



## Energy conversion in phosphate ore grain mixture activated via ultra-centrifugal mill



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### ABSTRACT

The aim of this investigation was to establish the optimal processing parameters of the comminuted phosphate grain mixture mechanical treatment by monitoring and evaluating the activated product characteristics. An ultra-centrifugal mill with a peripheral comminuting path was used as activator in the experiment. A mathematical model based on the theoretical principles of the grain mixture mechanical processing was applied in order to characterize the phosphate activation procedure. As the energy conversion that takes place by the interaction mill–material during activation is recorded on the processed phosphate grains, the increase in potential energy of the activated grains was measured by means of an automatic grain counter. Following the operating hypothesis of the automatic grain counter, the energy conversion and the properties of phosphate grains induced by mechanical force were monitored and expressed in the form of the grain inertia change. The phosphate ore grain mixture used in the experiment was thoroughly analyzed, particularly in its activated state. Analytically obtained results were supported by X-ray diffraction analysis for identification of crystalline phases and change in crystallinity, and SEM microphotography of initial and activated phosphate grains. Diverse comparison analyses disclosed that the optimal set of activation process parameters could reduce the negative effect of phosphate sample immanent properties on the final score and furthermore enhance the material reactivity.

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### 1. Introduction

Phosphate minerals, which are obtained from the phosphate rocks via various refining procedures, represent an essential basic material for different applications: industry of ceramic materials and anti-corrosion agents, as well as metallurgy (Guosheng et al., 2012; García-Páez et al., 2014); fertilizers, animal feed supplements and food preservatives (Delvasto et al., 2008; Al-Fariss et al., 2013); components of artificial bones (Hemmati et al., 2014; Tomoaia et al., 2014); and material for water treatment (Mesquita et al., 2011). Most of the worldwide phosphate reserves originate from sedimentary ore deposits. In terms of the application quality and P<sub>2</sub>O<sub>5</sub> portion, phosphate ores are divided into three groups: low grade ores (12–16% P<sub>2</sub>O<sub>5</sub>), intermediate-grade (17–25% P<sub>2</sub>O<sub>5</sub>), and high-grade ores (26–35% P<sub>2</sub>O<sub>5</sub>) (Mohammadkhani et al., 2011). The commercial phosphate ores originate from the deposits that are able to produce approximately 28–38% P<sub>2</sub>O<sub>5</sub> after previous mining and processing (Lassis et al., 2015). During the last decade, the worldwide demands for phosphates required approximate global production as high as 145 million tons per year (Abouzeid et al., 2009; Jasinski, 2007).

Constant development in the industry and global society imposes increased requisite for improvement in quality, as well as larger production of phosphates.

Phosphate rocks are mineralogically complex as they usually contain one or more phosphate minerals. The mineral which is the most suitable for commercial utilization is fluorapatite (3Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>·CaF<sub>2</sub>). The phosphate ore undergoes a refining procedure to match the “market grade” which is set at approximately 30% P<sub>2</sub>O<sub>5</sub>. The beneficiation method for upgrading of a phosphate ore is determined by the preset type of phosphate mineral as well as the associated gangue minerals (Straaten, 2002; Abouzeid et al., 2009). The most frequently utilized techniques are: size reduction and screening for relatively friable phosphate minerals; attrition scrubbing and classification when clays are the main gangue minerals (Abouzeid, 2008); electrostatic separation for calcareous (Hammoud, 1973) and siliceous ores (Abdel Moneim, 1971); magnetic separation when the major gangue constituent is magnetic (Shaikh and Dixit, 1993); chemical dissolution for carbonate calcareous phosphate ores (Gu, 2002); and flotation as a most common processing method used for a variety of siliceous and calcareous ores (Abouzeid et al., 2009; Mohammadkhani et al., 2011; Elgillani and Abouzeid, 1993; Qi, 1993; Guosheng et al., 2012). Activation of a phosphate mineral grain mixture via different mills (i.e. centrifugal, vibration, attrition, jet mill) is often applied as an addition to a mechanical or other refining

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**Table 1**  
Chemical composition of the phosphate ore.

Oxides	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	CaO	MgO	P <sub>2</sub> O <sub>5</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	S	F <sup>-</sup>	Cl <sup>-</sup>	Loi*
wt.%	50.09	5.57	3.29	0.45	15.04	0.32	20.08	0.28	2.76	0.04	0.03	0.01	2.04

\* At 1000 °C.

technique (Tönsuaadu et al., 2011; Balaz, 2003; Tkačova et al., 1993; Urakaev, 2011; Al-Wakeel, 2005). Comminution in finer sizes represents a crucial operation for achieving the required degree of liberation or specific surface, even though it requires certain energy consumption and process optimization (Razavian et al., 2014).

The mechanical activation technology is grounded on the action of mechanical energy on solid substance in a high-energy mill (Balaz, 2003; Senna, 2010). The changes in energetic (mainly gravitational and electromagnetic) properties of a processed material are the main outcome of activation (Boldyrev et al., 1993; Cheng et al., 1999). Namely, the activation procedure represents a closed system that contains the fields of active forces of devices and mineral grains in which centrifugal acceleration may exceed gravitational acceleration by 100 times (Petrov and Milošević, 1999). Also, gravitational force in the area around particles may exceed the weight of the grains by several tens of times which results in elastic and plastic modifications and changes in shape, volume and density of the grains. Besides improvement in grain-size composition of a treated material, mechanical activation induces an increase in surface and chemical reactivity as well as the potential energy of the system due to the excess of free energy and volume (Temujin et al., 2009; Senneca et al., 2011; Kumar and Kumar, 2011).

