was cooled. After cooling at room temperature 10 ml of $HgCl_2$ was added & placed in dark covering with watch glass. A slight milky precipitate was appeared at this stage. Then 20 ml acid & 100 ml distil water was added with stirring. Then titrate it with 0.1N $KMnO_4$. Faint pink color is observed (it should persist for 30 second).

Chemical Reaction:

$$Fe^{+++} + 3NH_3 + 3H_2O \longrightarrow Fe(OH)_3 + NH_4^{+}$$

7. Aluminum Oxide (Al_2O_3)

Calculation:

$$\% \text{ of } Al_2O_3 = [\% \text{ of } R_2O_3 - \% \text{ of } Fe_2O_3]$$

$$= [9.50\% - 2.98\%] = 6.52\%$$

Result: % of Al_2O_3= 6.52%

8. Insoluble Residue (IR)

Procedure: About 0.5 g cement sample was taken in a previously weighed small porcelain crucible. Then 1:1 NH_3 solution was added and a clear yellow solution was then appeared. 25 ml of H_2O was mixed to the solution and kept onto hot plate. The hot solution was filtered through Whatmann No.41 filter paper. The precipitate was cleaned with warm water for 6 times. The filtrate and washed material were kept for the treatment of SO_3. The ppt was then dissolved with 1%NaOH solution in a beaker and kept on hot plate. Concentrated HCl was added to the hot filtrate to make it acidic, while orange red color appeared. If not, little drops of methyl red indicator were added and filtered through Whatmann No.41 filter paper. The precipitate was cleaned using 2% ammonium chloride solution for 12-15 times. The filter paper was kept in a formerly measured porcelain crucible, charred and ignited in a high temperature burner at a temperature of 800-900˚C. The crucible was taken out, cooled in desiccators and weighed. The difference of the weight gave the IR content of the cement. The percentage of the IR was then determined.

9. Sulphuric Anhydride (SO_3)

Procedure: About 0.5 g cement sample was taken in a previously weighed small porcelain crucible. Then 1:1 NH_3 solution was added and a clear yellow solution was then appeared. 25 ml of H_2O was included to the solution and kept onto hot plate. The hot solution was filtered across Whatmann No.41 filter paper. The precipitate was rinsed with hot water for 6 times. Then the filtrate was taken to a volumetric flask of 250 ml and filled up to the level. This stock solution was warmed and about 1.0gm $BaCl_2$ (100 gm per liter) was added. The solution was then placed onto the hot plate for 3 hours and filtered through Whatmann No.41 filter paper. The precipitate was rinsed with warm water. The filter paper was placed in a formerly measured porcelain crucible, charred and ignited in a high temperature burner at a temperature of 800-900˚C. The crucible was taken out, cooled in desiccators and weighed. The difference of the weight gave the SO_3 content of the cement. The percentage of the SO_3 was then determined.

Chemical Reaction:

$$Ba_{++} + SO_4^{--} \longrightarrow BaSO_4$$

$$BaSO_4 \longrightarrow BaO + SO_3$$

10. Na/K Determination

In cement sample, Na and K are determined by **colorimetric analysis.** Colorimetry is apposited with the resolution of the concentration of a substance by computation of the relative absorption of light with respect to a known concentration of the substance. The technique is based on Beer-Lambert law:

$$A = €Cl$$

Where,

A = Absorbance

€ = Molar Absorption Coefficient

C = Concentration of Solution

l = Path Length

A plot of absorbance against concentration will give a straight line passing through the point C=0, A=0. This calibration line is used to establish the unspecified concentrations of Na and K in cement after the measurement of absorbance.

Procedure for Na: Different concentrations (e.g.2ppm, 4ppm, 6ppm, 8ppmetc.) of solutions were prepared from 100 ppm supplied solution. The Flame Photometer was zero balanced by using distilled water. Then absorbance was determined for each solution by Flame Photometer. The Photometer was zero balanced before taking each reading.

Procedure for K: The procedure of determining unknown concentration of K was exactly same as for Na.

Results and Discussion

Part A. Physical Analysis

*1. **Determination of Setting Time***

Two periods are distinguished:

a) Initial Setting Time: Initial setting time is interpreted as the interlude between the gauging and partial loss of plasticity.

