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Chemometric analysis of the influence of mechanical activation on the mica quality parameters

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Abstract

The differences in the set of the process parameters measured before and after mica mechanical activation and their influence on the grain size distribution related characteristics have been studied. The modification of the behavior for activated samples has been correlated to the particle size distribution effect produced by activation via an ultra centrifugal mill. The mechanical treatments are energetically and economically unsustainable procedures, therefore the mica activation was optimized on basis of assessment of the process variables effect on the final quality of product parameters. Response surface method, standard score analysis and principal component analysis were used as means of the optimization. Developed models showed r^2 values in the range of 0.816–0.988 and they were able to accurately predict quality parameters in a wide range of processing parameters. Standard score analysis highlighted that the optimal sample was obtained using sieve mesh of 80 μm set of processing parameters (SS = 0.81). Multiple comparison tests revealed that the optimal variation in the processing parameters could reduce the negative effect of mica samples inherent properties on the final score and improve activation procedure energetic and economic sustainability.

Keywords: A. Milling; B. Grain size; B. Electron microscopy; C. Mechanical properties; D. SiO_2; E. Thermal applications; Analytical modeling; Response surface analysis; Principal component analysis

1. Introduction

The mica comprises a group of 37 phyllosilicate minerals characterized by a layered texture. All mica minerals exhibit monoclinic symmetry with pseudohexagonal crystals, and the excellent cleavage due to the hexagonal sheet-like arrangement of atoms [1,2]. From the commercial aspect, the most frequently applied mica is muscovite: $KAl_2(AlSi_3O_{10})(OH)_2$ [3]. The applicability is based on the possibility of crystalline layers delamination into transparent to opaque thin sheets which are chemically inert, dielectric, elastic, flexible, hydrophilic, insulating, lightweight, reflective, refractive and resilient. This mica's unique set of physical properties brings about its stability towards electricity, light, moisture, and extreme temperatures exposure. Muscovite, which is both insulator and dielectric, and has a high dielectric breakdown, is thermally stable to 500 °C [3]. Phlogopite ($KMg_3(AlSi_3O_{10})(OH)$) mica remains stable at even higher temperatures (to 900 °C) [4]. Mica insulation ability is utilized in high temperature and fire-resistant materials like power cables in aluminum plants, blast furnaces, heaters and boilers, metal smelters, and tanks and furnace wiring [3,4]. Construction and ceramic materials sector represents a wide target area for mica application. Mica is used as a compound for gypsum and for filling blemishes in gypsum wallboards because it acts as filler and extender (provides a smooth consistency, improves the workability and cracking resistance) [5]. Mica is also used as an insulator in concrete blocks, but it can also be applied as a pozzolanic material. Some lightweight aggregates, such as diatomite, perlite, and vermiculite, may be substituted with ground mica when used as filler [6,7]. Mica can be a soil conditioner and an additive to drilling fluids [8]. Furthermore, it is applied in the paint

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Nomenclature

MS	sieve mesh size, μm
NRR	number of rotor revolutions, rotations per minute / rpm
CI	current intensity, A
MAP	mechanical activation period, min
CRS	circumferential rotor speed, m/s
Q	capacity, i.e. batch size of ultra centrifugal mechanical activator, kg/h
SEC	specific energy consumption (engine power/mill capacity), kWh/t
d_1, d_2	mesh sizes of the used sieves, μm[1]
R_1, R_2	cumulative over-sizes, %[1]
d'	average grain size, μm[1]
d_{95}	sieve mesh size appropriate to 95% cumulative undersize of the micronized product, μm[1]
n	level of micronization kinetics[1]
S_t	calculated (theoretical) specific surface area, m^2/kg[1]
S_{r*}	real specific surface area, m^2/kg*

Parameters derived and/or calculated from grinding kinetic model based on Rosin–Rammler–Sperling equation and dependent on particle size distribution of the activated sample. The parameters d' and n are obtained by analytical procedure which implies selecting of the two farthest points on the grain size composition diagram and fitting new curves through marked points. New curves are described by the equation: $\log\log\frac{100}{R_i} = n\log d_i - n\log d' + \log\log e$, $i=1$ or 2.

industry as a pigment extender, in the production of rolled roofing and asphalt shingles, in glass–ceramics, and as filler in syntheses of variety of advanced materials [3,9–13].

When mica is used in advanced ceramics the mechanical activation is regarded as a regular processing method, because traditional methods (comminuting, classification and flotation) can not produce fine enough mica concentrate [14]. Activation conducted via different mills results in changes in the crystal structure of treated mineral. Namely, the activation produces significant number of crystal lattice defects and meta-stable forms which cause changes in surface properties of solid phases and in the system structure [15–17]. Mechanical activation is a complex chemico-physical process that not only effects the simple change of the grain size, it also induces the increase in potential energy and chemical activity, surface reactivity and mineral phase alternations within the treated system. As a consequence, the activated system gains entirely different set of physical properties, which is followed by the alternations in the mechanical and thermal behavior of treated mineral [18–23]. Ultra-fine grinding can be economically unsustainable due to low mill capacity and high energy consumption; therefore an optimization of the activation procedure is necessary. In this study, response surface methodology (RSM) was applied as a means of the mica mechanical activation procedure optimization. The RSM is an effective tool for optimizing a variety of processes [24,25]. The main advantage of RSM is reduced number of experimental runs that provide sufficient information for statistically valid results. The RSM equations describe effects of the test variables on the observed responses, determine test variables interrelationships and represent the combined effect of all test variables in the observed responses, enabling the experimenter to make efficient exploration of the process.

The main objective of this study was to investigate the quality of the mechanically activated mica conducted by ultra centrifugal mechanical activator Retsch ZM-1 using different process parameters. To assess the quality of activated mica several parameters (d_1/d_2, R_1/R_2, d', n, d_{95}, S_t and S_r) were determined, and the influence of NRR, CI, MAP, CRS, and Q, were observed. Experimental results were subjected to analysis of variance (ANOVA) to show relations between applied assays. In order to enable more comprehensive comparison between investigated samples, standard score (SS) was introduced. Principal component analysis (PCA) was applied to classify and discriminate analyzed samples. Multiple comparison tests revealed that the optimal variation in the processing parameters could reduce the negative effect of mica samples inherent properties on the final score and improve activation procedure energetic and economic sustainability.

2. Experimental: materials and methods

2.1. Characterization of the mica

The mica ($KAl_2(Si_3Al)O_{10}(OH,F)_2$) from ore deposit in Bujanovac, Serbia was used as the starting material in this investigation. The mica ore samples consisted of platelets of about 10 cm in length and 2 cm in thickness. Subsequently to the initial separation and crushing, samples were lightly ground using a knife-mill and sieved under 2 mm. Obtained mica sample had density of 3.00 g/cm^3, humidity before/after drying 10.00/0.5%, and bulk density of 1.17 g/cm^3 [26]. Comminuted mica was submitted to treatment in flotation concentration plant. Thus obtained samples, with chemical composition [26] and grain size distribution [26] as showed in Table 1 and Fig. 1, were mechanically treated by means of an ultra centrifugal mill. The atomic emission spectroscopy technique performed by a PinAAcle 900 atomic absorption spectrometer (Perkin Elmer, Waltham Massachusetts, USA) was applied in the chemical analysis. The grain size distribution was analyzed by a cyclo-sizer (Warman International LTD, Australia).

A high speed rotor activator – an ultra centrifugal mill Retsch ZM-1 (Retsch GmbH, Haan, Germany) was used for mica processing in this investigation. Ultra centrifugal mill is a default device for rapid size reduction of soft, brittle, and

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