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# EFFECT OF INTENSIVE MECHANICAL STRESSES ON PHOSPHATE CHEMISTRY AS A WAY TO INCREASE ITS SOLUBILITY FOR FERTILIZER APPLICATION

Mechanical activation technique represents a unique process in which the mineral surface reactivity can be increased in addition to having new products. Phosphate is a one of the plant nutrients that can be produced from phosphate rock. However, direct usage of the phosphate rock has several limitations. In this study, the mechanical activation was used to modify the phosphate structure to be suitable for direct use as a natural fertilizer. It seems a better alternative for the highly sophisticated technology used worldwide to produce completely water-soluble fertilizers.

## INTRODUCTION

The phosphate is one of the important nutrients for the plant growth. Because various phosphate fertilizers differ widely in their solubility (Table 1), it is commonly observed that crop response to phosphate fertilizers varies under the same soil and crop conditions (Maene, 1999; Chien et al., 1990).

The direct application of the phosphate as a fertilizer is limited due to its structure which results in low solubility. The improvement of phosphate structure, through mechanical activation, changes phosphate chemistry and increases its solubility

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(Ibrahim et al., 2010). Substitution rates as measures of the relative effectiveness of alternative phosphorus fertilizersTable 1. illustrates the water-soluble and available phosphate % in common fertilizer sources.

Table 1. Water-soluble and available phosphate % in common fertilizer sources

| $P_2O_5$ Source | % $P_2O_5$ | | |
|---|---|---|---|
| | Total | Available | Water Soluble* |
| Superphosphate (OSP) | 21 | 20 | 85 |
| Conc. superphosphate (CSP) | 45 | 45 | 85 |
| Monoammonium phosphate (MAP) | 49 | 48 | 82 |
| Diammonium phosphate (DAP) | 47 | 46 | 90 |
| Ammonium polyphosphate (APP) | 34 | 34 | 100 |
| Phosphate rock | 34 | 3-8 | 0 |

*Water-soluble data are a percent of the total $P_2O_5$

However, the energy needed for fertilizer application in 2001 amounted to 3660 PJ, which represents about 1% of the global energy demand. Total energy demand has increased at an average rate of 3.8% per year. Despite significant energy efficiency improvements in fertilizer manufacture, improvements in energy efficiency have not been sufficient to offset growing energy demand due to rising fertilizer consumption (Ramírez, 2003).

Therefore, the trends have moved again towards using raw phosphate rock to reduce the cost. The high costs of the production of the fertilizers either by dry or by wet processes as well as the lack of micronutrients in highly soluble fertilizers which have high manufacturing costs are the main drawback of the current fertilizer industry. It was found that iron, zinc and manganese were tied up by phosphate.

In Zimbabwe, using phosphate rock appears to halve the cost of imported fertilizers. The main disadvantage of using the phosphate rock is its low solubility and consequently the low availability of the P for the plant growing. Therefore, the mixing of the phosphate rock with highly soluble fertilizers shows that the savings are impressive. In Kenya, a mixture of 90 percent local phosphate rock is blended with just 10 percent imported fertilizer (Morris, 1988).

Grinding of ordinary phosphorite powder of Central Kyzylkum with urea nitrate and ammonium carbonate salt to the particles size less than 0.16 mm is shown to lead to significant increase in content of $P_2O_5$ in the form consumable by plants (Seitnazarov et al., 2007; Latkin and Samatova, 1998).

It is clear that if the solubility problem of the phosphate rock could be solved then it would be ready to be directly used as a fertilizer. On the way to solve this problem and increase the phosphate rock solubility, highly intensive size reduction equipment like vibrating, centrifugal, jet and other types of mills were employed in the tribo-mechanical treatment of phosphate rocks (Heinike et al., 1977; Kolosov et al., 1979; Paudert et al., 1978; Kozlov and Kozyrev, 1982; Gock et al., 1986). However, many disadvantages in the comminution process were recorded (Gock et al., 1986; Kurrer

and Gock, 1986). That could be reason why such grinding machines did not show economic advantage in the field of phosphate activation under the mechanical forces (Gock et al., 1986).

