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Physicochemical Characterization of Ayuquduq Phosphorite by Xrf, Ftir and Xrd Methods

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Abstract:
The chemical, molecular and phase composition of a phosphorite sample from the Ayuquduq area was studied by energy-dispersive X-ray fluorescence (EDXRF), Fourier-transform infrared (FTIR) spectroscopy and X-ray diffraction (XRD). The sample contains 25.84 % P_2O_5, 45.0 % CaO, 8.1 % SiO_2, 3.97 % Al_2O_3, 2.49 % Fe_2O_3, 2.12 % MgO and 1.28 % K_2O, and the CaO/P_2O_5 mass ratio is 1.74. The FTIR spectrum shows phosphate bands at 605 and 571 cm^{-1} and a strong ν_3 PO_4 absorption near 1040–1100 cm^{-1}, together with carbonate bands at 1797, 1430–1450, 876 and 714 cm^{-1} and a quartz band at 799 cm^{-1}. XRD reflections of quartz and calcite were identified, while the apatite reflections were weak. The results indicate a carbonate-rich phosphorite in which a carbonate-fluorapatite phase coexists with calcite and quartz. The high CaO/P_2O_5 ratio and the 0.25 ratio of (Al_2O_3 + Fe_2O_3) to P_2O_5 should be considered when choosing acid processing routes.

Keywords: Phosphorite, Ayuquduq, X-ray fluorescence, FTIR spectroscopy, X-ray diffraction, carbonate-fluorapatite, calcite, CaO/P2O5 ratio.

Introduction

Phosphorites of the Central Kyzylkum region are the main domestic raw material for phosphorus fertilizer production in Uzbekistan. Their mineralogy is complex: the phosphate mineral is francolite, and the gangue includes calcite, dolomite, gypsum and quartz [1]. Neighbouring deposits differ noticeably. For example, the Aznek ore contains 22.55 % P_2O_5 and much less iron than the Tashkura ore, which makes it more suitable for wet-process phosphoric acid [2]. Thermal treatment of Kyzylkum phosphorites is also studied as a way to remove fluorine and obtain defluorinated feed and fertilizer phosphates [3].

The choice of a processing route therefore depends on the composition of each ore body. The most important parameters are P_2O_5 content, carbonate content (expressed by the CaO/P_2O_5 ratio), and the amounts of iron, aluminium and magnesium. Sedimentary apatites are usually carbonate-substituted, and their crystallographic and chemical features control how reactive they are towards acids [4]. Data

on the Ayuquduq phosphorite are scarce. The aim of this work is to characterise a sample of this raw material by three complementary methods: XRF, FTIR and XRD.[5]

Materials and Methods

A phosphorite sample labelled "Ayuquduq" was ground and analysed in powder form. The elemental composition was determined on a Rigaku NEX DE energy-dispersive X-ray fluorescence spectrometer. The measurement was made in air with a 10 mm diaphragm and without sample spinning. Three excitation conditions were used: Mo-scatter (60 kV, 200 µA), Mid-Z (35 kV, 342 µA) and Low-Z (6.5 kV, 352 µA), each for 100 s. Results were calculated by the standardless fundamental-parameter (FP) method with scatter correction and expressed as oxides.[6]

The infrared spectrum was recorded on a Shimadzu FTIR spectrometer in transmittance mode from 4000 to 400 cm^{-1}. The X-ray diffraction pattern was recorded in θ–2θ geometry from 10° to 80° 2θ. [Specify the diffractometer model, radiation (e.g. Cu Kα), step size, counting time, IR sample preparation (e.g. KBr pellet) and the ICDD/PDF cards used for phase identification.] Peak positions in Table 2 were read from the recorded profile with an accuracy of about ±0.1° 2θ.[7]

Results and Discussion

Chemical composition

The main components determined by XRF are listed in Table 1 and shown in Fig. 1. The sample is dominated by CaO (45.0 %) and P_2O_5 (25.84 %). Silica (8.1 %), alumina (3.97 %), iron oxide (2.49 %), magnesia (2.12 %) and potassium oxide (1.28 %) form the main impurities. SO_3 (0.307 %) and Cl (0.0545 %) are low.[8]

