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Manufacture of simple superphosphate from Tahoua rock phosphate

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Abstract

In this work, we used the rock phosphate Tahoua sample. It underwent several processing steps before obtaining the raw phosphate powder. However it presents a weak solubility and this weakness is due to the presence of metal oxides in the phosphate. Indeed, rock phosphate is the raw material of the fertilizer industry and it is rarely used directly as is, because of its low assimilability by plants. To solve this problem, we have transformed merchantable phosphate into phosphate fertilizers directly and easily assimilated. The objective of this work is to manufacture a simple superphosphate fertilizer (SSP) and study its solubility in perchloric acid and water. We manufactured a simple superphosphate based on crushed and ground Tahoua merchant phosphate. The studied granulometries are: (inferior to 63µm; 63µm; 100µm; 160µm). The attack was made with sulfuric acid (H_2SO_4) with a variation of temperatures (40°C; 60°C and 80°C) in order to determine the effects of the granulometrys and temperatures. The results obtained for granulometries (inferior to 63µm; 63µm; 100µm; 160µm) with ripening times (5th day, 12 days) and at these different temperatures are respectively 40°C (15.79%; 14.77%; 17.35%; 15.93%); 60°C (18.58% ; 8.32%; 8.32%; 12.67%); 80°C (16.40%; 19.73%; 16.54%; 21.56%) in the perchloric acid solution and 40°C (5.74%; 8.32%; 8.32%; 12.67%); 60°C (7.64%; 8.32%; 8.73%; 7.91%); 80°C (11.86%; 12.67%; 11.04%; 6.55%) in water These results allowed us to conclude that Tahoua rock phosphate is suitable for the manufacture of simple superphosphate fertilizer at 80°C (total P_2O_5 equals 21.54%) and at 60°C (and a soluble P_2O_5 equal to 12.87%) for a particle size of 160µm.

Keywords: Manufacturing; Simple superphosphate; Phosphate; Natural

1. Introduction

Phosphorus is a plant nutrient. Plants grow well when the soil contains more of the nutrient and give high yields. On the other hand, if the soil is deficient in one of the elements essential to plants, development and growth are limited and yields are reduced. To have a good yield, we can provide the crops with the elements they need. The phosphorus absorbed by the plants must be in soluble form in the soil [1]. Fertilizers can often be applied to the soil to give satisfactory yields. Phosphate fertilizers have been manufactured using Tahoua rock phosphate (TNP) as a raw material. These fertilizers are obtained by the attack of phosphates by acids. This is a leaching reaction between an ore and a solution that takes place essentially on the surface of the solid particle [1]. It is a property giving the most information on their agronomic efficiency since it conditions the availability of ortho phosphate ions ($H_2PO_4^-$ and HPO_4^{2-}) to plants [2]. The phosphate natural of Thaoua is a sedimentary phosphate of nodular type which is constituted mostly by fluorapatite and has an average P_2O_5 content of about 30%, but it contains impurities whose presence influences the quality of this product [3]. These impurities are the basis for the insolubility of PNT in water. We have carried out a particle size analysis and the production of superphosphate from Tahoua rock phosphate at several temperatures.

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2. Materials and Methods

2.1. Material used

2.1.1. Ore

Phosphate rock was collected from the Tahoua rock phosphate deposit in old drill holes drilled in the 1980s by the Geological and Mining Research Bureau (BRGM) of Niger in the form of nodules with an average content of about 30% P_2O_5 [4].The phosphate rock undergoes a mechanical treatment including crushing, grinding, screening and sieving, to reduce the dimensions to 15mm. The aim is to separate the phosphate material from the impurities consisting mainly of clays. The chemical analysis of Tahoua rock phosphate allowed us to determine its chemical composition. These results show that Tahoua rock phosphate has low heavy metal content (lead, chromium and cadmium). This confirms a guarantee of quality of the product on the environmental and industrial level for its production [5]. In this case, the general chemical formula of phosphate proposed: Ca_{10-x} (K, Na, Mg)$_x(PO_4)_{6-y}$ (CO_3F, SO_3, SiO_4) $_yF_{2-z}(OH)_z$. x, y and z represent successively the degree of molar substitution, of Ca by K, Na, Sr, Mg...; of PO_4 by CO_3F, SO_3, SiO_4 and of F by OH. The chemical composition of roc k phosphate Tahoua.