To overcome the disadvantages of the tribo-mechanical treatment, the development of the principle of rotary-chamber vibrating mill is based upon the introduction of freely moving chamber wheel in the milling tube. Caking of the ground product in such a mill is avoided by the effect of a paddle mixer. Beyond the surface expansion which occurs through the vibrating tube mill, the high collision energy conveyed by this method induces lattice break up through disintegration. This lattice destruction, which changes the initial lattice structure, leads to a growth of free enthalpy (Kurrer, Gock, 1986). The decreasing activation energy related to this has a decisive influence on the reaction kinetics of rock dissolution (Kurrer and Gock, 1986).

In this paper, the mechanical activation process using rotary chamber vibrating mill was tested to increase the solubility of the phosphate ore. The mechanically activated product was identified by XRD, FTIR, thermal analysis, and solubility tests. The comparison, from economic point of view, between the current fertilizer manufacturing and this process was presented.


# EXPERIMENTAL


## MATERIALS


Representative Red Sea phosphate sample of primary crushed ore was used. Chemical composition as well as size distribution of the crushed sample is given in Tables 2 and 3, respectively.


Table 2. Chemical analysis of Red Sea phosphate sample

| Constituents | % |
|---|---|
| $P_2O_5$ | 23.48 |
| CaO | 38.05 |
| $Fe_2O_3$ | 1.61 |
| $Al_2O_3$ | 2.38 |
| MgO | 2.64 |
| $SiO_2$ | 10.14 |
| $CO_2$ | 6.35 |
| Humidity | 3.83 |


Table 3. Size Analysis of Primarily Crushed Red Sea Phosphate Sample, (Mill Feed)

| Size, mm | Wt.% |
|---|---|
| >2.00 | 8.09 |
| -2.0+0.80 | 19.36 |
| -0.80+0.50 | 8.96 |
| -0.50+0.315 | 12.72 |
| -0.315+0.20 | 8.96 |
| -0.20+0.15 | 9.83 |
| -0.15+0.10 | 8.67 |
| -0.10+0.063 | 17.34 |
| <0.063 | 6.07 |

METHODS

THE ROTARY-CHAMBER VIBRATING MILL

All the mechanical activation tests involved in this study were carried out using the pilot scale, circulating oil lubricated, three tube rotary chamber, type "Palla U 35", Humboldt Wedag AG vibrating mill of "Institut fur Aufbereitung, Technische Universitat", Berlin, Germany, Figure 1. The mill was operated under the following predetermined optimum conditions:
- grinding tube capacity 13.4 liter, 300mm diameter, and 190mm height,
- grinding media: 50kg of steel balls, 30mm diameter ,
- machine working conditions: 150 A., 210 V., 95-100 kW, frequency was $1500min^{-1}$, and amplitude 8mm.
- the initial weight of the feed material was 1200g each, grinding time 90 min; 40 g samples were withdrawn every 15min, for size and surface area as well as for the measurements of the structural changes in the apatite lattice by X-ray diffraction and infra red spectroscopy.

The two phosphate samples were separately directed to be ground for 90 mins. The Rotary –Chamber vibrating tube mill, type Palla U35, was used for grinding tests (McClellan, Gremillian, 1976). The machinery conditions were adjusted at the predetermined optimum working parameters (McClellan and Gremillian, 1976).

PHYSICAL CHARACTERIZATION OF MILLED SAMPLES

X-RAY DIFFRACTION ANALYSIS

X-ray diffraction (XRD) was adapted to follow up the physical changes in the major constituents of the ground samples using "Philips – PW1050/25" X-ray diffractometer (Seitnazarov et al., 2007; Latkin, Samatova, 1998). The operating conditions were CuK tube, 40 kV, 20 m Amp. $4X10^2$ cps, and 1°/cm/10 sec scanning speed.

