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Geochemical Characterization and Ore Dressing Potential of Sokoto Phosphate Rock, Northwestern Nigeria

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Abstract Investigations were carried out for geochemical characterization of Sokoto Phosphate rock and to propose a suitable ore dressing process for the recovery of phosphate for fertilizer and allied applications. The study area is Iullemmeden Basin (Sokoto sector) in the Northwestern geopolitical zone of Nigeria. The phosphate rock occurs mainly in nodules, while some are found as pellets, especially in the southern part of the study area. The phosphate deposit occurs predominantly in the Dange Formation and a small occurrence in the Gamba Formation. The phosphatic nodular beds measure between 0.3 m and 0.1 m, while the nodular disseminations may be up to 6 m in thickness. The P_2O_5 values range from 20.18-36.25 wt. %, while the Jordan, Morocco, USA, Algeria, China and Syria phosphates have the range of 30-35 wt. %. A mixture of di(2-ethylhexyl) phosphoric acid (DEPA) and tri-octylphosphine oxide (TOPO) is proposed for the recovery of uranium from phosphates.

Keywords Sokoto, Phosphate Rock, Geochemical Characterization, Ore Dressing

1. Introduction

The study area is located in the Nigerian sector of the Iullemmeden Basin in the Northwestern geopolitical zone. According to Nwajide [11], the use of the term "Sokoto Basin" should be discontinued because it implied that there is an existence of a basin within a basin, which is far from being the case. Besides, the impression should not be created that the relatively small part of the basin that lies within Nigeria is in any way distinct or unique. In this paper, the Nigerian sector of the Iullemmeden Basin is referred to as the "Sokoto sector". The main aim of this paper is to geochemically characterize the Sokoto Phosphate rock and propose a suitable ore dressing process both for the efficient recovery of the phosphate as fertilizer, allied applications and for the full extraction of uranium and gypsum from the source mineral raw material. Sokoto phosphate rock occurs mainly in nodules, while some are found as pellets, especially in the southern part of the study area (Figure 1). According to Okosun and Alkali [19], the phosphate nodules occur either as thin nodular beds or as disseminations in shales and siltstones. The phosphate rock deposit has pronounced and extensive occurrence in the Dange Formation in comparison to its occurrence in the Gamba Formation. The phosphatic nodular beds measure between 0.3 m and 0.1 m, while the nodular disseminations may be up to 6 m in thickness [19]. The geochemical characterization of Sokoto phosphate rock in comparison to other phosphate rock deposits indicated that P_2O_5 values range from 20.18-36.25 wt. %, while the Jordan, Morocco, USA, Algeria, China and Syria phosphates have the range of 30-35 wt. %.

Ore dressing process requires separation of the selected mineral material from the associated uneconomic materials with the intent to minimize the impurities and increase the concentration of the desired mineral. The methods used include: size reduction via crushing and grinding, magnetic and electrolytic/wet chemical separation. There are three industrial solvent extraction methods of uranium from phosphate rock. They are (i) di(2-ethylhexyl) phosphoric acid and tri-octylphosphine oxide (DEPA-TOPO), (ii) Octyl pyrophosphoric acid and kerosene (OPPA), and (iii) mixture of mono-octyl phenyl phosphoric acid and di-octyl phenyl phosphoric acid (OPAP). However, the DEPA-TOPO method is preferred because it is the most cost-effective, chemically stable and exceptionally selective of uranium in hexavalent state (U^{6+}) coupled with its minimum solubility in phosphoric acid. From this process, gypsum is also recovered and the final beneficiated phosphate devoid of uranium can be used to produce environmentally-friendly fertilizers for food security [14].