Table 1 Chemical composition of Tahoua rock phosphate [4]

Major Elements	%	Trace Elements	Ppm
CaO	49.66	Ti	570.14
P_2O_5	27.98	Nd	486.14
Fe_2O_3	7.15	Sr	430.5
SiO_2	7.80	Zn	160.11
Al_2O_3	2.42	As	57.22
Na_2O	0.53	Cu	22.18
MgO	0.17	Ni	49.6
K_2O	0.13	U	32.17
Y_2O_3	0.15	Ba	20.52
MnO	0.11	Cr	19.52
		Pb	7.96
		Cd	3.9

2.1.2. Chemicals

The different chemicals used during this research are presented in table 2

Table 2 Characterization of the chemicals used

Products	Mass molaire [g. mol^{-1}]	Density [g.cm^{-3}]	Purety [%]	Origin
Nitric acid	63	1.51		Normapur
Sulfuric acid	98	1.83	98	Damaoren t
Perchloric acid	100.46	1.61	65	Prolabo
Mono vanadate d'ammonium	116.98	2.33	99	Acros organics
Sodium hydrogen phosphate dihydrate	177.90	0.85	98	Proanalys

2.1.3. Equipment

The equipment used during this work are:

- A molecular absorption spectrophotometer;
- An analytical balance;
- A semi-manual stirrer;
- Water bath;
- Some laboratory equipment;
- A filter paper ;
- A pH meter;
- An x-ray fluorescence spectrometer;

2.2. Methods

2.2.1. Preparation of simple superphosphate

After crushing, grinding and sieving, the phosphate was classified into three particle sizes: <63; 63; 100; 160 µm. In each particle size, 10 g was weighed into a 250 mL beaker using a Precisa 205 A type precision balance. This mass is attacked in a stoichiometric amount by a sulfuric acid solution. The mixtures are placed in a water bath at 40°C; 60°C and 80°C under stirring for 30 minutes. Then the obtained sludge is left to dry in the open air for 5 to 12 days (ripening time).

2.2.2. Preparation of P_2O_5 water soluble (P_2O_5 WS)

In a 250 mL beaker well rinsed with distilled water, 0.25g of the single superphosphate sample and 100 mL of distilled water are introduced. The mixture is kept under stirring with the magnetic stirrer at room temperature. The stirring speed is set to 500 rpm for a 20 minute stirring time. At the end of the stirring, each mixture is then filtered on a fast paper by collecting in a clean and dry plastic bottle (discard the first milliliters of the filtered solution). In order to evaluate the action of water in the simple superphosphate, a determination of the dissolved phosphoric anhydride (P_2O_5) level was performed on each filtrate. To ensure reproducibility of results, these measurements were performed three times.

2.2.3. Preparation of P_2O_5 total (P_2O_5 T)

A mass of 0.50 g of the SSP fertilizer weighed with a precision balance (type Précisa 205 A) is introduced into a 200 mL beaker previously dried. A few mL of distilled water is poured in and 7.5 mL of perchloric acid (PA) is added. The mixture is heated until all white coloration disappears on a plate. Then let it cool, transfer quantitatively into a 250 mL volumetric flask by carefully rinsing the beaker with distilled water and then homogenize, adjust the mixture to the mark. It was filtered on a pleated filter paper, discarding the first mL of the filtrate in a dry flask. The filtrate is ready for analysis. Finally, a determination of the dissolved phosphoric anhydride (P_2O_5) content was performed on the filtrate [1].

The dissolution reaction of phosphate by perchloric acid takes place in 3 steps:

$$Ca_3(PO_4)_2 + 2HClO_4 \rightarrow Ca(ClO_4)_2 + 2CaHPO_4 \quad (1)$$

$$3Ca^{2+} + 2PO_4^{3-} + 2H^+ + 2ClO_4^- \rightarrow 2Ca^{2+} + 2ClO_4^- + 2Ca^{2+} + HPO_4^{2-} \quad (2)$$

$$PO_4^{3-} + H^+ \rightarrow HPO_4^{2-} \quad (3)$$

2.2.4. Complexometric determination of P_2O_5

The complexation of dissolved phosphate ions during the attack was carried out using a complexing solution. It is a solution that consists of carefully preparing three different solutions in equal proportions and mixes them in order. Then we mixed them in order gently [1]. The solutions are prepared as follows:

- Solution 1: a solution of 250mL was prepared including one volume of nitric acid and two volumes of distilled water;
- Solution 2: We dissolved 0.625 g of ammonium mono vanadate NH_4VO_3 in a 250 mL volumetric flask with 150mL of boiling distilled water. After simple stirring, add 5mL of nitric acid HNO_3 (d =1.38) and make up to the mark with distilled water, allowing to cool;

- Solution 3: Dissolve 12.5 g of ammonium molybdate tetrahydrate Mo_7O_{24} $(NH_4)_6$ $4H_2O$ in 200 mL of boiling water and make up to 250 mL with distilled water.