Table 1. Main components of the Ayuquduq phosphorite (EDXRF, FP method), mass %

Component	Content	Component	Content
P_2O_5	25.84	MgO	2.12
CaO	45.0	K_2O	1.28
SiO_2	8.1	TiO_2	0.391
Al_2O_3	3.97	SO_3	0.307
Fe_2O_3	2.49	Cl	0.0545
SrO	0.0394	MnO	0.0421
CaO/P_2O_5	1.74	$(Al_2O_3+Fe_2O_3)/P_2O_5$	0.25

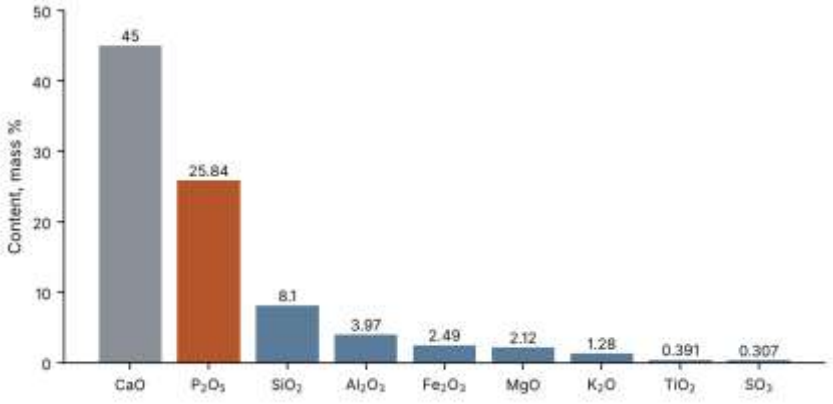


Figure 1. Main oxide components of the Ayuquduq phosphorite (EDXRF)

The CaO/P_2O_5 mass ratio of the sample is 1.74. Stoichiometric fluorapatite has a ratio of 1.32. If the whole P_2O_5 is assigned to fluorapatite, about 10.9 % CaO remains in excess. Assigned entirely to calcite, this excess corresponds to roughly 19.5 % $CaCO_3$. This is an upper estimate, because carbonate-

fluorapatite itself carries more calcium per unit of P_2O_5 than pure fluorapatite. The apatite phase is estimated at 61–70 % of the sample, depending on whether pure fluorapatite (42.2 % P_2O_5) or a francolite with about 37 % P_2O_5 is assumed. All of the MgO (2.12 %) would correspond to at most 9.7 % dolomite, but magnesium may also be held in clay minerals.[9]

The total of the determined oxides is about 92 %. The remainder is mainly carbonate CO_2 and fluorine, which EDXRF cannot measure, and possibly water. These components should be measured directly (loss on ignition or CO_2 analysis, and fluorine analysis). The $(Al_2O_3 + Fe_2O_3)/P_2O_5$ ratio is 0.25 and the MgO/P_2O_5 ratio is 0.082. These impurities pass into the acid during decomposition and affect phosphoric acid quality and fertilizer properties.[10]

The standardless result also reports MoO_3 (1.06 %), Ag_2O (0.728 %) and Nb_2O_5 (0.637 %). These values are unusually high for a sedimentary phosphorite. The Mo L, Nb L and Ag L lines lie in the 2.1–3.0 keV range, where they overlap with the strong P Kβ and S Kα lines and with the argon line from measurement in air. They are therefore treated as possible spectral-overlap artefacts and are not discussed further. They must be checked by an independent method such as ICP-MS. The EDXRF spectrum (Fig. 2) shows the dominant Ca and P signals, with Fe, Sr and Zr peaks in the mid-energy range.[11]

