The American Journal of Applied Sciences
ISSN 2689-0992 Volume 08 - 2026

# Sulphuric Acid Co-Processing of Central Kyzylkum Phosphorite Slime with Activated Sludge: Distribution of Humic Substances and Nutrients Between Liquid and Solid Organomineral Fertilizers

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Received: 16 Dec 2025 | Received Revised Version: 30 Dec 2025 | Accepted: 14 Jan 2026 | Published: 31 Jan 2026

Volume 08 Issue 01 2026 | Crossref DOI: 10.37547/tajas/Volume08Issue01-13

## Abstract

*Phosphorite slime (FSh) from Central Kyzylkum ore beneficiation was co-processed with municipal activated sludge (FL) into liquid and solid organomineral fertilizers (OMF). Mixtures containing 10- 80 wt.% FL were decomposed with 30 % H2SO4 at 50- 90 % of the stoichiometric acid demand of CaO and separated by vacuum filtration. The slime controlled P2O5 and CaO, whereas organic matter, humic acids (HA), fulvic acids (FA) and K2O followed the sludge share; the FA:HA ratio of the slurry (2.42- 2.44) equalled that of the sludge. On filtration, FA and water-soluble organic matter passed mainly into the filtrate, while 88.9- 95.3 % of HA and 86.0- 91.9 % of CaO remained in the cake. At FSh:FL = 60:40, raising the acid norm from 50 to 80 % increased P2O5 transfer to the liquid from 37.1 to 50.0 %. The process yields a fulvate-rich liquid fertilizer and a humic- and calcium-rich solid fertilizer.*

Keywords: Phosphorite slime, activated sludge, organomineral fertilizer, sulphuric acid decomposition, humic acids, fulvic acids, phase partitioning.

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**Cite This Article:** Mukhammatova Umida Khusan qizi, Temirov Uktam Shavkatovich, & Sharipov Sanat Shuhrat ugli. (2026). Sulphuric Acid Co-Processing of Central Kyzylkum Phosphorite Slime with Activated Sludge: Distribution of Humic Substances and Nutrients Between Liquid and Solid Organomineral Fertilizers. The American Journal of Applied Sciences, 8(01), 135–139. https://doi.org/10.37547/tajas/Volume08Issue01-13

## 1. Introduction

Phosphate rock is a finite resource concentrated in a few countries, so low-grade ores and phosphorus-bearing wastes are increasingly important for long-term supply [1], [2]. The Central Kyzylkum phosphorites, the main phosphate source of Uzbekistan, are carbonate-rich and low-grade, and their beneficiation leaves fine slime that is not processed further [3], [4]. Excess activated sludge from municipal treatment plants is another phosphorus-rich waste [5], but its direct land application is limited by

pathogens and potentially toxic elements [6]. Joint processing of the two wastes could convert both into fertilizer.

## 2. Related Work

Combining sewage sludge with a mineral phosphate source into organomineral fertilizers (OMF) gives products with controlled phosphorus release [7], [8]. The humic substances of the organic component can reduce phosphate fixation in soil through humic- metal-phosphate complexes and raise extractable phosphorus [9], [10]. Acid processing of Central Kyzylkum phosphorite with a humic component has been reported for oxidized coal [11], and the authors have described nitric acid processing of Aznek phosphorite with activated sludge into a single dried product [12]. Phosphorite slime has not been processed in this way, and it is not known how humic fractions and nutrients split between the liquid and solid products when a slime-sludge slurry decomposed with sulphuric acid is filtered.

## 3. Scope Of Research

The aim of this work was to establish how the FL:FSh mass ratio and the sulphuric acid norm affect (i) the composition of the slurry, (ii) the composition of the liquid and solid OMF obtained by filtration and (iii) the distribution of P2O5, CaO, K2O and humic fractions between the two phases, and on this basis to select working conditions for the process.

## 4. Methodology

Phosphorite slime (FSh), formed when Central Kyzylkum ore is washed free of chlorine, and activated sludge (FL) from the Navoi city treatment plant were used (Fig. 1). The slime was ground to below 0.25 mm. Mixtures with FL:FSh mass ratios of 10:90 to 80:20 were decomposed with 30 % H2SO4. The acid dose was 50, 60, 70, 80 or 90 % of the stoichiometric demand of the CaO in the mixture. Decomposition was run in a glass reactor at 50- 60 °C and 200- 250 rpm for 40- 50 min. The slurry was sampled and then separated on a Büchner funnel at 550- 600 mm Hg into a liquid OMF (filtrate) and a solid OMF (cake).