2. Geology and Stratigraphy

The Iullemmeden Basin is a roughly circular sedimentary cover extending from Mali through Niger Republic and Benin Republic to the northwestern Nigeria in parts of Zamfara, Sokoto and Kebbi States [6,11]. The Nigerian sector of the basin ("Sokoto sector"), is made up of four unconformity-bounded lithostratigraphic packages, namely: the Upper Jurassic to Lower Cretaceous Gundumi-Illo Formations (non-marine "Continental Intercalaire"), the Maastrichtian Rima Group (comprising Taloka, Dukamaje and Wurno Formations), the Late Paleocene Sokoto Group (consisting of Dange, Kalambaina and Gamba Formations), and the Eocene to Miocene Gwandu Formation also referred to as "Continental Terminal" [7,11-12].

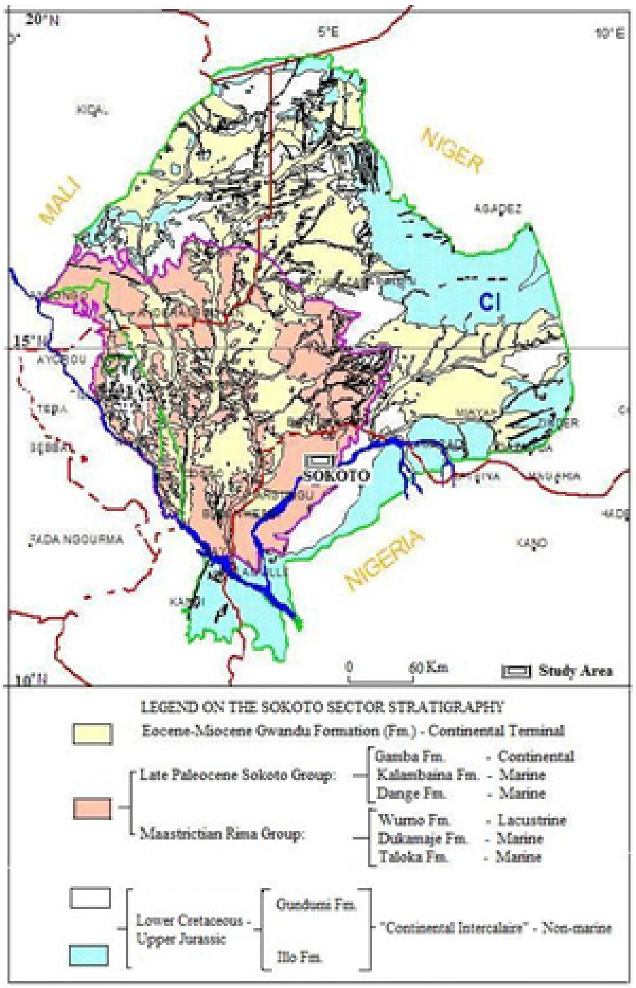

Figure 1. Geological Map of the Iullemmeden Basin showing Sokoto sector Study Area (After Inweh [26])

The Gundumi Formation and the Illo Formation are lateral equivalents and they are called "Continental Intercalaire." They are lateral equivalent to the Bima Sandstone in Northeast Nigeria. They have also been correlated to the Karoo Series of South Africa, and the upper part of the Nubian Sandstone in the Arabo-Nubian Shield. In addition, they have been correlated to the continental deposits of the Algerian-Moroccan Sahara which extends eastward to Egypt and the Sudan [6,12]. The Gundumi Formation lies unconformably on the Pre-Cambrian Basement Complex and it consists of basal conglomerates and gravels with sand and variegated clays increasing upwards. It has maximum thickness of 350m. The Illo Formation also lies unconformably on the Basement Complex and it consists predominantly of cross-bedded grits with a major intercalation of pisolithic and nodular clays. Laterites and lateritic ironstones form resistant cappings on top of the grits. The type section of Illo Formation is on a hill east of Gore village about 3.5 km north of Giro. Illo Formation has thickness of 240 m. The Gundumi and Illo Formations are made up of continental, fluviatile to fluvio-lacustrine sedimentary deposits [12].

