

## Solubility Test in Some Phosphate Rocks and their Potential for Direct Application in Soil

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**Abstract:** There are various methods to evaluating phosphate rock for direct application. The first approach is solubility test of Phosphate Rocks (PR) using chemical extractant. In this study to compare the effectiveness of direct application of PRs, two samples of a sedimentary unbeneficiated PR from Lar mine of Yasooj and two samples of concentrated PR of Asfordi igneous PR of Yazd in Iran along with one sample PR of Gafsa (Tunisia) were analyzed for the main nutrients and minerals with XRF and XRD. Calcite and quartz were the main ingredients of Yasooj PR which cause problems for direct application. Yazd PR in addition to apatite had some iron and magnesium minerals which are also undesirable for fertilizer producing. The reactivity of these PRs was determined using the common extractants (Formic Acid, Citric Acid and Neutral Ammonium Citrate) and classified on the basis of International Fertilizer Development Center (IFDC) proposed classification method for direct application. The reactivity of Iranian PRs was quite low compare to Gafsa PR. The long-term dissolution kinetics of these PRs were determined with Formic Acid (FA) and Citric Acid (CA) showed slow reaction initially (0-20 min.) because of the presence of free calcium carbonate but later on the reaction rate increased surpassing Gafsa PR. Therefore Yasooj PR could be recommended to use in soil after beneficiation and removal of lime. The kinetics of dissolution of Yazd PRs followed a zero order equation.

**Key words:** Phosphate rock . Solubility test . Direct application . Dissolution kinetic . Soil

### INTRODUCTION

Phosphorus (P) is an essential nutrient for plants and animals. PR is the primary raw material for producing P fertilizers. Manufactured Water-soluble Phosphorus (WSP) fertilizers such as superphosphates are commonly recommended for correcting P deficiencies. However, most developing countries import these fertilizers, which are often in limited supply and represent a major outlay for resource-poor farmers. In addition, intensification of agricultural production in these regions necessitates the addition of P inputs not only to increase crop production but also to improve soil P status in order to avoid further soil degradation. Therefore, it is imperative to explore alternative P inputs. In this context, under certain soil and climate conditions, the direct application of PRs is an agronomic and economically sound alternative to the more expensive superphosphates in the tropics [1, 2, 3, 4, 5]. PR deposit occur worldwide but few are mined (for use mainly as raw materials to manufacture WSP fertilizer) [3].

A lot of PR mines have been discovered in Iran including ore at Yasooj, Lar Mountain. This mine belongs to Paleocene, Eocene and Oligocene eras. The age of PR layers goes to the upper Eocene. The deposits of Lar mountain PRs are estimated to be 210 million tons with 10.5 % P<sub>2</sub>O<sub>5</sub> [6].

There is another igneous iron and phosphate mineral deposit in phosphate mine of Yazd, Asfordi contain 17 million tons of PR with 13.9 % P<sub>2</sub>O<sub>5</sub> which is being mine and its P content after beneficiation is 34 to 39 % P<sub>2</sub>O<sub>5</sub> [7].

There are various methods for evaluating PRs for direct application [1, 8, 9, 10, 11]. The first approach is solubility test of PRs using chemical extractant. solubility test with using chemical extractant offer a simple and rapid method for classifying and then selecting PRs according to their potential effectiveness. The most common solutions are Neutral Ammonium Citrate (NAC), Citric Acid (CA) and Formic Acid (FA). The solubility data for these conventional reagents differ depending on the strength of the extractants. However, they are closely related and rank the PRs in the same relative order [1, 3, 10].

Table 1: Proposed classification of PR for direct application by solubility and expected initial response [12]

Rock potential	Solubility (% P <sub>2</sub> O <sub>5</sub> )		
	NAC	CA	FA
High	>5.4	>9.4	>13.0
Medium	3.2-4.5	6.7-8.4	7.0-10.8
Low	<2.7	<6.0	<5.8

Table 2: Ranking system for some South American PRs by solubility and RAE [4]

Soluble P <sub>2</sub> O <sub>5</sub> in NAC (% P <sub>2</sub> O <sub>5</sub> )	RAE <sup>1</sup> (%)	Solubility ranking
>5.9	>90	High
3.4-5.9	90-70	Medium
1.1-3.4	70-30	Low
<1.1	<30	Very low

<sup>1</sup>RAE = [(yield of ground PR)-(yield of check)]/[(yield of TSP)-(yield of check)] x 100

There is no widely accepted simple system for classifying and grading PRs for direct application according to solubility measurements.