The relative intensities, $I/I_o$, of the X-ray impulses at a lattice plane of a material to be activated, were carried out, before ($I_o$) and after the mechanical stress. Every $I/I_o$ can be assigned to a definite reaction behavior, e.g. the leaching behavior of definite

prescribed reaction conditions. This can be interpreted as a compaction of information resulting from the influence such as grain-size, lattice changes as well as one-or multi-phased solid state reactions.

### INFRA -RED (IR) SPECTRA ANALYSIS

Similar to XRD, infra-red spectroscopy, IR using "PYE-Unicam Philips, SP 2000" IR spectrometer, were used under working conditions shown in the respective Figures. 1-2 gm of the finely ground sample was added to 200 mg KBr as an embedding medium and blended in an agate mortar for 10 minutes. The mixture was then pressed in an evacuated die for 4-5 minutes at about 10 t/cm$^2$ into a transparent pellet of 13 mm diameter and 1mm thickness. The pellets were then scanned 500-2000 cm$^{-1}$ at a total scan time of 10 minutes.

### PARTICLE SIZE ANALYSIS

Particle size measurements of the ground samples were carried out using "PABISCH" type 715 laser granulometer.

### DIFFERENTIAL THERMAL ANALYSIS, DTA

Differential thermal analysis, DTA of the starting and end products were conducted using "Linseis" unit.

### PROCESS KINETICS

Solubility tests according to the method of Stetter (1972) were carried out (Heinike et al., 1977). Other regulations prescribed by Gericke and Kurmies (1972) for earthy phosphates (Kolosov et al., 1979) were also taken into consideration. Accordingly, 2% citric acid (CA) and neutral ammonium citrate (NAC) at 65C$^o$ solutions were the leaching media for the reactivity measurements tests. The following tests conditions have been maintained:

| | | |
|---|---|---|
| Weight of the ore sample | : | 2.5g |
| Decomposition agent-initial concentration | : | 250 ml of 2% citric acid |
| Duration of reaction | : | 30 min |
| Temperature | : | 20$^o$C |
| Separation solid/liquid | : | Centrifuged 2 min at 4000 rpm |
| Reproducibility | : | 1% |

## RESULTS AND DISCUSSION

### MINERALOGICAL CHARACTERIZATION

The mineralogical investigation of the worldwide phosphate deposits has shown that the general fluorapatite composition applies more closely to igneous and high grade metamorphic deposit types than to sedimentary phosphates (Paudert et al., 1978). The composition, high crystallinity and low reactivity of such igneous apatites of Kola, former USSR and Phalaborwa, South Africa make them virtually useless for direct application (Paudert et al., 1978).

Sedimentary phosphate rocks contain apatitic phosphates of more complex chemical composition following, in general, the francolitic-type formula suggested by Lehr and McClellan (1973) (Kozlov and Kozyrev, 1982). The carbonate apatite in most sedimentary phosphate rocks is submicrocrystalline and occurs in several varieties of complex aggregates (Paudert et al., 1978).

Red sea phosphate grains are amorphous and cryptocrystalline cellophane. The ore in characterized by rounded, elongated and irregular grains of different sizes (50 to 200 μm). Biodetrital phosphate in the form of bone fragment, vertebrae and teeth are also identified in Red Sea phosphate cemented with calcareous siliceous matrices. The calcareous cement, most probable dolomite, is observed partially replacing the bone fragments and cellophane especially along the borders in the Red Sea sample.

### PHYSICAL CHARACTERIZATION

### X-RAY DIFFRACTION PATTERN

The principle of using the tribomechanical treatment is based mainly upon the application of the aggressive mechanical energy on the solid phosphates which leads to destruction in the phosphatic crystal texture (Gock et al., 1986). Such lattice imperfection or distortion could be followed by XRD technique (Kolosov et al., 1979). Measurements of the changes in the relative intensities I/Io of the x-ray impulses before ($I_o$) and after (I) the mechanical stress could reflect the amount of lattice imperfection. Peaks covering the range of phosphatic mineral extended along $2\theta = 30°$-$35°$. XRD diagrams of such peaks, at different milling times are illustrated in Fig.1. Extraneous gradual decay in the XRD phosphatic peaks, with increasing milling time proving the validity of the grinding treatment, was clearly noticed in Fig. 1.