2.2.5. Determination of dissolved phosphorus

The determination of phosphorus was determined by colorimetry at the absorption wavelength of the complex λ=430nm. The reaction is based on the formation of phosphovanadomolybdic complex and the reduction of a complex of ortho phosphoric acid and vanadomolybdic reagent. The reduction of the phosphovanadate complex is accompanied by a yellow coloration whose intensity is proportional to the amount of phosphorus present in the sample considered. The spectrophotometer used is the Evolution 300 type. The standard solutions and the solutions of the samples are studied under the same conditions to ensure the certainty of the results.

3. Results and Discussion

3.1. Manufacture of simple superphosphate

The manufacture of simple superphosphate by sulfuric acid on Tahoua rock phosphate (fluorapatite) was carried out. This process is a heterogeneous mixture with a few steps that takes place mainly in the diffusion domain. During this process, we noticed a rapid decomposition reaction that takes place on the surface of the phosphate particles until the total consumption of the sulfuric acid and the phosphoric acid formed during the reaction reacts with the remaining phosphates. But also, the natural phosphate is constituted of impurities whose reaction with the sulfuric acid causes secondary reactions. The work of Boulh, H, (2010) showed that the manufacture of SSP fertilizer is a heterogeneous multi-step process that takes place in the diffusion domain. The reactions are presented below:

$$Ca_5F(PO_4)_3 + 5H_2SO_4 + 5H_2O \rightarrow 5(Ca\,SO_4\,H_2O) + 3H_3PO_4 + HF \quad (4)$$

$$Ca_5F(PO_4)_3 + 7H_3PO_4 + 5H_2O \rightarrow 5[Ca(H_2PO_4)_2.H_2O] + HF \quad (5)$$

The sum of the two reactions will give the global equation:

$$2Ca_5F(PO_4)_3 + 7H_2SO_4 + 3H_2O \rightarrow 7(Ca\,SO_4) + 3Ca(H_2PO_4)_2.H_2O + 2HF \quad (6)$$

The secondary reactions would be between the metal oxides and sulfuric acid. Each sample obtained after this acid attack is left to dry in the open air for 12 days (ripening phase). At the end of 5 days and 12 days, we measured the amount of total P_2O_5 and water soluble P_2O_5.

3.2. Solubility of SSP obtained at 40°C in perchloric acid (PA) and WS after 20 minutes of agitation

The dissolution of simple superphosphate by perchloric acid (PA) and distilled water was carried out according to the granulometric slices and the wetting times. The results obtained are presented in the figures (Figure 1 and 2). They present the rate of total P_2O_5 and water soluble P_2O_5 in the simple superphosphate fertilizer ($Ca(H_2PO_4)_2$).

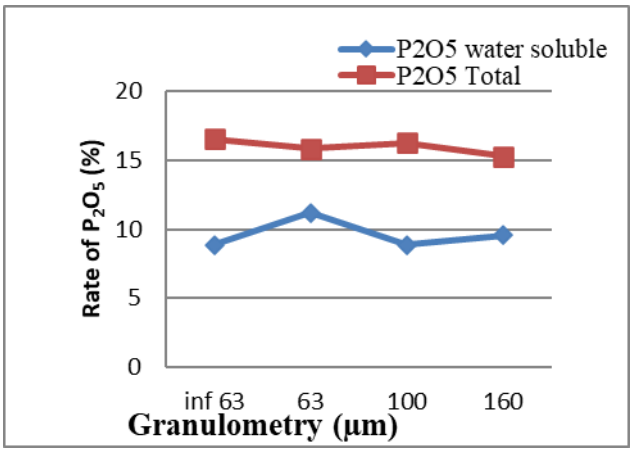

Figure 1 Variation of the P_2O_5 rate according to the granulometry (SSP Tahoua: T = 40 °C and residence time = 5 days).