Diamond [12] proposed a threefold classification system (low, medium and high reactivity) according to NAC, CA and FA solubility (Table 1). The system was based on IFDC data for the relative effectiveness of extraction media and the results of a wide variety of laboratory experiments and field trials.

Hammond and Leon [4] proposed a system with four solubility rankings (high, medium, low and very low) based on Relative Agronomic Effectiveness (RAE) and NAC soluble B<sub>2</sub>O<sub>5</sub> (Table 2). This study consisted of a relative comparison of PRs from several sedimentary, igneous and metamorphic sources based on a greenhouse experiment using *Panicum maximum* grass grown in an acid soil. These and additional data were analyzed further by statistical methods to show that the low and very low classifications were composed of sedimentary francolites with very low carbonate substitution and igneous PRs [13].

PR reactivity scales serve not only to compare several PR sources but also to predict their potential agronomic effectiveness. For example, the NAC solubility of PRs correlated well with grain yield of flooded rice in Thailand [9] and dry-matter yield of *Guinea* grass on an Oxisol from Colombia [14]. However, several studies have found FA to be the best indicator of crop response [1, 10, 11].

Common standard solubility tests are only qualitative in nature. This is because they use procedures that limit the dissolution of PR either by

the short reaction time or by the small PR sample/extractant solution ratio. Therefore, these conventional tests focus on the estimation of the short-term efficiency of PRs[3].

Truong and Fayard [15] have proposed an additional simple procedure for measuring the kinetics of PR dissolution over time. The advantage of this procedure is that it removes all the dissolved P and Ca. Moreover, there is no accumulation on the surface of particles preventing further PR dissolution. The extracted solutions can be analyzed at selected time intervals to draw a dissolution curve with time [15].

The above procedure is more time consuming than a single extraction. However, it is not more demanding than sequential or successive extractions and it provides more information on PR dissolution over time [3].

Transport-controlled dissolution reaction or those controlled by mass transfer or diffusion can be described using a parabolic rate law. But if the surface reactions are slow compared to the transport reaction, dissolution is surface-controlled. In surface-controlled reactions the concentration of solutes next to the surface are equal to the bulk solution concentrations and the dissolution kinetics are zero-order if steady-state conditions are operational on the surface. Thus, the dissolution rate,  $r$  is:

$$r = dC/dt = kA$$

and  $r$  is proportional to the surface area ( $A$ ) of the mineral. Thus, for a surface-controlled reaction the relationship between time and concentration ( $C$ ) should be linear [16]:

$$C = C_0 + kt$$

The objectives of this study are to compare short-term and long-term dissolution characteristics of Yasooj and Yazd PRs with those of Gafsa PR and determine the potential use of them for direct application in soil.

In this experiment both methods described above would be used to compare local PRs with reference Gafsa PR. Gafsa PR (Tunisia) was utilized as a reference PR for direct application because of its high reactivity [1, 17, 18, 19, 20, 21, 22, 23].

## MATERIALS AND METHODS

Two samples of sedimentary PR from Yasooj Lar mine and two samples of igneous PR from Yazd Asfordi mine and one sample of Gafsa PR were ground and passed through 100 mesh sieve (smaller than 149  $\mu$ m). The main ingredients in those samples were identified with an XRF model BRUKER (S4) and the

main peaks of the minerals were identified with BRUKER (D8) of an XRD instrument. Then, the main minerals were identified on the basis of ASTM charts and relative abundance of the components.

In order to determine the dissolution rate in NAC solution [24], one gram of PR was added to 100 mL of NAC and shaken for one hour at 65°C and in order to measure the amount of dissolved P in 2% FA and 2% CA, one gram of samples were added to 100 mL of this solution and shaken for one hour at room temperature and the suspension was immediately filtered. Also the long term dissolution of these PRs was determined using Troung and Fayard methods [15]. In this study one gram of PRs was placed in a funnel that contained filter paper then a 2% FA solution was dripped in to the funnel at a rate of about 1 drop/s. The exact volume of the filtrate was collected at given time (20, 40, 60, 100, 140, 180, 240, 300, 360, 420, 480, 540 and 600 minutes). The amounts of P in these solutions were determined with colorimetric method [25]. Likewise the rate of dissolution of the three PR samples (Yasooj 1, Yazd 2 and Gafsa) in 2% CA was determined and the amount of P in the collected solutions was determined with an ICP (Perkin Elmer 2000) instrument.