2.1. Maastrichtian Deposits (The Rima Group)

The second depositional phase in the Sokoto basin began in the Maastrichtian with the deposition of the Rima Group unconformably in the Pre-Maastrichtian continental beds. The Rima Group is made up of three formations, namely: Taloka, Dukamaje and Wurno. Taloka Formation consists of brownish to reddish-purple siltstone with interbedded yellowish-brown claystone, carbonaceous shale and grey siltstone with black lignitic siltstone. The type locality of this formation is Taloka village, about 2 km east of Goronyo. The type section is on a hill that forms part of a continuous exposed ridge between Shinaka, Taloka to Goronyo [13]. The Taloka Formation consists of thinly-bedded or laminated siltstones with small load casts and bioturbation structures that are indicative of low energy marine environment [12]. The presence of lenticular bedding (flaser bedding) and wavy bedding in this formation further confirmed tidal-flat marine environment of deposition.

The Dukamaje Formation lies above the Taloka Formation and it consists predominantly of shales with some limestone and mudstones. The type section is at Dukamaje. The formation is very fossiliferous. The shales are rich in gypsum, reptilian bones and ferruginous altered concretionary limestone [12]. The Dukamaje Formation is lateral equivalent of the Patti Formation in the Bida Basin (North-Central Nigeria) and Nkporo Shales in the Anambra Basin (South-East Nigeria). The last member of the Rima Group is Wurno Formation and it lies above the Dukamaje Formation. Wurno Formation consists of pale, friable, fine-grained sandstones, siltstones and interbedded mudstones. Small-scale load cast, bioturbation structures and flaser bedding are abundant in Wurno Formation, thus indicating similar origin with the deposit of the Taloka Formation [12-13].

2.2. Paleocene Deposits (The Sokoto Group)

The third phase of sedimentary deposition in the Sokoto sector occurred in the Paleocene with the unconformable deposition of the Sokoto Group on the Rima Group. The Sokoto Group consists of Dange, Kalambaina and Gamba Formations, respectively. The three formations have marine origin. The Dange Formation is of Late Paleocene age, and it consists of slightly indurated bluish-grey shales with bands of gypsum and numerous irregular phosphatic noodles and pellets [12-13,15,18]. The shales are interbedded with thin layers of yellowish-brown limestone. The Dange Formation is richly fossiliferous. The type section of the formation is at Dange village about 28 km south of Sokoto, along Sokoto-Gusau road. The thickness is about 45 m [11-13]. Okosun [17] recovered agglutinated benthic foraminifera and ostracods that suggest transitional/marginal marine, shallow marine to inner neritic environment for the Dange Formation.

Kalambaina Formation is made up of marine deposited white clayey limestones interbedded shales. The limestone is classified as bioclastic wackestone [11]. The formation is richly fossiliferous and it has thickness of 20 m [12,17,20,6]. The Gamba Formation consists of fossiliferous and gypsiferous grey shale with phosphatic pellets and coprolites. Thin phosphatic micro-conglomeratic intercalations contain fish teeth and scales and moulds of bivalves [11]. The type section is located within the Sokoto Cement Company and the type locality is Gamba. According to Nwajide [11], the folded appearance of this formation is caused by slumping due to solution cavities in the underlying limestones of the Kalambaina Formation. The formation is capped by ferruginous oolite dated Upper Paleocene by Kogbe [6]. Similar assemblage of calcareous benthic foraminifera and ostracods found in Kalambaina are also present in this formation except for their low diversity and abundance [17].

2.3. Eocene Deposits (The Continental Terminal)

The fourth and last phase of the lithostratigraphic packages is the Eocene deposits called the Continental Terminal. The term, as now widely applied in western Africa, refers to the continental stratigraphic package represented in Nigeria by the Gwandu Formation, which unconformably overlies the marine Sokoto Group. It consists predominantly of red and mottled clays with sandstone intercalations. The top of the formation has widespread lateritic ironstones forming resistant cappings to weaker rocks [6]. The type section is located near Dutsin Badua, along Birnin Kebbi – Argungu road. It underlies a very extensive area of about 22,000 km^2 [11].