Total P2O5 was determined photometrically as the phosphovanadomolybdate complex (λ = 440 nm) and CaO by EDTA titration. HA and FA were determined by extraction with 0.1- 0.5 M NaOH and precipitation of HA at pH 1- 1.5. Organic matter was determined by loss on ignition at 550 °C and water-soluble organic matter (WSOM) by aqueous extraction and gravimetry. The transfer degree is the percentage of a component's mass in the slurry that passed into the filtrate.

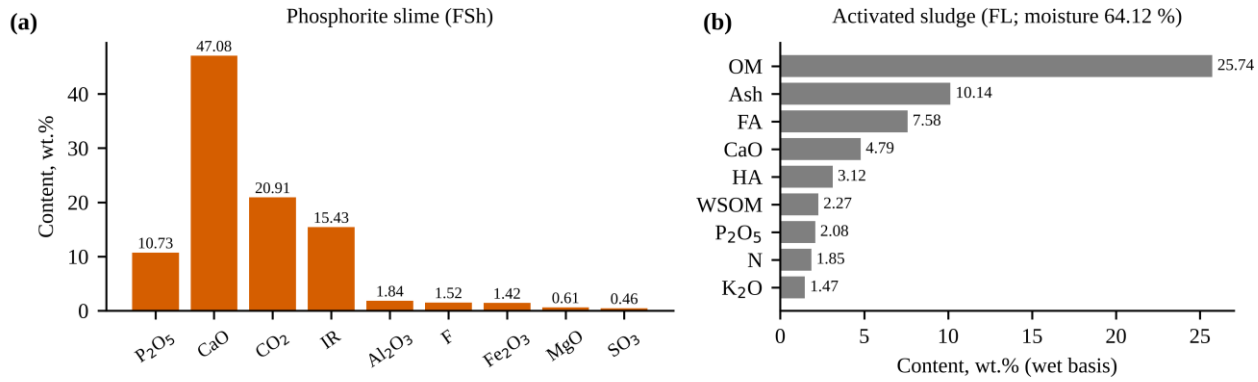



**Fig. 1. Composition of the raw materials: (a) phosphorite slime (FSh), IR - insoluble residue; (b) activated sludge (FL) on a wet basis, OM - organic matter, FA - fulvic acids, HA - humic acids, WSOM - water-soluble organic matter.**

## 5. Experimental Results and Discussion

A. Raw materials. FSh contains 10.73 % P2O5, 47.08 % CaO and 20.91 % CO2 (Fig. 1a). Its CaO:P2O5 ratio, calculated from these data, is 4.39, so most of the acid is consumed by calcite rather than apatite. Only 11.58 % of its P2O5 is citrate-soluble. XRD identified fluorcarbonate-apatite with calcite, gypsum and quartz,

and about 85 % of the particles fall in the −0.2+0.1 mm range. FL (64.12 % moisture) contains 25.74 % organic matter, including 7.58 % FA and 3.12 % HA (Fig. 1b).

B. Composition of slurry and products. Fig. 2 shows the slurry, filtrate and cake at the 80 % acid norm. As the FL share rose from 10 to 80 %, P2O5 in the slurry fell from 3.57 to 2.77 % and CaO from 14.39 to 9.20 %, while organic matter rose from 0.86 to 14.31 %. CaO was concentrated in the cake (28.93- 14.66 %), where it is retained as gypsum, and the filtrate contained only 2.62- 2.87 %. P2O5 was distributed more evenly: 3.59- 2.44 % in the filtrate and 3.54- 3.04 % in the cake. The humic fractions separated sharply. At FL 80 % the cake contained 3.024 % HA but the filtrate only 0.185 %, whereas the filtrate held 7.380 % FA against 1.575 % in the cake. K2O, which comes almost entirely from the sludge, reached 0.73- 0.89 % in both products.

