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Determination of Calciumin Talcum Powder by Complexometric Titration

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ABSTRACT

Talc is a rich in a magnesium silicate mineral of many base metal sulfide ore deposits that occurs as a gangue component around the world which is used in the pharmaceutical industry as a raw material of mineral origin. Talc quality for pharmaceutical raw material pharmaceutical uses is regulated by pharmacopoeias, which include several chemical and physicochemical tests. The aim of this study was to discuss the talcum powder: uses, safety, application, and estimate the presence of calcium quantity in four selected samples by complexometric titration in the presence of EDTA and EBT as indicator. The results of the study showed among the four selected sample, Ashoka has highest quantity of calcium among the others.

Keywords
Calcium ion, Talcum powder, properties, Complexometric titration

Introduction

Talc structural formula is $(Mg_6)[Si_8]O_{20}(OH)_4$ which belonging to the trioctahedral mineral [1]. The talc has the uniqueness properties such as high melting point, small hardness and generally has a slippery hand in the form of block, fiber, blade, or radial. Talc powder has good filling for electricity and heat, contraction force, low expansion, high dispensability, strong hiding power, good oil absorption and hydrophobicity, large slipperiness, acid and alkali corrosion resistance [2]. Talc is widely used in paint, paper, chemical, cable, rubber, ceramics, due to its stable chemical properties.

By metamorphic hydrated magnesium silicate talc powder provide superb opacity to the end products with a soapy feel and pearly luster [3]. Talc enhances the performance properties of the compound and has a greasy feel which led to produced creep resistance at both ambient and elevated temperature and the higher stiffness,due to the following properties of talc: Excellent blending characteristics with oleo resinous materials, platy in nature, electrical resistance, excellent thermal, chemically inert and smooth greasy feel led to a huge demand as a filler [4].

Talc molecule is neither explosive nor flammable and chemically has very little chemical reactivity led to lose its hydroxyl groups around 900°C. Therefore, it is considered a weak acid and practically insoluble in water. Talc's re-crystallizes into different forms of anhydrous magnesium silicate above 1050°C which has a melting point is at 1500°C. [5](**Figure 1**).

Talc is the softest mineral on earth which used in industrial products as talcum powder to protect, dry and perfume the skin for more than a century [6,7]. Talcum powder has the ability to provide lubrication and absorb moisture at the same time. Many studies nearly 40 years report, the powdery mineral used in cosmetic products is highly refined and safe [8]. There are some factors considered the main function of the talc powder due to its properties filling

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the master batch in the plastic product such as increase the hardness of the product, increase the proportion, reduce the cost, and reduce the product shrinkage rate [9].

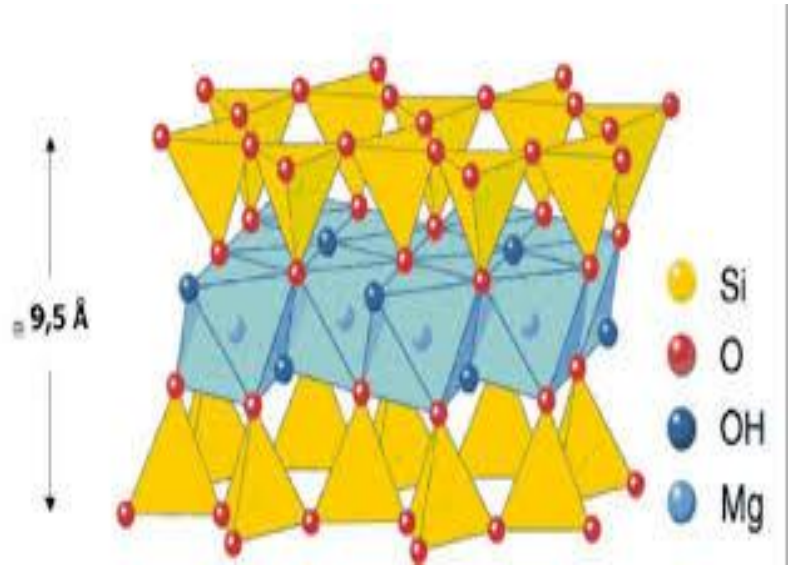

Figure 1: Crystal Structure of Talc [10]

In the field of pharmaceutical industry, talc is used as a sliding agent [11]. However, Talcum powdered is used as an adsorbent and an antiphlogistic agent [12]. It is used to improve the friability of the tablet mass which led to shows an antistatic and antiadhesive action [13].