Measuring the integrated relative area under each diffracted curve for each milling period, could provide another mean to emulate how much the crystal lattice was distorted. Planometer was used to measure such areas (Kurrer and Gock, 1986). The rational amounts of lattice imperfection were found to increase with milling time. It reached 43.95% after 90 minutes, Table 4, and then variation in unit cell due to activation process is shown in Table 5.
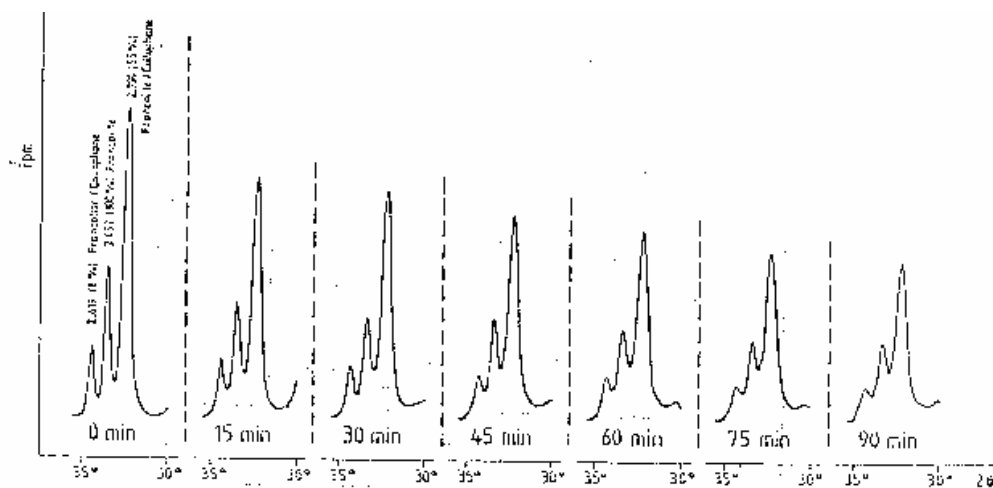
Fig. 1. The XRD analysis of raw Red Sea phosphate at different milling times

Table 4. The integrated relative area of XRD curves for each milling period

| Milling Time, min | Peak Area, $cm^2$ | Disorder % |
|---|---|---|
| 0 | 223 | - |
| 15 | 188 | 15.69 |
| 30 | 166 | 25.56 |
| 45 | 140 | 37.22 |
| 60 | 137 | 38.56 |
| 75 | 130 | 41.70 |
| 90 | 125 | 43.95 |

Boldyrev et al (1976) proved the formation of different coordinate $CaO_6F$ complexes, during the mechanical activation of some apatite minerals (Gock et al., 1986).

Gock and Jacob (1984) came to the conclusion that the destructions of the dolomite or calcite lattice plane causes the transformation of carbonate to oxide forms through $CO_2$ split off by the heat evolved during milling (McClellan, Gremillian, 1976).

Formation of a new compound, suggested to have the formula $Ca_7$ $(CO_3)$ $(Si_6O_{18}).2H_2O$, as a result of the reaction between the highly activated carbonate and silica fragments, was also concluded (1990) (Gock et al., 1986).

Table 5. Unit cell dimensions of the raw and mechanically activated phosphate sample

| Sample | $2\theta$ | $\theta$ | $C(A^o)$ | $a(A^o)$ | c/a |
|---|---|---|---|---|---|
| Crude | 51.82 | 25.910 | 6.8929 | 9.3352 | 0.7383 |
|  | 53.15 | 26.575 | - | - | - |
| Activated | 51.84 | 25.970 | - | - | - |
|  | 53.32 | 26.660 | 6.8723 | 9.3151 | 0.7378 |

The effect of tribomechanical activation of Red Sea raw ore can be ascribed in view of its exogangue minerals contents, mainly silica and silicates. They seemed to play an important role in the process. The same conclusion was reached by Malchikov et al (1983) who got better activation results by adding silica to some phosphate ores (Kurrer, Gock, 1986).