3. Material and Methods

Phosphate nodules and pellets from Dange and Gamba Formations, respectively, were analyzed for major elements, namely: P_2O_5, CaO, SiO_2, Al_2O_3, Fe_2O_3, MgO, Na_2O, K_2O, MnO, etc., using x-ray fluorescence (XRF) spectrometer with molybdenum secondary target and a molybdenum filter fitted with Cd-109 excitation source, ($t½$ = 1.34 years, E=22.2 KeV and 88.0 KeV) and Si (Li) detector with multi-channel analyzer. The phosphate samples were milled into 50 µm fine powder. 500 mg of each selected sample was dampened with 5-6 drops of binder made up of 10% styropore solution in toluene. Each sample was then carefully homogenized, dried and pressed into pellets of 100 mg/cm^2 for XRF analysis.

The ore dressing for the Sokoto phosphate rock was started with crushing, calcinations and flotation methodology. There are three solvent extraction methods of uranium from phosphate rock. They are (i) di(2-ethylhexyl) phosphoric acid and tri-octylphosphine oxide (DEPA-TOPO), (ii) Octyl pyrophosphoric acid and kerosene (OPPA), and (iii) mixture of mono-octyl phenyl phosphoric acid and di-octyl phenyl phosphoric acid (OPAP). The DEPA-TOPO method is preferred because it is the most cost-effective, chemically stable and exceptionally selective of uranium in hexavalent state (U^{6+}) coupled with its minimum solubility in phosphoric acid. Using the DEPA-TOPO method, < 30 wt. % P_2O_5 can be upgraded to concentration of 52-54 wt %.

4. Results and Discussions

Table 1 shows chemical composition of Sokoto and Ogun phosphates in comparison to deposits from other countries. Table 2 shows the chemical composition of Sokoto phosphate rock from ten different sample locations.

The five major types of phosphate resources in the world are marine phosphates, igneous phosphates, metamorphic phosphates, biogenic and phosphates from weathering [22]. Sedimentary marine phosphate rock deposits provide 75% of the world's phosphate resources, while 15-20 % come from igneous and weathered deposits and only 1-2% from biogenic (mainly from bird and bat guano accumulations) resources [22]. The Sokoto phosphate deposits are of the sedimentary marine origin. In Sokoto State, phosphates of Paleocene sedimentary deposits occur in the Dange Formation. The Dange Formation also contains gypsiferous shales and phosphate nodules [9,8]. The overlying Paleocene Kalambaina and Gamba Formations are dominated by limestones and laminated ('paper') shales. A horizon with phosphate pellets within the Gamba Formation [8], quoted by van Straaten [22], probably equivalent to the phosphate-containing marine sequence in neighboring Niger and Mali [24,5]. Exploration work by the Nigerian Geological Survey Agency also established the occurrence of phosphate nodules and pellets in Dange and Gamba Formations, respectively [15]. The thickness of phosphate deposits ranges from 1 to 5 m in the Dange Formation and the phosphatic nodules/pellets occur in sizes of 0.1-1 cm with varying concentration in different locations [3,18], quoted in Ogunleye [15]. The Sokoto phosphates have estimated reserves of 5-10 million tons [2].

Table 1. Chemical Composition of Sokoto Phosphate Rocks compared to other Phosphate Deposits (Source: [1]Akinrinde and Obigbesan [2]; [5]Ghoridashrath [4],Chemicalland21[25]; [2]NIPC [10]; [3]van Kauwenberg [21] in Okosun [16]