Calcium is vital component and has significant role in the development of bones and teeth. However, our body has more than 95% of calcium which found in bones and teeth [14]. Many elderly people affected by osteoporosis due to the decrease calcium, therefore, to avoid this problem, calcium tablets such as calcium salts binders, coloring agents, flavoring agents, etc may be taken to supplement the daily intake from diet [15].

In the present study, the calcium amount in four selected talcum powder was determined by titration with a standard solution of ethylenediaminetetraacetic acid (EDTA). EDTA is formed very strong 1:1 complex with most metal ions and commonly used as a chelating agent. The structure of EDTA is shown below (**Figure 2**).

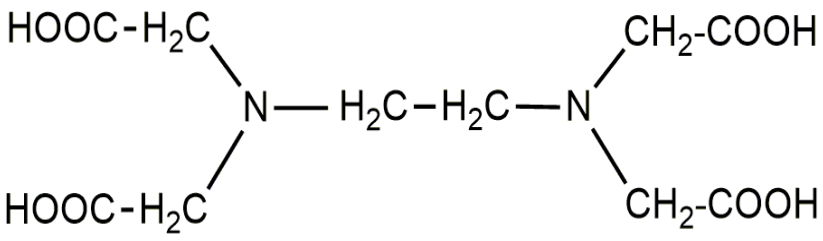

Figure 2: EDTA Structure

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Literature Review

Talc is basically belongs to the class of silicates and aluminosilicates [16]. The crystal structure of talc has a magnesium tetrasilicate and was determined by the X-ray crystallography. The fundamental structural elements of talc are silicon oxide [SiO] [17,18]. However, three corners layers of planar lattices with the adjacent tetrahedrons formed by the silicon oxide groups which led to three atoms of oxygen belong at the same time to two silicon atoms, while the fourth oxygen atom belongs to one tetrahedron only. **Figure 3** showed the balanced between the charge of the anion layers and the magnesium cations by the appropriate numbers of magnesium arranged regularly between them. In nature, talc minerals occurred rarely pure substances. Talc contains a mixture of 4.75% H,O, 31.90% MgO, and 63.35% SiO, the minerals differing in chemical composition containing distinct contaminations of the crystal lattice depending on the geochemical conditions and mixture [19].

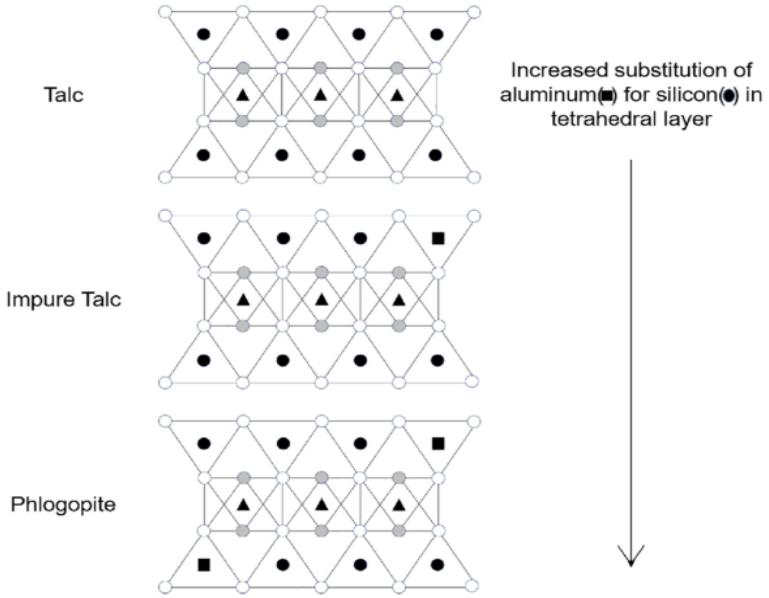

Figure 3: Talc constitution

Some physical properties reflected the crystal structure of talc through weak interactions of the vander Waals bonds. The talc layer gives an excellent cleavage while, the softness of talc is due to the ease of displacement of these layers which led to bad electrical and thermal conductor [20].

