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Rapid Determination of Mg, Fe, Al, Etc., in Rock Phosphate Samples using Atomic Absorption Spectrophotometer

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Abstract

There are several methods for the determination of Mg, Fe, Al and other elements in phosphorite samples. The method of analysis is developed in chemical laboratory of RODL, IBM, Bangalore for rock phosphate sample which is not only very fast and accurate but also maximum number of required elements can be analysed in the prepared solution. This method was used for the analysis of ore dressing products obtained while carrying out the beneficiation studies on rock phosphate samples from China.

The sample is treated with aquaregia followed by perchloric acid. The prepared solution is used for the determination of maximum number of elements like Mg, Fe, Al etc., by Atomic Absorption Spectrophotometer. By taking suitable aliquot from the solution, P_2O_5 can be analysed by volumetric or gravimetric method. Residue is used for the determination of SiO_2.

INTRODUCTION

Rock phosphate is mainly valued for its phosphorous content. The processing of phosphate ores can be divided into three categories. These are acid, electric furnace treatment and physical treatment. The optimum beneficiation route is naturally determined by the character of the ore and desired end product of the process. As the predominant use of the phosphate rock is in the production of fertilizers, most of the rock is used for making wet process phosphoric acid, the principle intermediate compound in the fertilizer manufacture.

The acidulation converts the phosphate into a form more readily usable in agriculture. Several phosphate products are used in pharmaceutical, ceramics, silk, textile, sugar refining etc. Trisodium phosphate is used as a detergent.

The rock phosphate used in fertilizer and other industries has to meet certain specifications, *e.g.* for fertilizer and phosphoric acid industries, P_2O_5 content should be minimum 32 – 35%. The presence of total aluminium and iron in terms of oxide beyond 3% is regarded not desirable as they unnecessarily consume too much of suphuric acid and also cause reversal of water soluble compound in super phosphate. In the same manner, the presence of MgO beyond 0.5% is not desirable, as it tends to insoluble compound in super phosphate, and also causes filtration difficulty.

In order to meet the requirements of mineral industry, beneficiation studies were carried for rock phosphate sample received from China in Indian Bureau of Mines, to get the desired grade by adopting different techniques. During the process, large number of products like concentrates, middlings and tailings were generated at various stages. It is very much essential to analyse number of samples rapidly for various parameters. Conventional methods like volumetric and gravimetric estimations for the determination of Al, Fe, Mg etc., in the lower range are not only tedious and time consuming (as it involves many separation steps during analysis), but also expensive. AAS method is most suitable for the determination of aluminium, iron and magnesium in rock phosphate samples.

Because of the above mentioned reasons, chemical analysis technique for the analysis of phosphorite sample was developed in the chemical laboratory of Indian Bureau of Mines to meet the requirements of mineral processing engineers.

Sample Decomposition Technique

0.5 gram of weighed sample is taken into a 250 ml beaker. About 20 ml of aquaregia (a 3:1 mixture of hydrochloric : nitric acid) is added and then evaporated to dryness on a hot plate. This helps in the determination of SiO_2 in the same treatment of the samples. The beaker is removed from the hot plate and cooled. About 15 ml of 70% perchloric acid is added to the beaker and allowed to fume off for half an hour in the fuming cupboard (under a fume hood). Digestion of the mass with perchloric acid enables the dissolution of most metal perchlorates easily in hydrochloric acid. Moreover, dilute perchloric acid does not cause an interference when used in AAS. Then it is extracted with 15 ml of 1:1 hydrochloric acid.

Contents of the beaker are transferred to the filter (whatman filter paper No.40) and washed with minimum volume of hot water and the solution is made to 250 ml in a standard volumetric flask with water.

This solution is used for the determination of various constituents like MgO, Fe_2O_3, Al_2O_3 etc.,

Determination of SiO_2

Filter paper along with residue is taken in a platinum crucible and ignited at 950° C in a muffle furnace for half an hour. The crucible is cooled in a dessicator and weighed (W_2) . About 3 – 4 drops of H_2SO_4 and 10 ml of concentrated HF are added to the platinum crucible and evaporated the solution on the hot plate to volatilize SiO_2. Residual H_2SO_4 is fume off and finally the crucible is ignited at 950° C for 10 minutes in the furnace. The crucible is cooled and weighed (W_1). The percentage of SiO_2 is calculated from the difference in weight.

Experimental Methods

The estimation of Al, Mg and Fe were carried out using a GBC Avantha, AAS. All the other parameters are prescribed in the instrument's manual. The instrument was calibrated using different E-Merck standard solutions of Fe, Mg and Al to read concentrations directly.

All the reagents used were AR grade and all solutions prepared in demineralized water.

Determination of Mgo

Suitable aliquot is taken into 100 ml flask and 1 ml of 20% Strontium chloride / Lanthanum chloride is added and the solution is made up to 100 ml with water. (Strontium chloride / Lanthanum chloride is added to control the chemical interference from Si, P, Al etc.,). The solution is aspirated to AAS and compared with the standards.

Wave length : 285.2 nm
Air acetylene flame is used.

Determination of Fe_2O_3

Suitable aliquot is taken from the prepared solution into a 100 ml flask and made up the solution with water and then aspirated to AAS.

Wave length : 248.3 nm
Air acetylene flame is used.

Determination of Al_2O_3

Suitable aliquot is taken from the prepared solution into a 100 ml flask and 1 ml 20% potassium

chloride solution is added and the solution made up with water (potassium chloride solution is added to suppress the ionization of aluminium). Then the solution is aspirated to AAS.

Wave length : 396.2 nm
Nitrous oxide - acetylene flame is used.

Determination of P_2O_5

50 ml of aliquot is taken from the prepared solution. P_2O_5 is estimated volumetrically / gravimetrically using quinoline and sodium molybdate solution.

RESULTS AND DISCUSSION

Chemical analysis results of some of the ore dressing products of Rock phosphate sample are given below

Beneficiation products	Values in Percentage				
	SiO_2	Al_2O_3	Fe_2O_3	MgO	P_2O_5
1	5.80	0.82	1.07	0.57	33.20
2	4.42	0.81	1.31	1.15	31.86
3	11.98	1.00	2.02	0.57	28.87
4	57.23	1.34	4.71	0.33	4.82
5	5.86	0.76	1.05	0.66	32.77
6	28.73	2.21	6.16	1.55	15.11

Complete analysis of rock phosphate samples can be carried out rapidly in one single treatment for various parameters with good precision and accuracy using AAS. The chemical analysis technique adopted is cost effective and the time required for the analysis of the samples is considerably reduced.