	SOKOTO	[1]SOKOTO	[2]SOKOTO	[3]SOKOTO	[4]OGUN	[5]OTHERS
P_2O_5	20.18-34.7	32.50	36.25	34.2	30.50	30-35
CaO	35.96-56.60	44.23	52.30	47.9	19.23	45-52
F	2.26-3.64	-	3.84	3.4	-	4
Cl	-	-	-	-	-	0.04-0.2
$CaCO_3$	-	79.0	-	-	34.30	-
MgO	0.10-0.99	0.95	-	0.10	1.35	0.5
Na_2O	0.15-0.95	-	-	0.24	-	0.2-0.8
K_2O	0.03-0.05	-	-	0.08	-	0.1-0.3
Fe_2O_3	0.22-3.57	3.19	1.50	3.00	7.28	1
Al_2O_3	0.43-6.24	1.79	1.50	1.70	6.91	2
SiO_2	2.7-9.71	4.20	3.44	4.2	6.68	5-8
H_2O	1.17-2.81	-	0.75	0.77	-	2.4
Solubility in 2% citric acid	-	45.55	-	35	38.42	-
Moisture %	-	-	-	-	-	1.5-2.5
Cd (mg Kg^{-1})	-	0.63	-	-	9.70	-

Table 2. Chemical composition of Sokoto phosphate rock samples (Source: [1]van Kauwenberg [21] in Okosun [16])

MAJOR ELEMENT	[1]R1	A1	A2	A3	A4	A5	A6	A7	A8	A9	A10
P_2O_5	34.20	34.70	29.70	29.66	29.23	29.04	28.85	23.16	25.92	23.39	20.18
CaO	47.9	47.70	56.60	53.45	55.36	53.29	52.04	49.98	49.54	42.20	35.96
F	3.40	3.64	2.60	2.44	2.84	2.68	2.72	3.03	2.83	2.63	2.26
Cl	-	-	-	-	-	-	-	-	-	-	-
$CaCO_3$	-	-	-	-	-	-	-	-	-	-	-
MgO	0.10	0.99	0.99	0.10	0.87	0.20	0.21	0.12	0.39	0.27	0.65
Na_2O	0.24	0.28	0.26	0.25	0.95	0.26	0.15	0.16	0.31	0.15	0.19
K_2O	0.08	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.05
Fe_2O_3	3.00	3.57	0.57	0.93	0.22	0.70	0.68	0.61	2.39	1.04	2.22
Al_2O_3	1.70	1.73	0.53	0.43	0.69	1.53	2.43	2.09	2.17	2.93	6.24
SiO_2	4.20	4.70	6.7	2.70	3.21	6.58	7.87	10.05	15.05	19.06	19.71
H_2O	0.77	1.26	1.22	1.72	0.77	0.82	1.05	1.21	1.17	1.44	2.81
L.O.I.	5.3	5.44	5.4	5.55	5.36	5.33	3.15	3.21	6.68	3.48	5.12
NAC P_2O_5	5.30	-	-	-	-	-	-	-	-	-	-
CaO/P_2O_5	1.40	1.38	1.91	1.80	1.89	1.84	1.80	2.16	1.91	1.80	1.78
F/P_2O_5	0.10	0.08	0.09	0.08	0.10	0.09	0.09	0.13	0.11	0.11	0.11

4.1. Geochemical Characterization of Sokoto Phosphate Rock

According to Akinrinde and Obigbesan [2], Sokoto phosphates have calculated average percentage of 20.18-36.25 wt. % P_2O_5, while the Ogun phosphates have 26.3-32.0 wt. % P_2O_5. These average values for the Nigerian phosphates are comparable with those of Togo phosphates which contain the values of 28.0-36.6 wt. % P_2O_5 [2]. From the data in Table 1, Nigerian phosphates have values of P_2O_5 that range from 20.18-36.25 wt. %, while the Jordan, Morocco, USA, Algeria, China and Syria phosphates have the range of 30-35 wt. %.