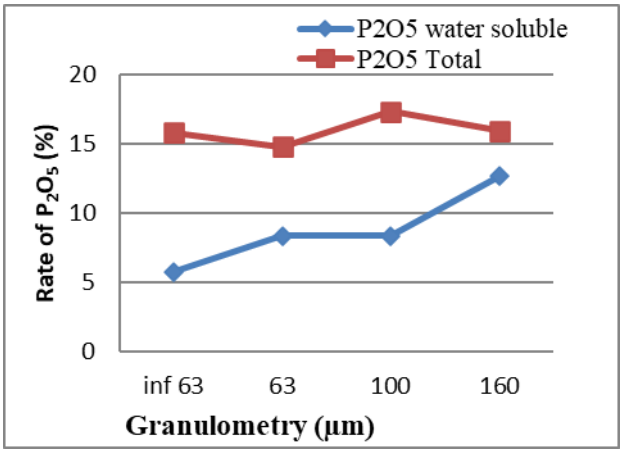

Figure 2 Variation of the P_2O_5 rate according to the granulometry (SSP Tahoua; T=40°C and residence time = 12 days)

The analysis of these results obtained shows that the highest rate of total P_2O_5 is obtained at the granulometry <63µm (Figure n°1) and 100 µm (Figure2). These rates of total P_2O_5 16.54%, 17.35% are respectively in the ripening times of 5th day and 12th day. This shows that the P2O5 content is higher after 12 days of ripening time and at a particle size of 100 µm. Then the water soluble P_2O_5 rate can reach up to 11.17 % for a curing time of 5 days and a granulometry of 63 µm while for 12 days stay time of 160 µm granulometry is 12.67 %. The total phosphate content and the soluble P_2O_5 content are higher in the 12th day of ripening. This increase could be related to the effect of the specific surface of phosphate particles. In this sense, the results we obtained are in agreement with those of BOULAHBEL, Hani [1].

3.3. Solubility of SSP obtained at 60°C in AP and WS after 20 minutes of stirring:-

The determination of phosphoric anhydride in the filtrates resulting from the attack of simple superphosphate manufactured at 60°C by perchloric acid and distilled water allowed us to obtain the results reported in the figures (Figure 3 and 4).

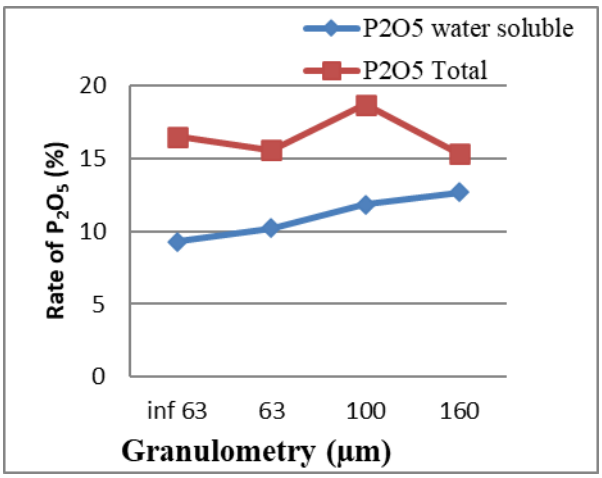

Figure 3 Variation of the P_2O_5 rate according to the granulometry (SSP Tahoua: T = 60 °C and residence time = 5 days).

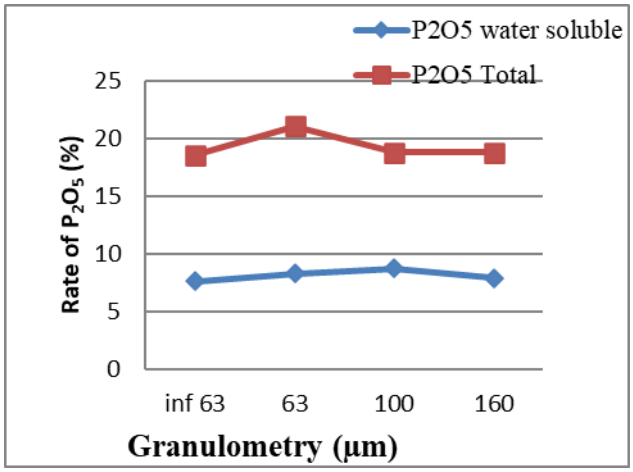

Figure 4 Variation of the P_2O_5 rate according to the granulometry (SSP Tahoua; T=60°C and residence time = 12 days)

