Experiment 2: Estimation of CaO in cement solution by rapid EDTA method

Significance of the experiment: Cement is part of concrete and the strength of bonding of cement is contributed by CaO. Cement contains oxides of calcium, Aluminium, Magnesium, iron, and silica.[1] Calcium oxide in a prime constituent of cement. Excess lime reduces the strength of cement and presence of lime in amount lesser than needed also reduces the strength of cement and results in quick setting. Understanding of the role of different compounds of cement during hydration is important for engineers to identify the behaviour of cement concrete.[2,3] Calcination of cement raw materials in the kiln produces a material called clinker. Free lime (CaO) in clinkers has to be closely monitored to ensure the quality of cement. Excess of free lime results inundesirable effects such as volume expansion, increased setting time or reduced strength. In addition, constant monitoring of free lime allows the operator to determine and maintain the optimum operating point of the kiln inorder to obtain maximum reactivity and to reduce thermal consumption. With increased reactivity, grinding of theraw meal can also be reduced leading to further economies of energy. Now a days, from dams to apartments to buildings are constructed using cement and the strength of the structure is required for human well being.

Aim: To determine percentage of CaO in the given sample of cement solution using standard Na_2EDTA solution.

Principle: Cement contains silicates of iron, aluminum, and calcium. Calcium oxide is a prime constituent of cement. The general composition of a Portland cement is given below.

Constituent	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	Na_2O & K_2O	SO_3
%	60-66	17-25	3-8	2-6	0.1-5.5	0.5-1.5	1-3

In the estimation of calcium in cement, the given cement sample is treated with concentrated hydrochloric acid. The insoluble silica is filtered off and the filtrate, which contains calcium ions, is titrated at a pH of 12-14 against Na_2EDTA using Patton and Reeder's indicator. This is used because Eriochrome Black T forms very weak complex with calcium ions. Diethylamine is added to maintain pH of about 12.5 and glycerol is added to get a sharp end point. This method facilitates the determination of calcium only although Mg^{2+} ions are present in the cement solution, which can readily form complexes with Na_2EDTA (*i.e.* Mg^{2+} ions do not react with Na_2EDTA in the above conditions).

Procedure:

Part A: Preparation of standard solution of disodium salt of Na_2EDTA.

Weigh Na_2EDTA accurately and transfer to a 250 mL volumetric flask through a funnel. Add around 5 mL of ammonia (NH_3) solution. Dissolve the crystals in ion exchange water, dilute up to the mark and mix well.Calculate the molarity of Na_2EDTA solution.

Part B: Estimation of CaO

Pipette out 25 mL of the given cement solution into a clean conical flask. Add 3 mL of 1:1 glycerol and 3 mL of diethylamine with constant mixing. Add 4 mL of 4N sodium hydroxide. Add a pinch of Patton and Reeder's indicator and mix the solution well. Titrate against Na_2EDTA solution taken in the burette till the color changes sharply from wine red to clear blue. Perform the titration slowly towards the end point and repeat the experiment for agreeing values.

Result: CaO in the given sample of cement solution = --------------- %.

Links to the external sources of information about the topic:

1. http://www.indiastudychannel.com/projects/3905-Estimation-of-CaO-in-cement-solution.aspx
2. https://www.thermoscientific.com/content/dam/tfs/ATG/CAD/CAD%20Documents/Application%20%26%20Technical%20Notes/Cement,%20Coal,%20Minerals%20Sampling%20and%20Online%20Analysis/Cement%20Online%20Elemental%20Analyzers/Free-Lime-Determination-Clinker.pdf
3. http://scifun.chem.wisc.edu/chemweek/PDF/LIME_CalciumOxide.pdf
4. http://www.uobabylon.edu.iq/eprints/publication_11_17959_1586.pdf

Experiment 2: Observation and Calculations

Part A: Preparation of standard solution of disodium salt of EDTA.

1. Weight of bottle + Na_2EDTA salt = W_2 = --------------- g

2. Weight of empty bottle = W_1 = --------------- g

3. Weight of Na_2EDTA salt = W_2-W_1 = ------------------ g

$$Molarity\ of Na2EDTA\ = \frac{Wt\ of\ the\ salt\ (W2 - W1)\ \text{X}\ 4}{Molarmass\ of\ Na2EDTA\ (372.24g)}$$ ---- (`Z')

Part B: Estimation of CaO in cement

Burette Reading	**Trial** I	**Trial** II	**Trial** III
Final Reading			
Initial Reading			
Volume of Na_2EDTA run down (mL)			

Volume of Na_2EDTA consumed = ----------- mL (a)

Weight of cement in 250 mL = -------------- g (*w*). (`w' will be provided to you).

1000 mL of 1M Na_2EDTA = 56.08 g of CaO (molar mass of CaO = 56.08 g)

`a' mL of `Z' M Na_2EDTA $= \frac{56.08\ X\ Z\ X\ a}{1000\ X\ 1}$ of CaO = ------------- g (b)

Amount of CaO in 25 mL of cement solution = ------------ g (b)

Amount of CaO in 250 mL of cement solution = 10 x b = ------------ g (c)

Percentage of CaO in given cement solution = $\frac{c\ X\ 100}{Weight\ of\ cement\ (w)}$

Result : CaO in the given sample of cement solution = ---------------- %.