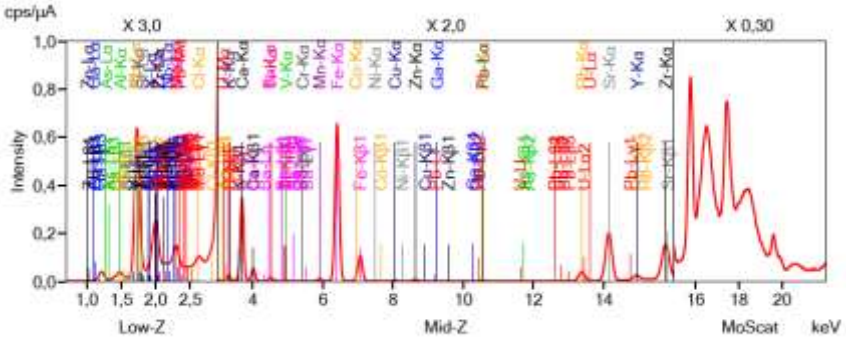


Figure 2. EDXRF spectrum of the Ayuquduq phosphorite (Low-Z, Mid-Z and Mo-scatter conditions)

FTIR spectrum

The FTIR spectrum (Fig. 3) contains three groups of bands. Phosphate groups give a very strong broad absorption near 1040–1100 cm^{-1} (ν_3 PO_4), bands at 605.65 and 570.93 cm^{-1} (ν_4 PO_4) and a band at 470.63 cm^{-1} in the ν_2 region. These bands confirm an apatite-group phosphate. Carbonate gives a strong broad band at about 1430–1450 cm^{-1} (ν_3 CO_3), a sharp band at 875.68 cm^{-1} (ν_2), a band at 713.66 cm^{-1} (ν_4), and combination bands at 1797.66 cm^{-1} and 2515.18 cm^{-1}. This set matches calcite [5]. The band at 875 cm^{-1} may also include carbonate substituted in the apatite lattice, which is typical of sedimentary francolite [4].

The band at 798.53 cm^{-1} belongs to the Si–O symmetric stretching of quartz [6], which agrees with the 8.1 % SiO_2 found by XRF. Weak bands at 2924.09 and 2873 cm^{-1} indicate C–H bonds of organic matter. The bands at 2368.59 and 2345.44 cm^{-1} come from atmospheric CO_2, and the absorption above 3400 cm^{-1} together with the band near 1620 cm^{-1} come from adsorbed water.

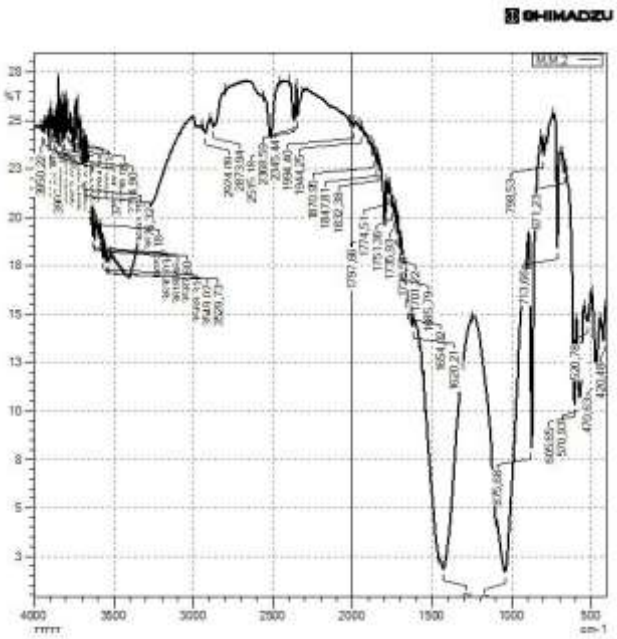


Figure 3. FTIR spectrum of the Ayuquduq phosphorite

X-ray diffraction

The main reflections of the diffraction pattern (Fig. 4) are summarised in Table 2. The strongest peaks at about 26.8° and 21.0° 2θ, together with reflections near 36.6, 39.6, 42.6, 50.3, 60.1 and 68.3° 2θ, correspond to quartz. The reflection at about 29.6° 2θ, with weaker peaks near 43.4, 47.7 and 48.7° 2θ, corresponds to calcite. This agrees with the carbonate bands in the FTIR spectrum. The positions are shifted by about +0.1–0.2° relative to the reference values, probably because of sample displacement.[12][13]