The mechanical activation by an ultra centrifugal mill was applied as an additional grain mixture treatment in this study, because phosphate ore treated via mechanical comminution (i.e. crushing and grinding) inherently contains a high level of impurities that decreases the quality of a final product. The phosphate grain energy conversion and properties that are induced by mechanical force were monitored and expressed in form of the grain inertia change through the proposed operating hypothesis of an automatic grain counter. The main goal of the study was to highlight the optimal set of activation parameters that can reduce the negative effect of phosphate immanent properties on the final score and to enhance the material reactivity which can be efficiently used in rationalization of processing of a target material.

## 2. Materials and methods

### 2.1. Material

The phosphate ore was acquired from the “Lisina” deposit in Serbia. The investigated ore is classified as an intermediate grade sedimentary

**Table 2**  
Granulometric composition of the initial phosphate ore sample.

Size classes, mm	Fraction, % weight	Accumulated retained, %	Accumulated passing, %
+ 25.40	26.23	26.23	100.00
– 25.40 + 22.20	9.57	35.80	73.77
– 22.20 + 19.10	9.39	45.19	64.20
– 19.10 + 15.90	11.05	56.24	54.81
– 15.90 + 11.10	14.13	70.37	43.76
– 11.10 + 9.52	6.96	77.33	29.63
– 9.52 + 7.93	2.81	80.14	22.67
– 7.93 + 6.35	4.27	84.41	19.86
– 6.35 + 5.00	1.15	85.56	15.59
– 5.00 + 3.15	5.94	91.50	14.44
– 3.15 + 2.00	2.54	94.04	8.50
– 2.00 + 0.00	5.96	100.00	5.96
Total	100		

phosphate rock. A representative ore sample of 300 kg, obtained from the deposit according to the standard ore sampling campaign (Merks, 1986) was used in the investigation. After initial rough crushing via a primary jaw crusher and secondary cone crusher, the samples were split and bagged in 10 kg sub-samples. The chemical analysis of the phosphate ore was performed by means of the atomic emission spectroscopy on a PinAAcle 900 spectrometer (Perkin Elmer, Waltham Massachusetts, USA). The five samples were acquired by the cone & quartering method from a 10 kg sub-sample. The samples for the chemical analysis were further pulverized in a laboratory mill. Obtained average values are given in Table 1.

A standard dry sieve analysis was applied to assess the grain-size distribution of the initial ore sample (ASTM C136/C136M-14). The analyses were performed on five samples obtained by the cone & quartering method from a 10 kg sub-sample. Average results are presented in Table 2.

The grain mixtures from 10 kg sub-samples were submitted to further grain size reduction as a preparation for the subsequent mechanical activation procedure. Comminuting of the samples was performed in three grinding stages in order to obtain the grain size that could be used as input for the activator. The first stage of the comminution was carried out in a laboratory jaw crusher with 10 mm output opening working in a closed circle with a screen. Afterwards, the sample was subjected to secondary size reduction in a laboratory roll crusher with 5 mm output opening. The third stage of grinding was conducted in a ceramic-lined ball mill. The mill, in which ceramic balls were used as the grinding media, was working in the closed circuit with an air classifier. The grain-fraction content of the sample prepared for mechanical activation was obtained by cyclo-sizer diffraction particle size analysis (Cyclo-sizer – Warman International LTD, Australia). The obtained density of the sample was 1.76 g/cm<sup>3</sup>.

### 2.2. Mechanical activation principle

The investigated phosphate grain mixture was mechanically activated by means of a Retsch ZM-1 ultra-centrifugal mill with a peripheral comminution path (Gemini BV, Netherlands).<sup>1</sup> The mill was supplied with high alloyed steel rotor with a 100 mm diameter. A number of rotor revolutions were adjusted at either 10,000 rpm or 20,000 rpm. By being in conjunction with vibrating adder, the mill could operate discontinuously or continuously. The mill contained a high alloyed steel ring sieve of variable mesh size which together with the rotor made a reacting system for transfer of energy from a device to a dispersive phase, i.e. activation product. Mechanical reduction of coarse particles was performed by a dynamic counter-balancing of material between working element (rotor) and ring sieve. This shape of rotor cogs (three-sided prisms placed on a basis) enabled streaming of air and fluidized dispersed material. The tolerance between rotor cogs and ring sieve was 1 mm. The created ring volume was 4.74 cm<sup>3</sup>. Due to a high rotor speed and strong centrifugal force, this space was permeable in one direction only – towards the sieve, i.e. the particles moved from the rotor towards the sieve and vice versa in the ring zone. The material was able to leave this zone only when the particle size became smaller than the mesh size.

<sup>1</sup> Working principle: <https://www.youtube.com/watch?v=Q8onmaJJN1I>; manufacturer: [http://www.geminibv.nl/labware/retsch-maler-zm1-en?set\\_language=en](http://www.geminibv.nl/labware/retsch-maler-zm1-en?set_language=en).

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