Experiments with the dissolution of simple superphosphate prepared at 60°C, show that the total P_2O_5 content is the highest when the granulometry is 63µm in the twelfth day of the slurry stay. Then the dissolution of simple superphosphate in distilled water gives better results at the temperature of 60°C and at the granulometry 160µm. This rate increases progressively to a value of 12.87% at the twelfth day of sample ripening. These high values of dissolved P_2O_5 could be explained by the effect of specific surface of phosphate particles; the effect of temperature and the consumption of hydronium ions in the medium. In this sense, authors [3; 4; 5; 6] have shown that the increase of dissolved P_2O_5 rate is related to the important consumption of H_3O^+ ions, the effect of specific surface of phosphate and the effect of temperature. This is in accordance with the results found by several works [6; 10], which are more important in the granulometry between 100 to 160µm. Therefore, when manufacturing simple superphosphate, it is important to use phosphate with a granulometry of 63 to 160 µm.

3.4. Solubility of SSP obtained at 80°C in PA and WS after 20 minutes stirring:-

The dissolution of single superphosphate by perchloric acid and distilled water was performed. The results obtained are presented in Figure5 and Figure 6. They show the rate of P_2O_5 dissolved in the simple superphosphate fertilizer.

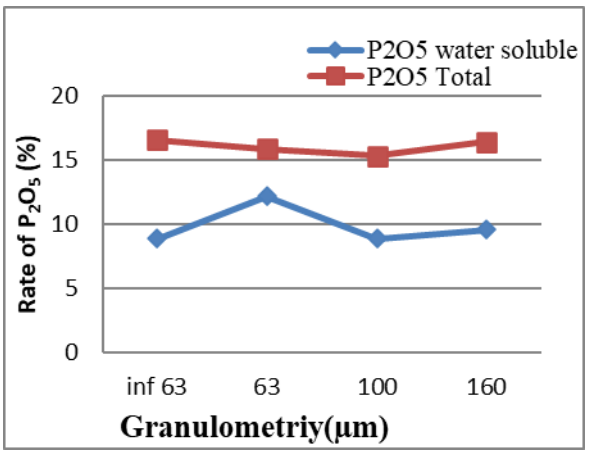

Figure 5 Variation of the P_2O_5 rate according to the granulometry (SSP Tahoua: T = 80 °C and residence time = 5 days).

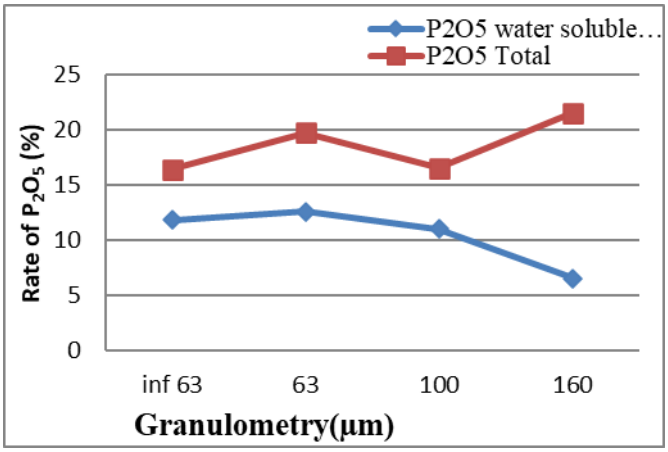

Figure 6 Variation of the P_2O_5 rate according to the granulometry (SSP Tahoua; T=80°C and residence time = 12 days)

The results obtained, after the dissolution of simple superphosphate by perchloric acid and soluble water at 80°C, revealed that the rate of total P_2O_5 is higher at the 160µm granulometry and at the 12 days of ripening time. The dissolution of SSP (21.56%) is the best P_2O_5 rate at 80°C (Figure 6). This value obtained is higher than the results obtained [10] for the dissolution of simple superphosphates (Kaf Essnoune phosphate; Blad Elhadba phosphate and Djmidjma phosphate). Similarly, the rate of P_2O_5 soluble water (12.17%) is higher on the fifth day of ripening of the slurry with the granulometry 63µm (Figure 5). This could be due to the effect of the specific surface of ores and the release of the P_2O_5 molecule by the undesirable elements on the one hand and on the other hand could be related to the attack of H_3O^+ ions in the medium. Researchers have shown that the dissolved P_2O_5 rate is related to the effect of the specific surface area of ores and the release of the P_2O_5 molecule [5; 6]. The low value of water soluble P_2O_5 rate of SSP fertilizer could be due to the presence of impurities (iron and aluminum).