## RESULTS AND DISCUSSION

**The composition of PRs:** The concentrations of elements contained in PRs were found with XRF instrument is given in Table 3.

The Fig. 1-5 show the peaks related to the main compounds of PR that are obtained with XRD.

PRs generally contain endogangue and exogangue materials, such as calcite, dolomite, gypsum, as well as quartz, iron and aluminium oxides and clays [26] which were present in our PRs as well.

Table 3: [0]The concentration (%) of ingredients in different PRs

	Yasooj 1	Yasooj 2	Yazd 1	Yazd 2	Gafsa
L.O.I <sup>1</sup>	20.10	26.80	1.20	1.42	10.08
P <sub>2</sub> O <sub>5</sub>	7.87	7.34	34.00	35.00	27.54
CaO	32.34	40.32	37.03	37.78	42.04
Ca	0.64	0.65	2.50	2.47	2.68
Al <sub>2</sub> O <sub>3</sub>	4.79	4.25	0.52	0.70	1.26
Fe <sub>2</sub> O <sub>3</sub>	2.62	2.16	7.35	5.11	0.52
F	0.61	0.62	2.37	2.35	3.03
MgO	0.85	0.82	3.48	3.59	1.39
SiO <sub>2</sub>	28.49	16.10	10.10	10.10	5.04
SO <sub>3</sub>	0.40	0.22	0.81	0.28	5.03
Na <sub>2</sub> O	<0.10	<0.10	<0.10	<0.10	<0.10
Cl	<0.10	<0.10	0.34	0.30	0.13
K <sub>2</sub> O	0.82	0.68	0.06	0.1	0.18

<sup>1</sup>L.O.I = loss on ignition

In Fig. 1 the relative abundance of minerals in Yasooj-1 is shown to be calcite (d-spacing = 3.03 Å) quartz (d-spacing = 3.33 Å) apatite (d-spacing = 2.78 Å), clay minerals (d-spacing = 15.05, 4.44 Å) and hematite (d-spacing = 2.69 Å) indicating that calcite and quartz are found in considerable amounts and in Yasooj 2 relative abundance of minerals follow in order calcite, quartz and apatite but calcite is the dominant mineral with quartz and apatite much less. In Asfordi phosphate concentrate (Fig. 3 and 4) in addition to apatite some hematite (iron oxide) and quartz are also found. Gafsa PR contains apatite, calcite, gypsum (d-spacing = 7.69 Å) and some clay minerals (d-spacing = 14.83 Å).

### The PRs potential for direct application as fertilizer

The solubility data for the three conventional reagents (NAC, 2% CA and 2% FA) are shown in Table 4.

Indeed, PR sources suitable for direct application are considered to be 'problem ores' because of their low grade and the presence of accessory minerals and impurities [3]. Besides suitability for beneficiation, the most important factors in the assessment for direct application are grade (P<sub>2</sub>O<sub>5</sub> content) and the reactivity of the apatite (solubility) [3]. Table 3 shows degrees of purity and Table 4 shows amount of soluble P in the extractants as percent of PR and as percent of total P in 5 studied samples.

Table 4 shows a means comparison between soluble P in the extractants as percent of PR and as percent of total P by Duncan test at 0.05 probability. Results shows that Gafsa PR was in first order and the local PRs were in later order. Also, a comparison between soluble P in the extractants as percent of PR (Table 4) and Table 1 shows that the reactivity and the potential of local PRs for direct application are low. However [0], the reactivity of Gafsa PR which is used as a worldwide reference is quite high. A comparison between soluble P in the NAC extractants as percent of PR and Table 2 also shows that the relative agronomic effectiveness (RAE) of local PRs would be less than 30 % and would be classified as PR of very low reactivity while Gafsa PR is among the very soluble PR and its effectiveness in comparison with triple super phosphate would be more than 90 %.

Data in Table 4 shows that solubility of Yasooj PRs as percent of total P are greater than as percent of total P especially in FA. This is because of other mineral components that make it more reactive. Looking at Fig. 1 and 2 the relative abundance of calcite (free carbonate) in Yasooj-1 and Yasooj-2 PRs are quite high. Among all of minerals, free carbonates would have a major influence on PR dissolution because they are more soluble than apatite. They also

Consume a part of the chemical reagents, especially weak extractants such as NAC, used for the solubility tests. To overcome this problem of preferential reaction in the solubility measurements, Chien and Hammond [4] discarded the first NAC extract and measured the amount of PR dissolved in the second NAC extract. Mackay *et al.* [10] and Rajan *et al.* [11] found the sum of four extracts to be more representative. Associated minerals have less influence on acidic extractants such as FA [8, 11]. By increasing the strength of the CA from 2 to 5 percent and then to 15 percent, Mackay *et al.* [10] were able to extract 21, 44 and 59 percent, respectively, of the P in the PR. The influence of free carbonates decreases with the strength of the extractants. Of the three methods that are most commonly used, the 2-percent FA extractant would be preferred for a single chemical extraction procedure [1, 10, 11].