A strong reflection at about 27.9° 2θ is not assigned to quartz or calcite. It may belong to a feldspar, which would agree with the 1.28 % K_2O and 3.97 % Al_2O_3 found by XRF, but this requires confirmation. The main apatite reflections expected at about 25.9, 31.8, 32.2 and 32.9° 2θ are weak in this pattern, even though the sample contains 25.84 % P_2O_5. Possible reasons include the poor crystallinity typical of sedimentary francolite [14], or a difference between the sample used for XRD and the one used for XRF. Full phase identification and quantification, for example by Rietveld refinement, should be done on the raw diffraction data.[15]

Table 2. Main XRD reflections and their tentative assignment

2θ (approx.), °	Relative intensity	Assignment
21.0	medium	quartz
26.8	very strong	quartz
27.9	strong	unassigned (feldspar?)
29.6	medium	calcite
36.6; 39.6; 42.6	weak	quartz (39.6 also calcite)
43.4; 47.7; 48.7	weak	calcite
50.3; 60.1; 68.3	weak	quartz

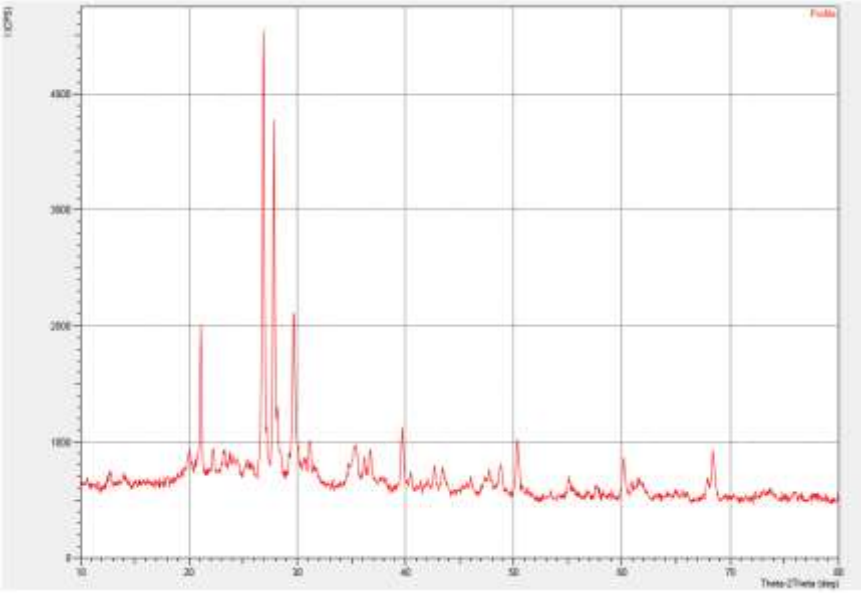


Figure 4. X-ray diffraction pattern of the Ayuquduq phosphorite

Conclusion

The Ayuquduq phosphorite contains 25.84 % P_2O_5 and 45.0 % CaO (CaO/P_2O_5 = 1.74). The main impurities are SiO_2 (8.1 %), Al_2O_3 (3.97 %), Fe_2O_3 (2.49 %), MgO (2.12 %) and K_2O (1.28 %).

FTIR confirms an apatite-group phosphate (ν_3 PO_4 near 1040–1100 cm^{-1} and ν_4 PO_4 at 605 and 571 cm^{-1}), calcite (1430–1450, 876, 714 and 1797 cm^{-1}) and quartz (799 cm^{-1}).

XRD clearly identifies quartz and calcite. The apatite reflections are weak, and the phase composition must be refined on the raw diffraction data.

The ore is a carbonate-rich phosphorite. The excess CaO (up to about 19.5 % calcite) and the (Al_2O_3 + Fe_2O_3)/P_2O_5 ratio of 0.25 will increase acid consumption and should be considered when choosing a processing route. The reported Mo, Nb and Ag contents need independent verification.

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