Thermal Decomposition

Talc is typically with small endothermic DTA effect without any change in the optical properties or crystal structure driven off between 653 and 773 K[21]. Water is liberated from talc during the heating of the mineral through differences in bonding energy. The talc products decomposed above 873 in strong acid and strong base solutions led to form free magnesium oxide [22].

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Chemistry of Talcum Powder

The chemical composition of talc expressed as oxides of magnesium oxide 63.5%, silicon dioxide 31.7%, and 4.8% water representing the formula $Mg_3Si_4O_{10}(OH)_2$ [23]. Talc considered containing a small portion of aluminum silicate as a powdered native hydrous magnesium silicate. However, talc structural consists of three sheets, two layers of tetrahedrally-linked silica layers and the third layer coordinated magnesium hydroxide groups (brucite layer)[24,25]. Talc and asbestos are form under different geological conditions which are separated into adjacent but not associated [26]. Moreover, the absence of asbestos in talc samples is confirmed by analytical techniques [27].

Composition of Talcum Powder:

a. Talcum

Talc also known as soap stone powder steatite is our main cosmetic products such as adult body, baby powder, and facial powdersas well as in other consumer products. Generally, talc that has asbestos is being able to cause cancer if inhaled [28].

b. Calcite

Calcite chemical formula of $CaCO_3$consist of rock-forming mineral which is decomposed above 700°C, resulting in quicklime (CaO) and carbon dioxide (CO_2). Calcite is one of the most widely used minerals. The calcite crystals produced by the single-cell led to spherulite disk, as shown below (**Figure 4**) [29].

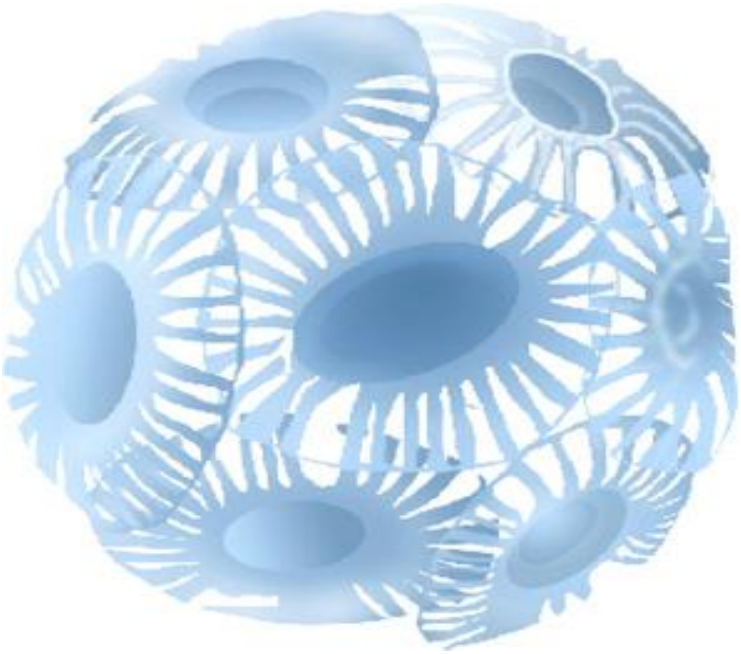

Figure 4: Hollow Structure [29]

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c. China Clay Powder

China clay plays a lead role in the cosmetic clays which you can easily get from any china clay powder exporter that formed by the granite rocks decomposition [30]. China clay is characterized by its fine particle size, lamellar particle or shape plate and chemical inertness. Gentle clay after mixed with water used to remove oil from the skin keeping the nose from looking shiny by facial masks and also soap makers in shaving and oily skin soaps. China clay also used in the formulation of natural deodorants, poultices and scrubs and plays a lead role for using it in the ceramic industry [31].

d. Dolomite Powder

Dolomite is a rich in magnesium and calcium carbonate which is belong to limestone [32]. Dolomite contain two types of materials, a dolomite limestone which is a mixture of calcium and magnesium carbonates and, a true chemically uniform of calcium magnesium carbonate with the chemical formula $CaMg(CO_3)_2$[33]. The mineral dolomite normally consists of a three layer, a magnesium layer, then a carbonate layer, then calcium layer (Figure 5). Limestone or calcites are less hard than dolomite which forms of calcium carbonate [34].