Our data (Table 4) shows that the reactivity of igneous Yazd-1 and Yazd-2 PRs are very low. This result is similar to Lehr and Mc Clilan [26] study. They showed that solubility of igneous PRs is lower than sedimentary PRs. As PRs are relatively insoluble minerals, their geometric surface area is an important parameter determining their rate of dissolution. In igneous PR with coarsely crystallized structures possessing no internal surfaces, the geometric and total

surface areas are similar. In the case of sedimentary PRs, the geometric surface area is about 5 percent of the total surface area because of the porous structure of PR particles [26].

After 20 minutes the rate of dissolution of Yasooj-1 increases greatly (0.67 %P solubilized/min) so that even surpasses that of reference Gafsa PR (0.32 %P solubilized/min) (Fig. 6). This research clearly shows that if Yasooj 1 is concentrated and its mineral contents (lime) are removed it could be considered as a very reactive PR that can be directly used as P fertilizer. This PR in short term (single steps) extraction was classified as low reactivity PR because part of dissolving agents was consumed by the lime. Likewise, this study shows

Table 4: Comparison of dissolution of P in 3 extractant (% P<sub>2</sub>O<sub>5</sub>)

	% of PR			% of total P		
	FA	CA	NAC	FA	CA	NAC
Yasooj 1	2.8b	0.6d	0.1d	29.3b	7.6b	1.1b
Yasooj 2	1.9d	0.1e	0.16e	23.0c	1.2e	0.6c
Yazd 1	2.6c	1.7c	0.25c	7.5d	4.9d	0.8c
Yazd 2	2.8b	1.9b	0.3b	7.6d	5.4c	0.9bc
Gafsa	18.9a	10.6a	5.8a	67.7a	37.8a	20.8a

Mean values with different letters in the column are significantly different (p<0.05)