4.2. Ore Dressing Route for Sokoto Phosphate Rock

Phosphate rock (phosphorite) is a marine sedimentary rock which contains 18-40 wt. % P_2O_5, as well as some uranium and all its decay products, often 65 to 200 ppm U, and sometimes up to 800 ppm. The main mineral in the phosphate rock is apatite, and most commonly, fluorapatite - $Ca_5(PO_4)_3F$ or $Ca_{10}(PO_4)_6(F,OH)_2$. This is insoluble, so cannot directly be used as a fertilizer (unless in very acidic soils). It must therefore be processed. This is normally in a wet process phosphoric acid (WPA) plant where it is first dissolved in sulphuric acid. About 2-4 wt. % fluorine is usually present [14]. When phosphate rock is treated with sulphuric acid in sub-stoichiometric quantity, normal superphosphate is formed. If more sulphuric acid is added, a mixture of phosphoric acid and gypsum (calcium sulphate) called “phosphogypsum” is obtained.

After the gypsum is filtered out, the resultant phosphoric acid can be treated to recover uranium [23].

The basic reaction is:

$$Ca_3(PO_4)_2+3H_2SO_4+6H_2O \rightarrow 2H_3PO_4+3CaSO_4.2H_2O$$

(exothermic reaction)

The process used produces different varieties of calcium sulphate such as anhydrous: $CaSO_4$, hemihydrate: $CaSO_4.½H_2O$ or dihydrate: $CaSO_4.2H_2O$.

The phosphoric acid is treated with ammonia produced from natural gas (Haber process) to produce diammonium phosphate (DAP) as final product, which is adjusted to industry standard composition of 18-40-0 in N-P-K.

$$2NH_3+H_3PO_4 \rightarrow (NH_4)_2HPO_4$$

Phosphate rock is digested with sulphuric acid to produce a phosphoric acid solution (called wet process phosphoric acid) and an insoluble calcium sulphate (gypsum). Sokoto phosphate rock contains significant quantities of uranium around 65 ppm [15]. Attempts to recover uranium from wet-process phosphoric acid have centered on the use of solvent extraction processes in which the uranium is transferred to an organic phase, stripped from the organic phase and subsequently recovered as a uranium precipitate. The uranium-free wet-process phosphoric acid is then processed conventionally to form various phosphate-containing fertilizer products [14]. Uranium from phosphate extraction using a mixture of di(2-ethylhexyl) phosphoric acid (DEPA) and tri-octylphosphine oxide (TOPO) is dissolved in an organic diluent. This solvent extraction mixture have a high affinity for uranium in the hexavalent oxidation state (U^{6+}) achieved by using H_2O_4 oxidation agent [14].

Uranium extracted as yellow cake (U_3O_8) and the phosphoric acid free of uranium is recovered for fertilizer production and allied industrial applications. The gypsum is also of economic importance. Many countries such as USA, Canada, Belgium, Syria, Israel, Taiwan, Egypt, Morocco, etc. have active process plants for the extraction of uranium from phosphates. The yellow cake is used as nuclear fuel for electricity production. Using DEPA-TOPO method, 52-54 wt. % P_2O_5 can be obtained from phosphogypsum containing 25-28 wt. % P_2O_5. In addition, 0.32 mg of uranium oxide (yellow cake, U_3O_8) and about 27 wt. % of gypsum can be recovered from one gram of P_2O_5.

Nigeria has some fertilizer production plants such as the Superphosphate Fertilizer Company located in Kaduna, fertilizer production at Crystal Talc Limited at Kagara, Fertilizer Concentrate Company, Edo State and the National Fertilizer Company of Nigeria (NAFCON), Port Harcourt. Presently, there is no uranium from phosphates extraction plant in Nigeria. Figure 2 shows set of photographs of the morphologies of the phosphatic noodles and the phosphate rock outcrop. On the other hand, Figure 3 shows the ore dressing process flowchart for the extraction of uranium from the Sokoto phosphate rock.