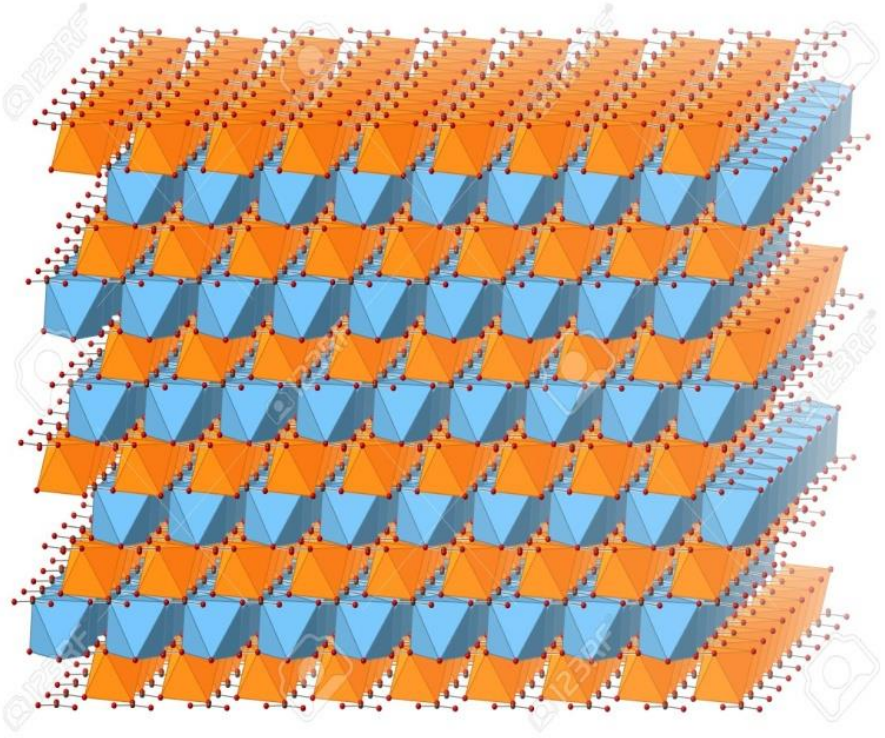

Figure 5: Mineral Dolomite Crystal

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The chemical compounds of talcum powder composition are given in the (**Table 1**) with their percentage.

Table 1: Percentage of Chemical Compounds Exist in Different Type of Talcum Powder

Name	Chemical Compound	Rate
Talcum	Silicon dioxide (SiO_2)	60% - 67%
	Magnesium oxide (Mgo)	30 – 31 %
	Aluminum tri oxide	0.5 %
Calcite	$CaCO_3$	95 -97%
	Free silica	1%
	Other oxides	0.55%
China Clay Powder	SiO_2	45-47%
	Al_2O_3	33-38%
	Fe_2O_3	1% max
	Cao	1% max
Dolomite Powder	$CaCO_3$	45-54%
	$MgCO_3$	35-38%
	Silica total insoluble	10% max

Methodology

This method is applied to determine the calcium content in four different talc powders which called a complexometric titration. EDTA (ethylenediaminetetraacetic acid) is a very large molecular was used to form a complex with calcium ions. In this study four samples of talcum powders were selected and determined their quantity of calcium by the complexometric titration method.

A metallochromic indicator was used before titration to determine the endpoint of the reaction which acts as complex agents that is responsible for the color change after the combination with metal ions.In EDTA titrations, a variety indicator can be used. In this research, the Eriochrome black T (EBT) indicator will use as shown in the structure below (**Figure 6**).

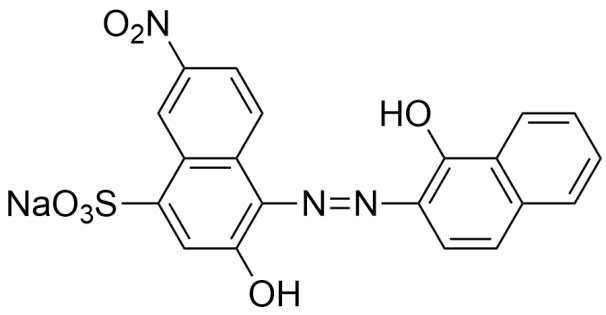

Figure 6: Eriochrome black T (EBT) Structure

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Experimental Section

1. Determination of Calcium by $KMnO_4$

Materials: 100 mL beaker, 250 mL conical flask, 250 mL standard flask, filters paper, No. 20 mL pipette, and burette.