Procedure: The test block was situated below the rod bearing the needle. Lowered the needle with a mild manner to assure contact with the surface of the cement paste and released rapidly to pierce the block. The procedure was performed again and again till the needle failed to penetrate the test block to a point of 5(±) mm, measured from the bottom of the plate. The time passing between the times of water was added and the time the needle failed to pierce to the bottom of mould was the initial setting time.

b) Final Setting Time: It is the time needed for the gauged cement to gain enough fineness to counter a certain obvious pressure.

Procedure: The above needle was replaced by the one with an annular attachment. The cement should be believed eventually set when upon applying the needle lightly to the surface of the block, the needle make an intuition therein, while the attachment fails to do so. The time proceeding between the times when the attachment fails to make to make an impression, was the final setting time.

Calculation:

Time when water was added to cement is 9:25 am

Initial setting time recorded at 10:10 am

Therefore, the initial setting time (10:10 – 9:25) am = 45 mins

Final setting time recorded at 10:40 am

Therefore, the final setting time (10:40 – 9:25) am = 1 hr 15 mins

Table 1: Setting time of OPC

Batch	Initial Setting Time (mins)	Final Setting Time (mins)
Cement: Water (4 : 1)	45 min	1 hr 15 min

2. Determination of Compressive Strength

Cubes (mortar) with mould were placed in 98% humid condition for 24 hours. After that the test specimens were detached from the mould and dipped in water until the day of testing. Compressive strength was determined after 3 days and 7 days by an apparatus called Hydraulic press.

During testing the test specimens were taken out of water and the surface water was wiped out by tissue and placed on the Press machine. Load was applied to break the test specimen and the results were noted from the dial gauge.

Table 2: Batch composition of OPC

Batch(cement: sand)	Cement (gm)	Sand (gm)	Water (ml)
1:3	200	600	200

Table 3: Compressive strength of OPC

Batch (cement: sand)	Strength (lbs)	Average strength (lbs)	Average strength (Psi = lbs/4)	Times (days)
1:3	11700 11500 11600	11600	2900	3
1:3	15000 13000 14000	14000	3500	7

3. Determination of Surface Area

Procedure: The permeability cell was connected to the manometer tube and checked that airtight link was attained. A small stopcock grease was registered to the standard taper connection. The effectiveness of the connection can be fixed by fasten the cell to the manometer, then suspending the valve. Any continual drop in pressure specifies a leak in the system. The perforated disc was kept on the ledge in the permeability cell, marked face down. A filter paper disc was placed on the metal disc and pressed the edges down with a rod having a diameter moderately smaller than that of the cell. About 2.8 gm of cement was measured and placed in the cell. The side of the cell was

tapped sparingly so as to level the bed of the cement. A filter paper disc was placed on the surface the cement and compressed the cement with the plunger until the plunger collar was get in touch with the top of the cell. The plunger was withdrawn slowly a little distance, rotated about 90°, repressed & then steadily withdrawn. The permeability cell was connected to the manometer tube and checked that airtight attachment was achieved. Care was taken not to disorder the developed bed of cement. The air was evacuated slowly in the one arm of the manometer U- tube until the liquid reached the highest mark and then closed the valve tightly. The timer was started when the bottom of the meniscus of the manometer liquid reached the second (next to the highest) mark, stopped when the bottom of the meniscus of the liquid reached the third (next to the bottom) mark. The time interval was measured and recorded in seconds. The temperature of the experiment was taken and recorded in °C.

The specific surface value (S) was calculated in m^2/kg as follows:

$$S = \frac{S_s\sqrt{T}}{\sqrt{T_s}}$$

Where,

S_s = specific area of the standard sample used in calibration of the apparatus m^2/kg

T = measured time interval of manometer drop for test sample

T_s = measured time interval of manometer drop for the standard sample used in calibration of the apparatus.

4. Determination of Specific Gravity

Procedure: The weight of the cement sample was taken in air. The weight of the gravity bottle was also taken. Then the gravity bottle was filled with the fluid (one which does not react with sample e.g kerosine) and weighed. The upper content of the gravity bottle was filled with the sample, which was then put on the bottle and weighed again. The difference in the weight is the lost weight of the sample in kerosene. The SG was then calculated.