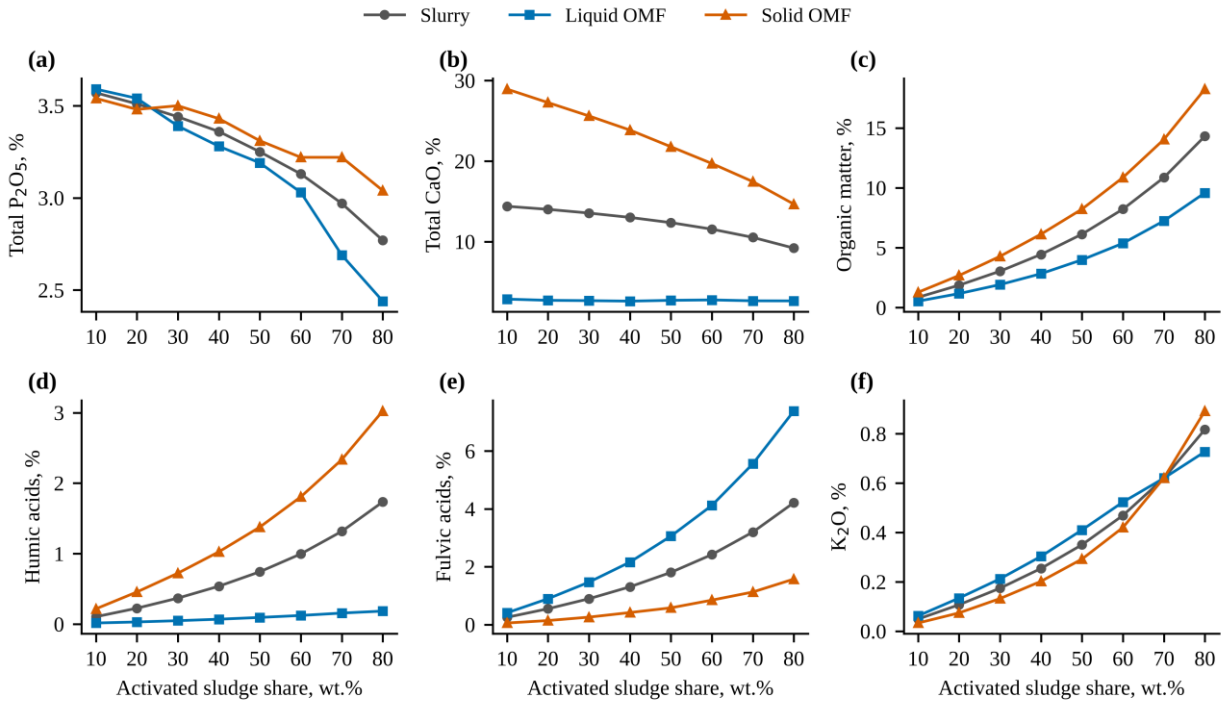



**Fig. 2. Composition of the slurry, liquid OMF (filtrate) and solid OMF (cake) at an acid norm of 80 % as a function of the activated sludge share: (a) total P2O5, (b) total CaO, (c) organic matter, (d) humic acids, (e) fulvic acids, (f) K2O.**

Across all 40 combinations of ratio and acid norm the FA:HA ratio of the slurry stayed within 2.42- 2.44, equal to that of the sludge (7.58:3.12 = 2.43). The humic substances therefore come from the sludge, with no measurable new humification during the short treatment. Between the 50 and 90 % norms, HA and FA in the slurry fell by the same fraction at each ratio, from 30.7 % at FL 10 % to 14.7 % at FL 80 %, and P2O5 and CaO fell as well. This pattern indicates dilution by the larger volume of 30 % acid rather than selective destruction of humic matter.

C. Distribution between phases. The transfer data (Fig. 3) divide the components into three groups. FA (61.2- 90.6 %) and WSOM (55.7- 85.0 %) pass mainly into the liquid. P2O5 (32.7- 57.2 %), K2O (32.5- 71.6 %) and total organic matter (23.7- 35.2 %) are split between the phases. HA (4.7- 11.1 %) and CaO (8.1- 14.0 %) remain mainly in the cake. This follows the operational definition of the fractions: HA are insoluble below pH 2, whereas FA remain soluble in both acidic and alkaline media [14]. At FSh:FL = 60:40, increasing the acid norm from 50 to 80 % raised P2O5 transfer from 37.1 to 50.0 % and FA transfer from 64.9 to 84.3 %; going to 90 % added only 1.1 and 0.7 percentage points, so 80 % is a reasonable working norm. The rise agrees with the known increase in apatite decomposition with acid excess [13]. At a fixed norm, a higher sludge share

reduced the transfer of every component except CaO, most strongly for K2O (from 70.6 to 40.4 %), which suggests that part of the sludge potassium is held in undissolved solids; the present data do not identify its form.