Reagents: EDTA (ethylenediaminetetraacetic acid), Sodium hydroxide, distilled water, dilute hydrochloric acid**,** oxalic acid prepared by dissolving 0.21g/100 mL, $KMnO_4$ solution prepared by dissolving approximately 0.26 g in 250 mL standard flask .

Principle: The estimation of Ca^{+2} is carried out by conversion into oxalate which is estimated by titrating against standard $KMnO_4$ solution is standardized by using standard oxalic acid solution. In acidic medium $KMnO_4$ oxidizes oxalate ions to CO_2 gets reduced to Mn (11) ion

$$2MNO_4^- + 5C_2O_4^{2-} + 16H^+ \longrightarrow 2Mn^{2+} + 10CO_2 + 8H_2O$$

I. Standardization of KMnO4

The solution of oxalic acid and $KMnO_4$ is prepared. $KMnO_4$ solution is standardized by titrating against standard oxalic soln. For this purpose take 25 mL of oxalic acid solution in a flask. Add 20 mL dil. H_2SO_4 and heat the contents to above 70 ^{0}C. Run in $KMnO_4$ solution from the burette till a permanent pink colored is produced. Repeat the titrations till concurrent readings are obtained. The strength of $KMnO_4$ is calculated as follows 25 mL of N/30 oxalic acid =1/2 mL of N_2 $KMnO_4$ solution. Normality of $KMnO_4$ = N_2 = $25/v_2*30$.

II. Standardization of the EDTA Solution

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.

III. Preparation of Sample Solution:

Weigh accurately about 1g of talcum powder into 100 mL beaker and add 10 mL of conc. HCI with measuring jar and boil carefully till the volume reduces to 2 mL. Then add 30 mL of distilled water and filter through paper No.42. Collect the filtrate quantitively along with the washings with distilled water into 250 mL standard flask. Make up the volume up to the mark with distill water. Shake well for uniform concentration.

IIII. Estimation of Ca^{+2}:

Take 25 mL of these samples solution into a conical flask. Then add 20 mL of dil . H_2SO_4 and heat the contents to about 70℃. Run in $KMnO_4$ solution from the burette till a permanent pink colored is produced. Repeat the titration till concurrent readings are obtained, then

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calculate the morality of this solution and calculate the amount of Ca^{+2} present in this solution (**Table 2**).

Table 2: Amount of Ca^{+2} present in the selected sample by $KMnO_4$

Samples	Weight g/250 mL
English lavender	0.024
Ashoka	0.028
Emami	0.026
Wipro	0.022

2. Determination of Calcium by Substitution Method

Principle: Calciumions are titrated with EDTA a relatively stable calcium complex is formed. The calcium ions are alone no sharp endpoint can be obtained with eriochromeblak–T and the transition from red to blue.

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

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 (**Table 3**).

I. 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.

II. 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 3. Amount of Ca^{+2} present in the selected sampleby substitution method

Samples	Weight gm/250 ml
English lavender	0.032
Ashoka	0.034
Emami	0.024
Wipro	0.021

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Results and Discussion

The result of the study indicated the EDTA-complex was obtained through six coordination bonds between the EDTA and the calcium metal ion (**Figure 7**). The equivalence point of the reaction was determined through the solution color by the Eriochrome Black T indicator.