Calculation:

Here,

Weight of empty gravity bottle = 16.1463g

Weight of cement = 0.4463g

Weight of gravity bottle + cement + kerosene = 36.5380g

Weight of gravity bottle + kerosene = 36.2148g

Lost weight of cement in kerosene = 36.5380g - 36.2148g

= 0.3232g

$$SG = \frac{\text{(Weight of Cement in Air x SG of Kerosene)}}{\text{Loss of Weight in Kerosene}}$$

$$= \frac{0.4462 \times 0.84}{0.3232}$$

= 1.159

Calculation:

Here,

Specific surface area of the standard sample, S_s= 377.4 m^2/kg

Measured time interval of manometer drop for test sample, T= 21 secs

Measured time interval of manometer drop for the standard sample used in calibration of the apparatus, T_s= 62 secs

Specific surface area of the test sample

$$S = \frac{S_s\sqrt{T}}{\sqrt{T_s}}$$

$$= \frac{377.4\times\sqrt{21}}{\sqrt{62}} \text{ m}^2/\text{kg}$$

$$= 219.29 \text{ m}^2/\text{kg}$$

Part B: Chemical Analysis

1. Loss on Ignition

Calculation:

Weight of blank crucible = 18.7886g

Weight of sample taken = 1.0025g

Weight of blank crucible + sample taken (before ignition) = 19.7911g

Weight of blank crucible + sample taken (after ignition) = 19.7836g

Weight lost = (19.7911-19.7836) g = 0.0075g

Loss on ignition = [(0.0075 × 100)/1.0025] % = 0.75%

Result: Loss on ignition = 0.75%

2. Silica (SiO_2)

Calculation:

Weight of blank crucible = 18.9690g

Weight of blank crucible + Weight of SiO_2 = 19.0737g

Weight of SiO_2 = (19.0737-18.9690) g = 0.1047g

% of SiO_2 = [(Weight of SiO_2 / sample weight) × 100] %

= [(0.1047/0.5004) × 100] % = 20.92%

Result: % of SiO_2 = 20.92%

3. Sesquioxide (R_2O_3)

Calculation:

Weight of blank crucible = 18.9690g

Weight of blank crucible + Weight of R_2O_3 = 18.9785g

Weight of R_2O_3 = (18.9785-18.9690) g = 0.0095g

% of R_2O_3= [Weight of R_2O_3 / (sample weight×50)] × 250× 100 %

= [0.0095g / (0.5004×50)] × 250× 100 % =9.50%

Result: % of R_2O_3= 9.50%

4. Lime (CaO)

Calculation:

Weight of blank crucible = 18.9690g

Weight of blank crucible + Weight of CaO = 19.0331g

Weight of CaO = (19.0331-18.9690) g = 0.0641g

% of CaO = [Weight of CaO/ (sample weight×50)] ×250× 100 %

= [0.0641 / (0.5004×50)] ×250× 100 % = 64.05%

Result: % of CaO = 64.05%

5. Magnesium Oxide (MgO)

Calculation:

Weight of blank crucible = 18.9690g

Weight of blank crucible + Weight of MgO = 18.9739g

Weight of MgO = (18.9739-18.9690) g = 0.0049g

% of MgO = [Weight of MgO/ (sample weight×50)] × 250×0.362×100 %

= [0.0049/ (0.5004×50)] × 250×0.362×100 % = 1.76%

Result: % of MgO = 1.76%

6. Iron Oxide (Fe_2O_3)

Calculation:

Weight of blank crucible = 18.9690g

Weight of blank crucible + Weight of Fe_2O_3 = 18.9720g

Weight of Fe_2O_3 = (18.9720-18.9690) g = 0.0030g

% of Fe_2O_3= [Weight of Fe_2O_3 / (sample weight×50)] × 250× 100 %

= [0.0030 / (0.5004×50)] ×250× 100 % =2.98%

Result: % of Fe_2O_3= 2.98%

7. Aluminum Oxide (Al_2O_3)

Calculation:

% of Al_2O_3= [% of R_2O_3 - % of Fe_2O_3]