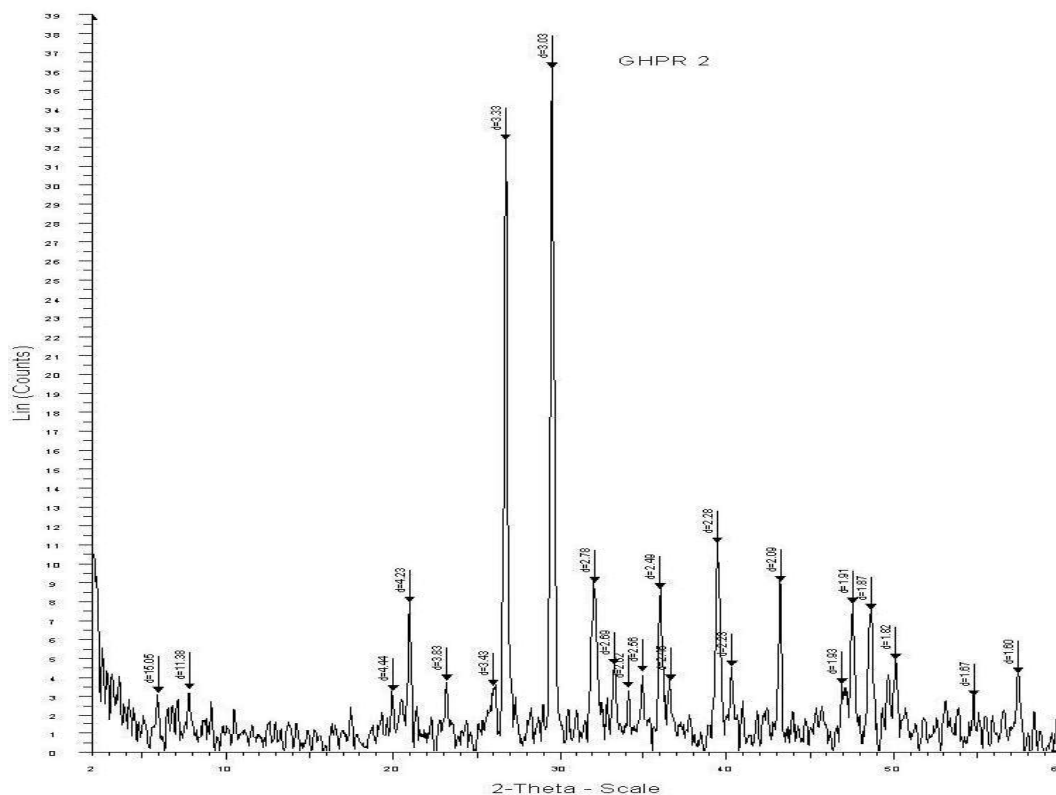


Fig. 1: XRD pattern of Yasooj-1

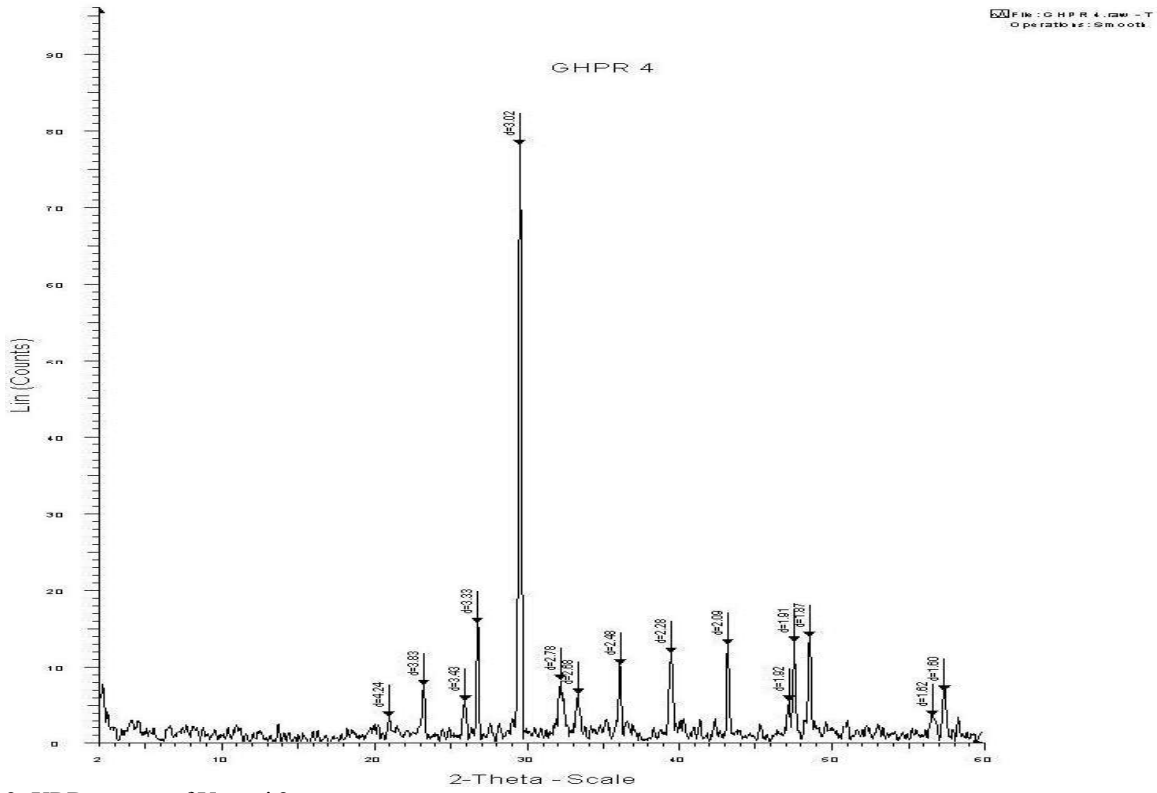


Fig. 2: XRD pattern of Yasooj-2