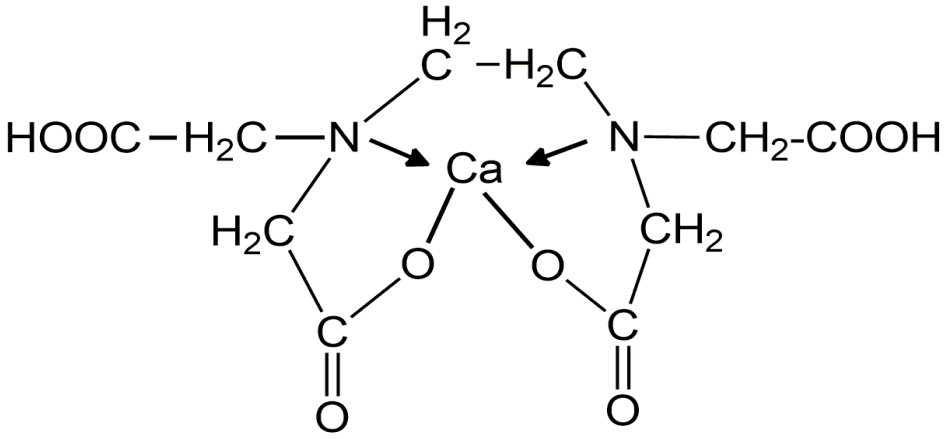

Figure 7: Calcium-EDTA Complex

In complexometric titrations, the Eriochrome Black T indicator combined with a metal ion then changes the color to red after forming the complex compound. However, the calcium ion complex titrated with EDTA to form a stronger complex at certain pH value.The sample solution containing the calcium ions was mixed with the indicator to form a complex with the pink/red calcium ion-indicator (**Figure 8**).The EB-T indicator reverts to its blue color indicating the endpoint when the calcium ion complex has been completely replaced by the calcium ion-EDTA complex (**Figure 9**).

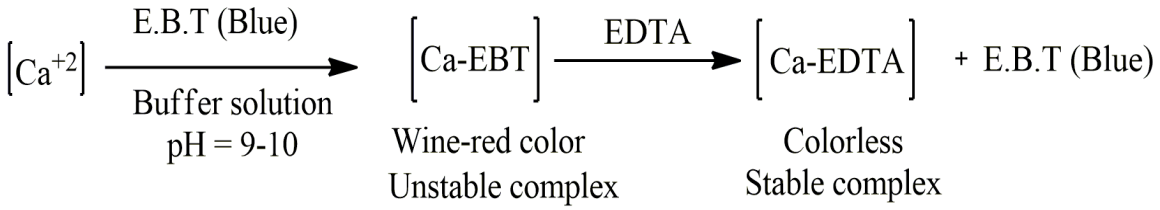

Figure 8: Complexometric Titrations of Calcium ion

The reactions with metal ions are pH dependent since the EDTA is an acid substance with four weak acid dissociations and the titration should be in acidic solution. Therefore, pH must be controlled by using the buffer solutions. This reaction required at least pH 8 values for the successful titration of calcium ions with EDTA and for magnesium ions are about 10. The calcium metal is difficult to titrated with the EDTA because, the complex formed between the calcium ion and the EBT indicator are not stable.Therefore a sharp color change of EBT indicator during the titration may not observed.

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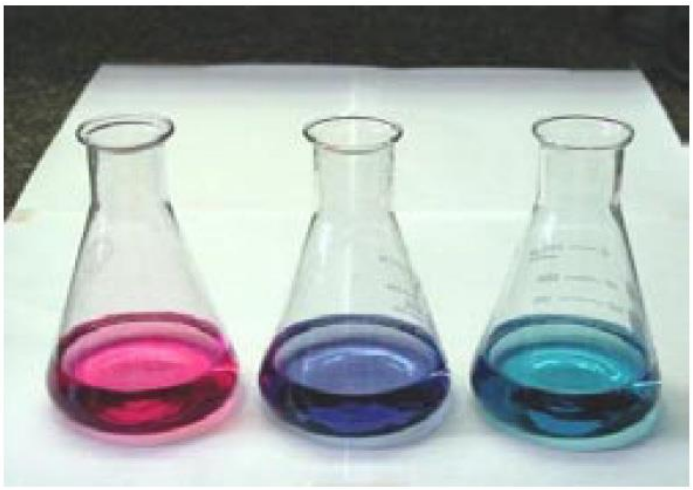

Figure 9: Colour changes for calcium-EDTA titration. Left flask: pink/ red colour before endpoint. Centre flask: before the endpoint purple colour. Right flask: blue colour at endpoint of titration.

Conclusion

The significant role of the talcum powder is interested around the world due to the importance of calcium ion in their mixture. Therefore, four selected talcum powder from different brand were used to determine the calcium ion in their composition by two method, by $KMnO_4$ and substitution method. The result showed that the Ashoka has highest quantity whereas Wipro was the lowest quantity among the others in both methods. However, the calcium ion is important in the manufacture of talcum powder.

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