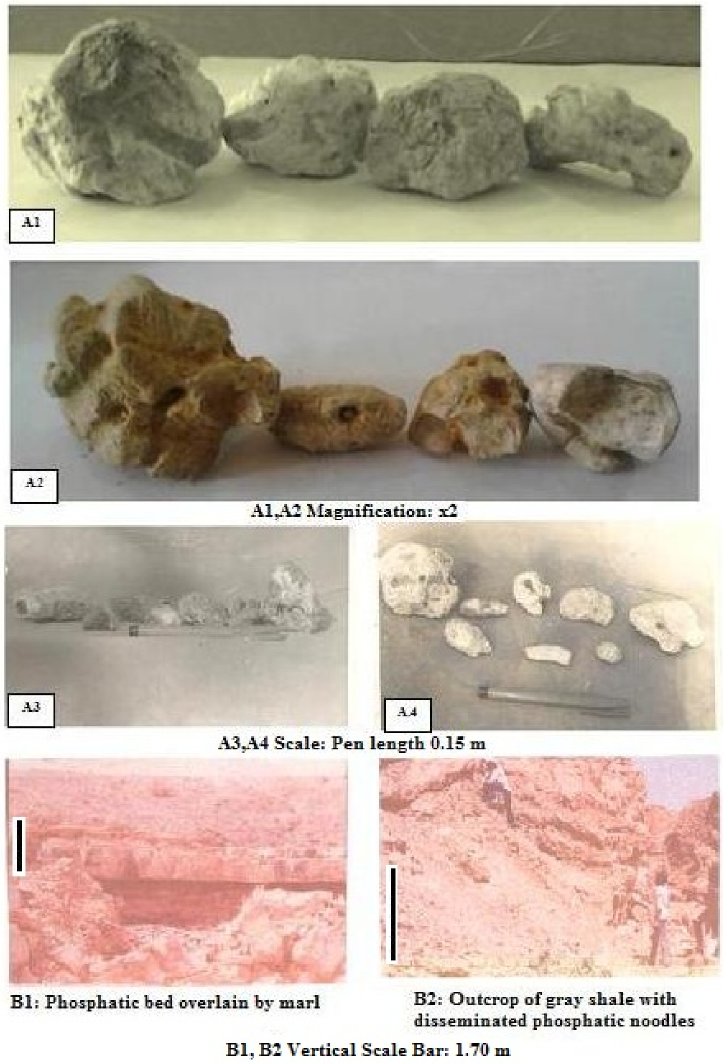

Figure 2. Photographs of the morphologies of the phosphatic noodles (A1-A4) and Phosphate Rock outcrop (B1,B2)