3.5. Solubility of SSP obtained at 40°C, 60°C, 80°C in water after 20 minutes agitation:-

The dissolution of single superphosphate manufactured at these three temperatures in water was performed. The results obtained are shown in Figure 7 and Figure 8. They show the rate of water soluble P_2O_5.

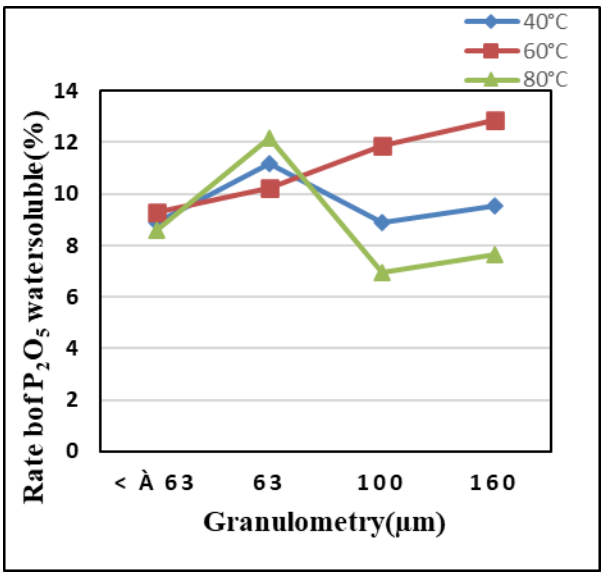

Figure 7 Variation of the P_2O_5 water soluble rate to according the granulometry (SSP Tahoua: residence time = 5 days)

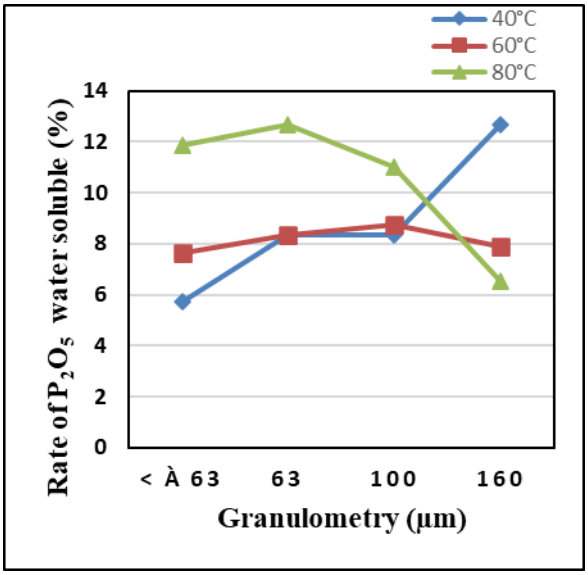

Figure 8 Variation of the P_2O_5 water soluble rate according to the granulometry (SSP Tahoua; residence time = 12 days)

The results of the analyses obtained show that the rate of P_2O_5 soluble water in the simple super phosphate is not stable according to the granulometry (Figure 7 and Figure 8). We note from these last two figures, the rate of P_2O_5 soluble water is high at the fifth day of the ripening time and at a temperature of 60°C. In this medium, the solubility is also higher in the ripening time of five days and at the temperature of 60°C. This result can be explained by the effect of the specific surface, the attack of H^+ protons and the effect of the temperature of this medium. Therefore, there is a consumption of H_3O^+ ions in this medium [6; 7]. This can be said, the greater the temperature the greater the dissolution. B, Hani [1] proved that the solubility of simple superphosphate of natural phosphate of djmidjma by soluble water is between 12 to 14% at a temperature of 60°C and even at an attack time of 20 minutes. Thus the water soluble P_2O_5 content of simple rock phosphate superphosphate from Tahoua is lower than that of rock phosphate superphosphate from several deposits in some country in the 160µm particle size range and in all the ripening periods of the simple superphosphate slurry.

3.6. Solubility of SSP obtained at 40°C, 60°C, 80°C in perchloric acid after 20 minutes agitation

The dissolution of simple superphosphate made at these three temperatures by perchloric acid was carried out. The results obtained are presented in Figure 9 and Figure 10. They show the total P_2O_5 content.