<p style="text-align:center">INFRA-RED SPECTROSCOPY</p>

It was interesting to record the effect of increasing mechanical treatment time through IR technique, Figure 2. The gradual corruption in the major P-O bands at V1 and V3 modes was obvious. They became identical after milling time of 90 minutes. Figures 2 shows the complete disappearance of the calcite peaks at the frequencies 878 and 1420 cm$^{-1}$. Remarkable decrease in the peak of quartz at 798 cm$^{-1}$ frequency was also observed, Figure 2.
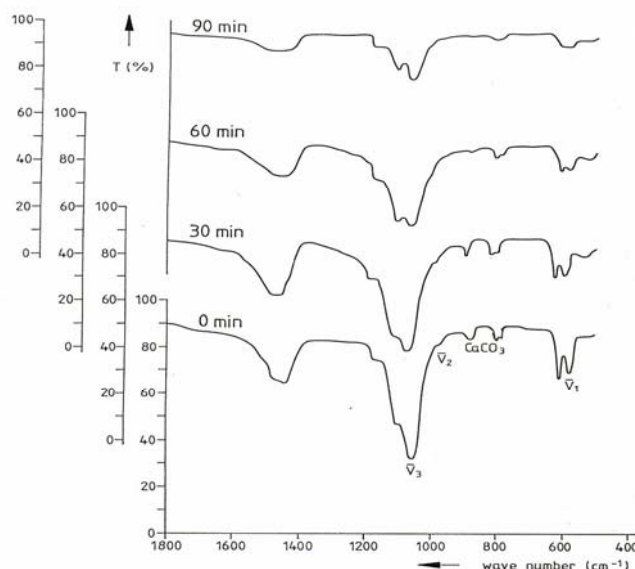


Fig. 2. IR spectra of phosphate samples at different milling times

However, the enhancing effect of the silica on tribo-mechanical activation of the ore is expected to diminish after 90 minutes, where due to over-grinding, the silica-related peaks began to disappear.

<p style="text-align:center">THERMAL BEHAVIOUR OF THE TREATED SAMPLES</p>

Differential thermal analysis of mechanically activated Red Sea raw phosphate sample recorded broad exothermic peak in the temperature range 350-550°C. It may be due to the combustion of organic carbonate materials, which was not affected by mechanical activation. It seems probable that the closed system of milling is

responsible for the non-oxidation of such materials under that high temperature conditions.

The endo-exothermic peaks, appearing in the DTA of the original sample above 700° C, completely disappeared after the mechanical activation process, Fig.3. This may be explained by the decomposition of the inorganic carbonates, due to the excessive mechanical stress and free energy created by activation (McClellan and Gremillian, 1976).
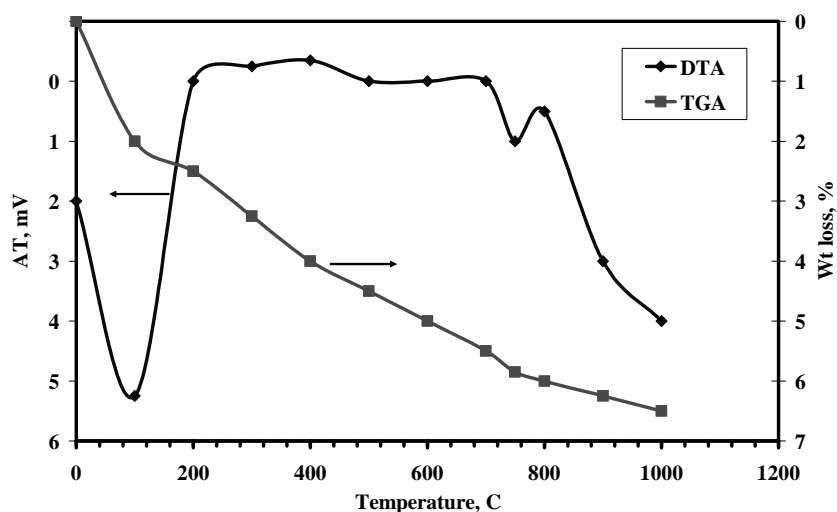