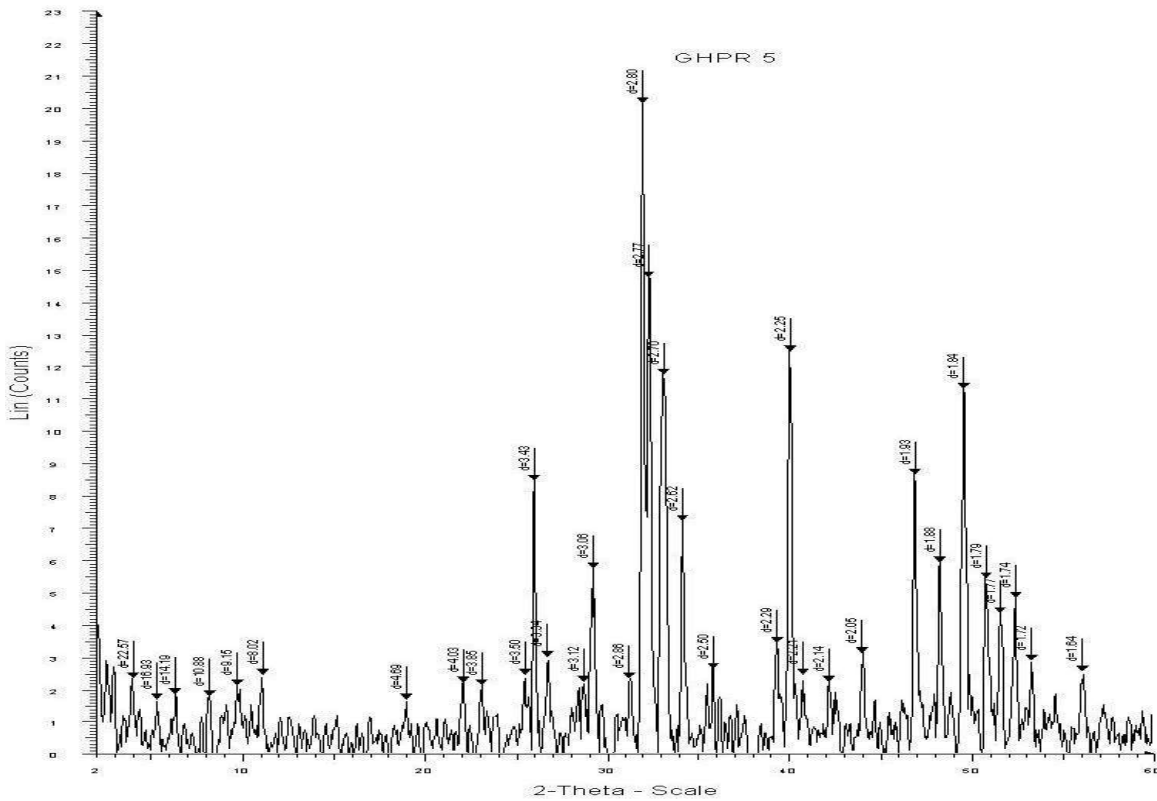


Fig. 3: XRD pattern of Yazd-1

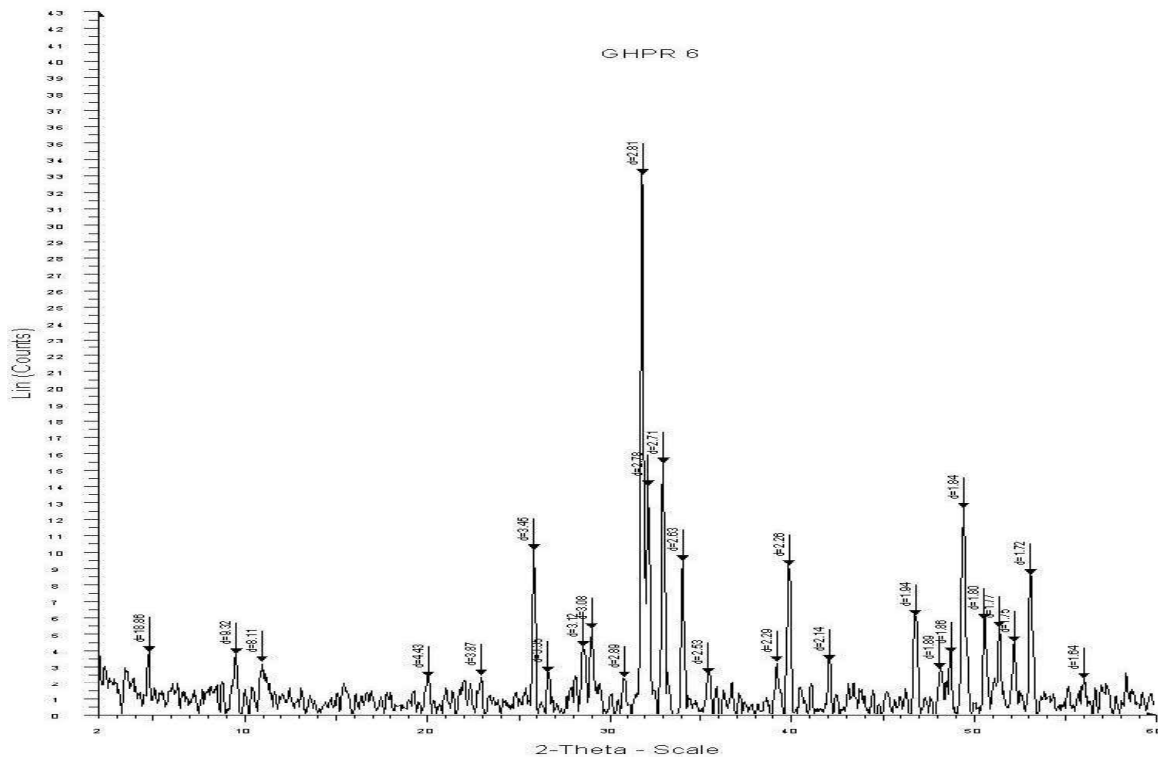


Fig. 4: XRD pattern of Yazd-2

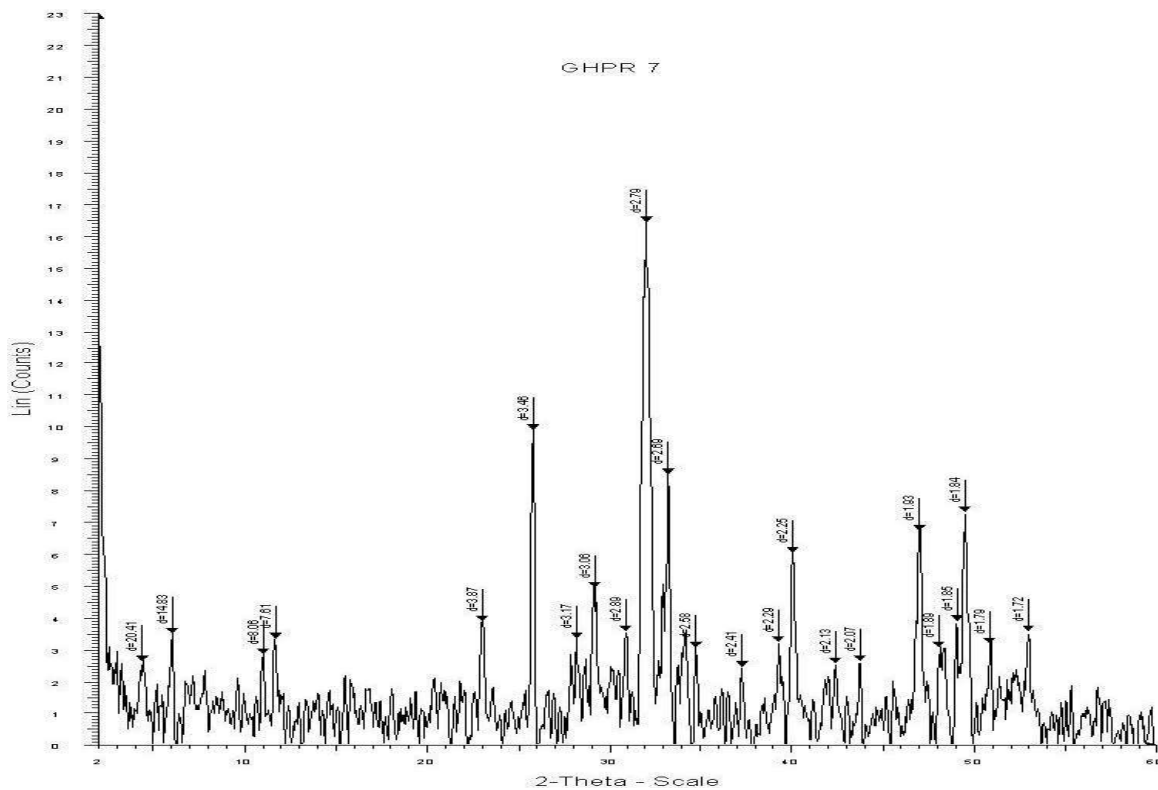


Fig. 5: XRD pattern of Gafsa (Tunisia)

Table 5: Linear and linear form of parabolic equation of dissolution kinetic of PRs in Formic Acid