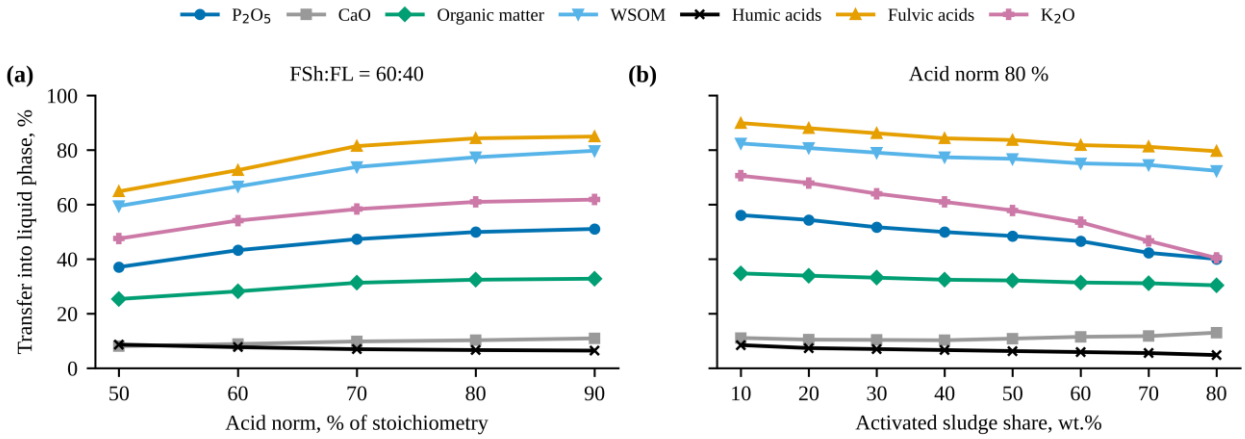



**Fig. 3. Transfer of components from the FSh- FL slurry into the liquid phase on vacuum filtration: (a) as a function of the acid norm at FSh:FL = 60:40; (b) as a function of the activated sludge share at an acid norm of 80 %.**

The two products are therefore complementary. The liquid OMF is a fulvate-rich solution containing P2O5 and K2O, with an FA:HA ratio of 23.7- 41.5 at FL 80 % (acid norms 50- 90 %). The solid OMF carries HA, gypsum-bound calcium and residual phosphate; retaining HA in the phosphate-bearing solid is useful because humic- metal- phosphate associations limit phosphate fixation in soil [9], [10]. For FSh:FL = 60:40 and the 80 % norm, about half of the P2O5, 84.3 % of FA and 61.0 % of K2O go to the liquid OMF, and the cake retains 89.7 % of CaO and 93.3 % of HA. The proposed process therefore consists of mixing, decomposition, vacuum filtration, and drying of the cake at 80- 95 °C to 14- 15 % moisture followed by granulation. Available P2O5 and heavy metals [6] were not determined for these products and must be assessed before agronomic testing.

## 6. Conclusion

In sulphuric acid co-processing of Central Kyzylkum phosphorite slime with activated sludge, the slime controls P2O5 and CaO, while organic matter, humic substances and K2O are governed by the sludge share. The FA:HA ratio of the slurry (2.42- 2.44) equals that of the sludge, and its decrease with the acid norm reflects dilution. On filtration FA and WSOM pass mainly into the liquid OMF, more than 88 % of HA and 86 % of CaO remain in the solid OMF, and P2O5 and K2O are shared. At FSh:FL = 60:40 an acid norm of 80 % brings P2O5 transfer to 50 %. The process yields a fulvate-rich liquid fertilizer and a humic- and calcium-rich solid fertilizer.

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