Fig. 3. Thermal analyses of Red Sea raw phosphate sample

The mechanism of activating phosphate rocks by the mechanical treatment does not mean structural distortion in the internal phosphatic composition only, but it may signify also a corresponding enhancement in lattice substitutions especially $CO_3^{-2}$ for $PO_4^{-3}$ (Sikora et al.,1989). It was also reported that the activation process increased in $CO_2$ milling atmosphere (Gock et al., 1986).

PROCESS KINETICS

CHEMICAL REACTIVITY AND SOLUBILITY

The absolute solubility index (ASI) is defined as `(Lehr, McClellan, 1973):
ASI= solvent –soluble $P_2O_5$ % / theoretical $P_2O_5$ concentration of apatite %

The absolute solubility index (ASI) of Red Sea phosphate in 2 % citric acid, CA and neutral ammonium citrate (NAC) were measured, Figs 4-5. A remarkable increase in the dissolution rates of the activated products was recorded, indicating the efficiency of the mechanical activation process. Gradual improvement in the ASI

values of the milled products with increasing milling time, signifies the advantage of the grinding of the extended time.
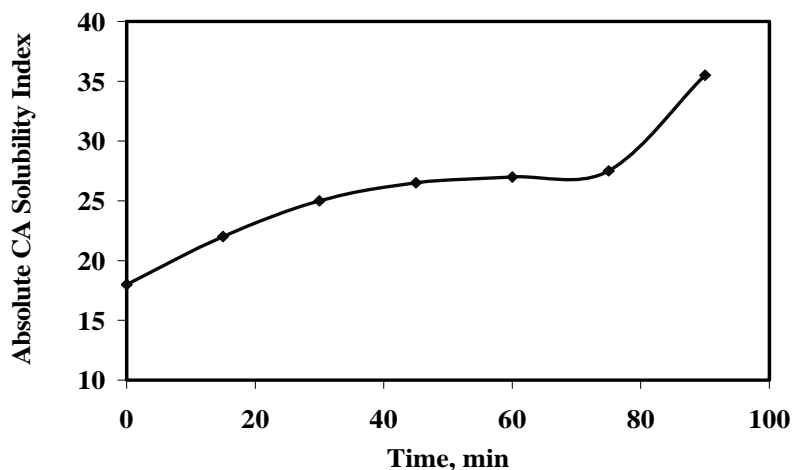


Fig. 4. Solubility Index of raw phosphate in 2% citric acid at different milling times

It is obvious from the curves that the solubility of the mechanically activated began to increase gradually for about 75 minutes after which a rapid increase in solubility took place. The absolute CA solubility Index values increased from 26 to 35.55 between 75 and 90 min.
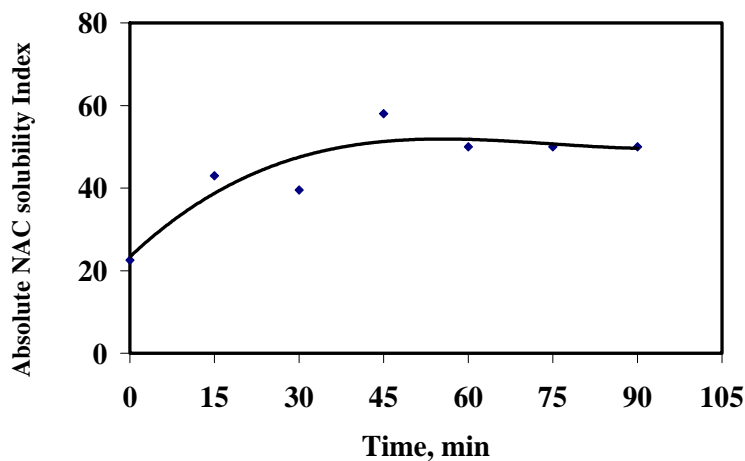