PRs	Linear equation		
	Equation	R <sup>2</sup>	SE
Yasooj 1	0.15t+25.2	0.73	18.5
Yasooj 2	0.16t+9.7	0.92	10.2
Yazd 1	0.065t-0.7	0.998	0.5
Yazd 2	0.084t-1.2	0.98	1.5
Gafsa	0.16t+16.6	0.92	10.1
Linear form of parabolic equation			
Yasooj 1	4.2t+2.1	0.95	7.2
Yasooj 2	5.1t-21.1	0.99	7.9
Yazd 1	1.9t-12.5	0.89	5.8
Yazd 2	2.7t-18.7	0.91	4.9
Gafsa	4.9t-11.8	0.99	3.6
Linear Equation in 0-20 minutes			
Yasooj 1	0.15t	1	0.0
Yasooj 2	0.19t	1	0.0
Gafsa	0.52t	1	0.0
Linear Equation in 20-100 minutes			
Yasooj 1	0.67t-10.0	0.997	1.5
Yasooj 2	0.28t-1.8	0.999	0.2
Gafsa	0.32t+4.8	0.990	1.35
Linear Equation in 100-600 minutes			
Yasooj 1	0.05t+65.9	0.87	2.9
Yasooj 2	0.11t+31.4	0.89	4.2
Gafsa	0.11t+38.2	0.93	3.8

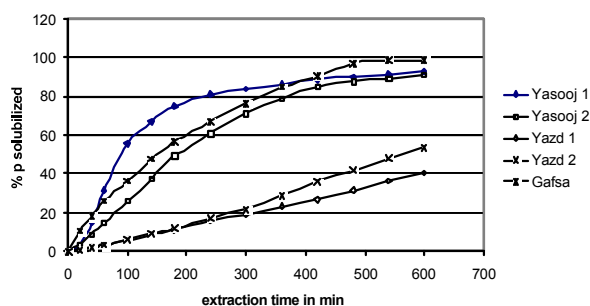


Fig. 6: Long term dissolution of PRs in 2 % formic acid

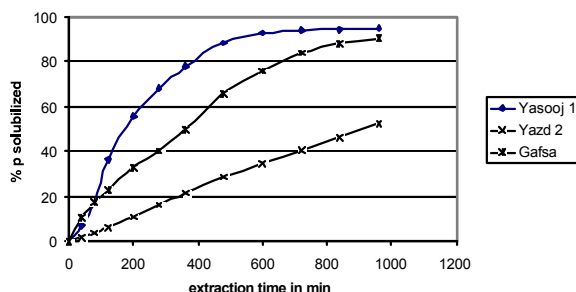


Fig. 7: Long term dissolution of 3 PRs in 2% citric acid

that FA is an effective reagent in beneficiation of this PR and in removing the lime which this result is similar to that of Zafar *et al.* [27].

Truong and Fayard [15] have proposed this procedure for measuring the kinetics of some PR dissolution over time. In general, the curves have shown two parts. The first is a zone of fast dissolution for about 50-60 min, which could be related to the short-term efficiency. The curves for different PRs are steeper and close to each other during this part. The second is a zone of slow dissolution with a pronounced change of slope, representing the long-term effect [15].

Dissolution kinetics of Yasooj-1, Yazd-2 and Gafsa PR in 2% CA is shown in Fig. 7. The results are similar to dissolution kinetics by 2% FA (Fig. 6). So there is no difference between CA and FA as extracting agents for the different PRs. However, the rate of dissolution of these PRs in FA is 1.5 times that of CA. considering that there is no possibility of measuring P dissolved in CA by colorimetric methods [28] therefore FA is preferable as an extracting agent.

## CONCLUSION

The reactivity and dissolution of Yasooj-1 in CA and FA after removal of lime is even higher than reactivity of Gafsa. Therefore by removing calcite and the associate minerals and purifying these PRs, its usage in direct application in certain soils and climates would be very agronomic effectiveness and this PR can be considered quite promising and deserves studies to make it economically and practically feasible.

As it was shown in the case of Yasooj-1 use of single steps dissolution study in order to determine the reactivity of these PRs would not be sufficient for comparison purposes. This PR in single steps dissolution study would classify as low reactive PR, however with kinetic studies its reactivity surpasses that of Gafsa.

The reactivity rate of igneous Yazd PRs are very low and possibly directly application as a fertilizer would not be so effective even in suitable soil and climate conditions.

The correlation between the common extracting agents (FA, CA) is quite high however, because measuring the concentration of dissolved P in FA by colorimetry is possible and the acidity of this extractant is high therefore, this method is preferred to the other two reagents.

## ACNOWLEDGMENTS

This research was conducted at soil science department of Tehran University and with supporting of

Soil and Water Research Institutes and cooperation of the organization of mine and industries of Kohkulye Boyerahmad province and Asfordi mine of Yazd is also appreciated.

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