= [9.50% - 2.98%] = 6.52%

Result: % of Al_2O_3= 6.52%

8. Insoluble Residue (IR)

Calculation:

Weight of blank crucible = 18.9690g

Weight of blank crucible + Weight of IR = 18.9732g

Weight of IR= (18.9732-18.9690) g = 0.0042g

% of IR = [(Weight of IR / sample weight) × 100] %

= [(0.0042/0.5003) × 100] % = 0.84%

Result: % of IR = 0.84%

9. Sulphuric Anhydride (SO_3)

Calculation:

Weight of blank crucible = 18.9690g

Weight of blank crucible + Weight of SO_3 = 19.0055g

Weight of SO_3 = (19.0055g-18.9690) g = 0.0365 g

% of SO_3 = [(Weight of SO_3 / sample weight) ×0.342× 100] %

= [(0.0365 /0.5004) ×0.342× 100] % = 2.5%

Result: % of SO_3 = 2.5%

10. Na/K Determination

Table 4: Data for the determination of conc. of Na

Concentration (ppm)	Absorbance	Absorbance of the solution of unknown conc.
2	2	7
4	4	
6	6	
8	8	

Then a plot of absorbance vs. concentration was drawn. Again absorbance was determined for a solution of unknown concentration. The unknown concentration was determined from graph as shown below.

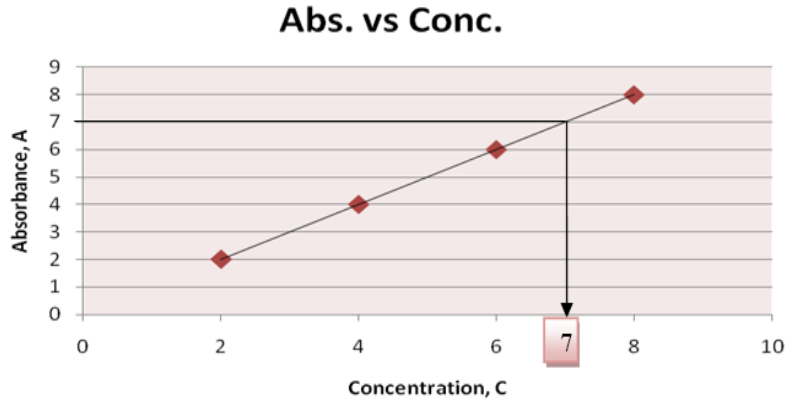

Fig. 2: Plot of absorbance vs. concentration for the determination of conc. of Na

Table 5: Data for the determination of conc. of K

Concentration (ppm)	Absorbance	Absorbance of the solution of unknown conc.
2	4	16
4	8	
6	12	
8	16	

Then a plot of absorbance vs. concentration was drawn as below and the unknown concentration was determined from the following graph.

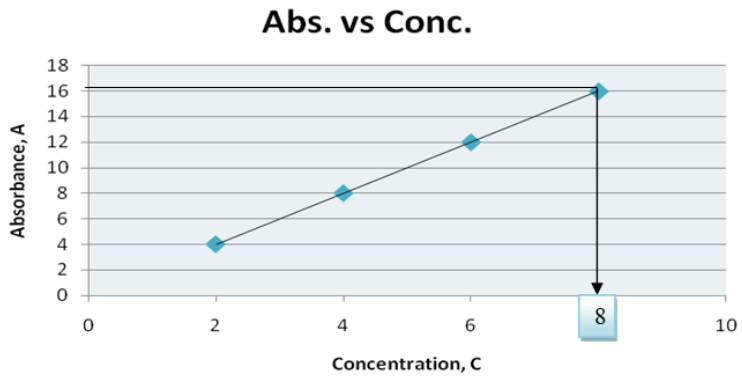

Fig. 3: Plot of absorbance vs. concentration for the determination of conc. of K

Result:

From graph,

1. The unknown concentration of Na is 7.0 ppm.
2. The unknown concentration of K is 8.0 ppm.

Conclusion

We carried out a brief literature study about the Ordinary Portland Cement analysis. Both physical and chemical parameters were analyzed. Most of our test results complies with ASTM standard.

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