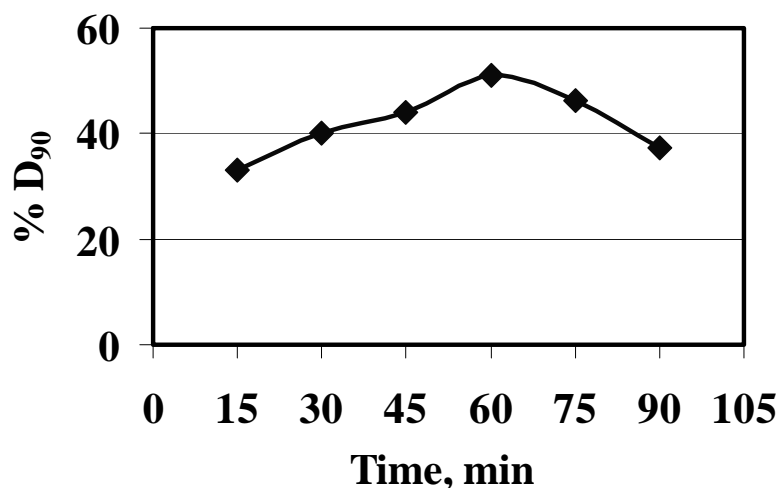Fig. 5. Solubility Index of phosphate in neutral ammonium citrate at different milling times

Fig. 6. Effect of milling time on %D$_{90}$ of ground products

The solubility behaviour of either unactivated or activated samples in the alkaline medium was altered, Fig.5. Treated raw samples showed increase in the leaching efficiency till 60 min milling time. After that the absolute solubility index reaches asymptotic value. This trend is identical with that of both XRD pattern and IR spectra related to quartz, where it began to loose its frictional rubbing effect due to intensive size reduction after 60 min. Hard silica particles enhance the milling inside the mill by acting as small diameter grinding media, and accordingly increasing the internal lattice alteration of phosphate grains, which is leading to an increase in their the solubility character.

Therefore, the solubility optimum value at 45 min milling time in NAC was also observed (Fig. 5). This latter trend is identical with that of the change in %D$_{90}$ of the phosphate by the increase in activation time (Fig. 6) which illustrates the effect of the particle size on its solubility in NAC.

Mechanical activation of phosphate in highly intensive size reduction equipment such as modified vibrating mill results in the dispersion of the milled product (Gock, Jacob, 1984). Gradual increase in the %D$_{90}$ values with increasing milling time is shown in Fig. 6.

Evidently, symptoms of agglomeration were recorded after 60 min. The progressive structural changes in the apatite lattice with time are proving that dispersion is not necessarily the main process affecting rock reactivity during mechanical activation (compare Fig. 4 and 6).
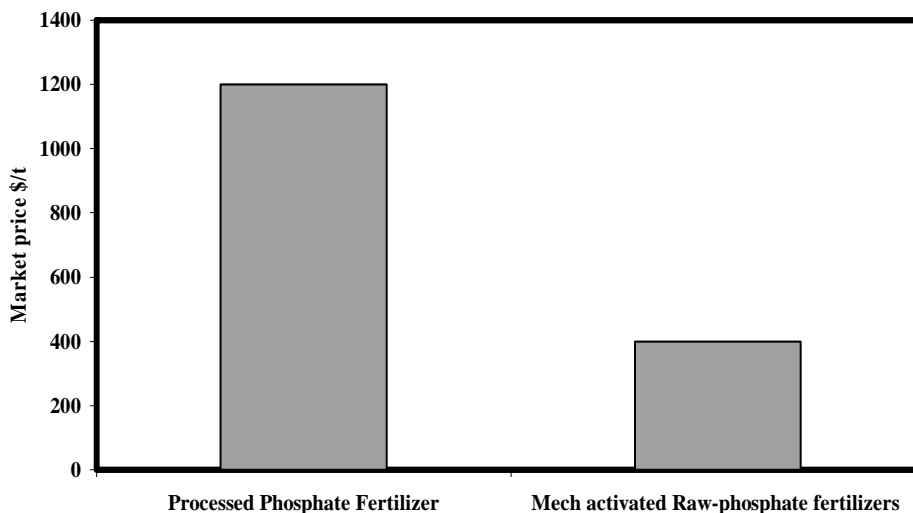
Fig. 7. Costs comparison of standard fertilizers to mechanically activated rock fertilizers