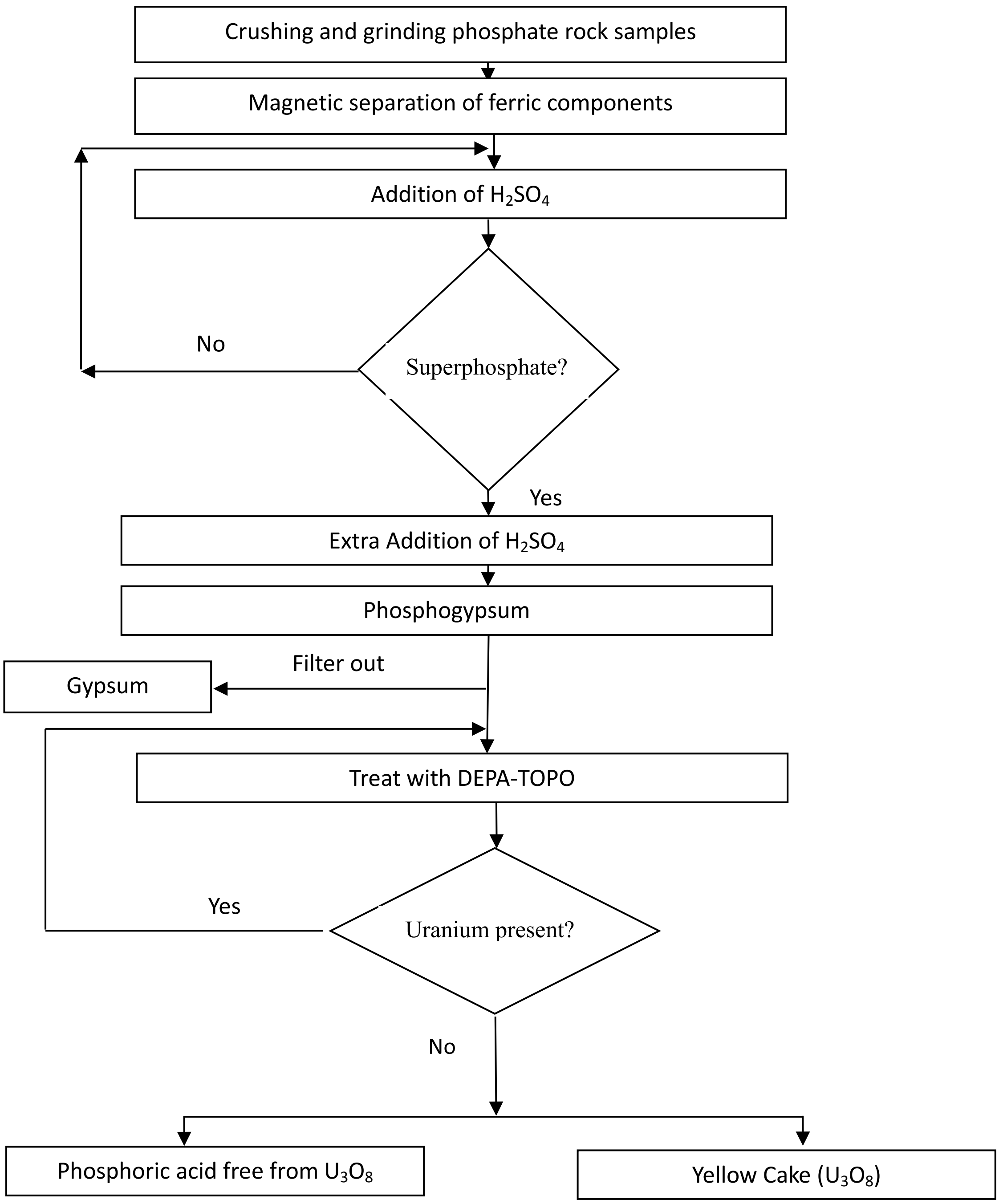

Figure 3. Ore dressing process flowchart for Sokoto phosphate rock

6. Conclusions

The geochemical characterization of Sokoto phosphate rock has been presented. An ore dressing process has been proposed for the efficient recovery of the phosphate as fertilizer, allied applications and for the extraction of uranium and gypsum from the source mineral. The phosphate rock occurs mainly in nodules, while some are found as pellets, especially in the southern part of the study area. The phosphate deposit occurs predominantly in the Dange Formation and a small occurrence in the Gamba Formation. The phosphatic nodular beds measure between 0.3 m and 0.1 m, while the nodular disseminations may be up to 6 m in thickness. The geochemical characterization of the phosphate rock indicated that P_2O_5 values range from 20.18-36.25 wt. %, while the Jordan, Morocco, USA, Algeria, China and Syria phosphates have the range of 30-35 wt. %. There are three solvent extraction methods of uranium from phosphate rock. They are (i) di(2-ethylhexyl) phosphoric acid and tri-octylphosphine oxide (DEPA-TOPO), (ii) Octyl pyrophosphoric acid and kerosene (OPPA), and (iii) mixture of mono-octyl phenyl phosphoric acid and di-octyl phenyl phosphoric acid (OPAP). The DEPA-TOPO method is preferred because it is the most cost-effective, chemically stable and exceptionally selective of uranium in hexavalent state coupled with its minimum solubility in phosphoric acid. Using the DEPA-TOPO method, < 30 wt. % P_2O_5 can be upgraded to concentration of 52-54 wt %. 0.32 mg of uranium oxide (yellow cake, U_3O_8) and about 27 wt. % of gypsum can be recovered from one gram of P_2O_5. The recovery of gypsum from the Sokoto phosphate rock will boost its reserve in Nigeria.

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