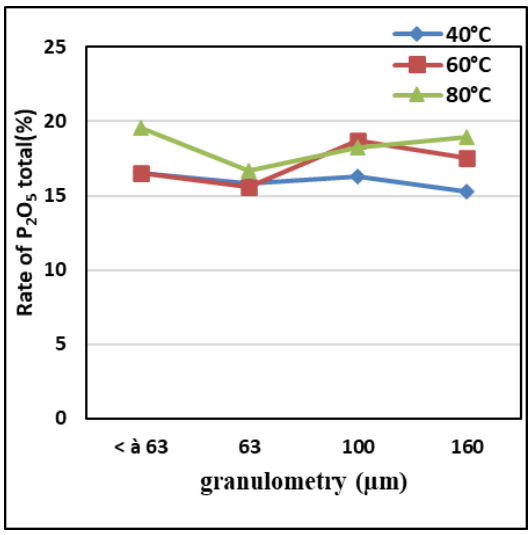

Figure 9 Variation of the P_2O_5 total rate according the granulometry (SSP Tahoua: residence time= 5 days).

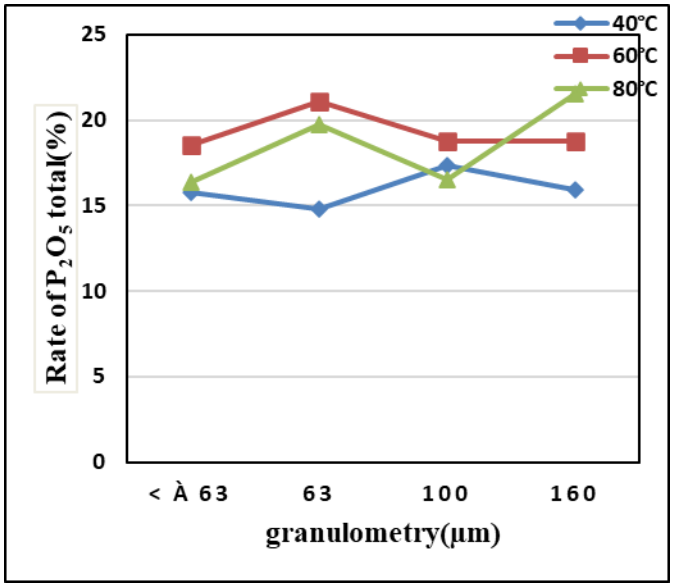

Figure 10 Variation of the P_2O_5 total rate according to the granulometry (SSP Tahoua residence time= 12days)

The analyses of the results obtained of dissolved P_2O_5 rate according to different granulometrys show that the P_2O_5 rate is more important in the twelfth day of ripening time of the simple superphosphate slurry (15 to 21.54%) than in the fifth day (15 to 19.59%) of all temperatures. From the latter figures we see that the total dissolved P_2O_5 content is higher in the twelfth day of ripening time of phosphate fertilizer at temperature 80°C. This result could be due to an increase of the specific surface of particles caused by the thermal effect. Thus the production of simple superphosphate fertilizer can be proportional to the temperature. This results in an important consumption of H_3O^+ ions in the medium by increasing the dissolution of ores [8; 9; 10]. The rate of solubilization that we obtained after the simple super phosphate made with Tahoua rock phosphate is more important than those obtained [1] for the dissolution of simple super phosphate Blad Elhadba; of simple super phosphate of Kaf Essnoune and of super phosphate of Djmidjma

4. Conclusion

The manufacture of simple superphosphate was carried out according to the granulometric slices and the three different temperatures. The samples were left in the open air for the ripening times. The results obtained show that it is possible to produce ssp based on Tahoua rock phosphate, although it contains impurities that slow down the dissolution. These results show that Tahoua rock phosphate is suitable for the manufacture of simple superphosphate fertilizer, because it gives a total P_2O_5 rate that could reach up to 21.54% P_2O_5 rate at 80°C and a water soluble P_2O_5 rate of 12.87% at 60°C in the 160µm granulometry The dissolved P_2O_5 rate is more important at the granulometry between 100 and 160µm and at the temperature between 60°C and 80°C. The rate of water soluble P_2O_5 is the highest at a small ripening time and the rate of total P_2O_5 is the highest at a large residence time. This research work that we have carried out on natural phosphates has allowed us to say that the natural phosphates of Tahoua, compared to other phosphates in the world, are not studied in a more thorough manner.

Compliance with ethical standards

Disclosure of conflict of interest

The authors declare that they have no competing interests.

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