ASSOCIATED GANGUE MINERALS EFFECT

Most of the phosphate ores contain impurities, which may affect the mechanical activation process either positively or negatively. The prevailing impurity, in most of the phosphate ores, is silica. In case of adding the raw phosphate as a fertilizer, the silica impurities does not harm the soil because of it represents one of the soil components.

In regard to mechanical activation process, it was reported that the effect of tribo-mechanical activation of Red Sea phosphor-concentrate, after such a change in its chemical and mineralogical characteristics, should be interesting, where it showed lower tendency to the mechanical activation compared to the raw ore (Ibrahim, 1989). Such behavior may be ascribed to its exo-gangue minerals contents, mainly silica and silicates. They seem to play an important role in the process. The same conclusion was reached by Malchikov et al (1977) who got better activation results by adding silica to some phosphate ores (Malchikov et al., 1977).

ECONOMIC PREFERENCE OF MECHANICAL ACTIVATION

The comparison was carried out between the costs of mechanical activation of the phosphate ore with the running of different beneficiation steps to produce a concentrate suitable for the production of the phosphoric acid, which is an intermediate step of all the fertilizer manufacturing process. This comparison shows that the activation will lead to substantial savings, Fig.7. Initially, the saving is due to

the reduction in the capital and operating costs of the beneficiation steps. Moreover, the same thing will happen to the wet process that cost much more than beneficiation processes due to dealing with acids (i.e., one ton of sulfuric acid costs about $ 700) and the corrosion problems, which increase the maintenance costs as well.

## CONCLUSIONS

Raw phosphate represents an alternative for currently used phosphate fertilizers due to costs and energy limitations. One of the reasons behind the rare use of the phosphate as a direct application fertilizer is its solubility. To overcome this limitation, the mechanical activation was tried and has achieved a marked increase in the reactivity of the treated phosphate samples. The chemical reactivity of activated raw samples in 2% citric acid and neutral ammonium citrate, showed improved leaching characteristics for all all the milling times tested.

Therefore, the technology of tribo-mechanical activation of phosphate rocks for direct application, due to its simplicity, may open the way for utilization of indigenous ores in many developing countries and serve regional markets when arbitrary grade specifications do not apply and would give the security to be independent of the international chemical concerns.

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Technika aktywacji mechanicznej jest unikalnym procesem, w którym reaktywność powierzchni mineralnej może wzrastać dając w rezultacie nowy produkt. Fosforan jest jednym z ważnych składników odżywczych dla upraw roślinnych, który może być produkowany z rud fosforanowych. Jednakże, bezpośrednie zastosowanie rudy fosforanowej posiada szereg ograniczeń. W tych badaniach, aktywacja mechaniczna była zastosowana dla modyfikacji struktury wewnętrznej, w taki sposób by był on użyty bezpośrednio jako nawóz mineralny. Wydaje się, że dobrą alternatywą dla wysoce skomplikowanej technologii produkcji nawozu fosforowego jest użycie całkowicie rozpuszczalnego w wodzie nawozu otrzymanego po mechanicznej aktywacji rudy fosforanowej.

*słowa kluczowe: aktywacja mechaniczna, zaburzenia sieci krystalicznej, nawozy fosforowe, reaktywność*