Serbian Committee for Mineral Processing

Technical Faculty in Bor of the University of Belgrade
Institute for Mining and Metallurgy Bor
ITNMS Belgrade
Mining Faculty Belgrade of the University of Belgrade

XXI

INTERNATIONAL SERBIAN SYMPOSIUM ON MINERAL PROCESSING

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Edited by:
Zoran S. Marković and Ljubiša Andrić

JU "SPORTSKI CENTAR" Bor
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THE INFLUENCE OF MECHANICAL ACTIVATION ON RAW MATERIAL PROPERTIES FOR HEAVY CLAY PRODUCTION

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ABSTRACT

This paper contains the comparative review of experimentally determined results of raw material /clay/ properties prepared following both classical procedures of processing and mechanical activation. Classical procedure of raw material processing was carried out by milling in perforated rolls and milling on differential rollers up to grain size not exceeding 1 mm. Mechanical activation of the same raw material was carried out in lab mill "Pulverisette 6" (Fritsch, Germany) over the time periods of 30, 60, 90 and 120 minutes. Both inactivated and mechanically activated samples were subjected to testing of technological features relevant for process of forming, drying and firing heavy clay products. Obtained outputs indicate essential change of activated samples features in comparison to sample prepared following classical procedure.

Keywords: mechanical activation, technological investigations, masonry product

INTRODUCTION

Although clay and loam have already been thousands of years used for making bricks and tiles[1], the relation between their properties, technological „behaving" in production process and properties of fired product has to be always carefully studied from the beginning. Reason for this lay in fact that there are numerous varieties of clay which might be, for example, of the same chemical composition but also completely different technological features[2]. The process of examination usually comprises examination of raw deposit area in detail, lab testing of raw materials or clay mixture composite and preparation of "industrial trial".

In principal, raw materials used in production of masonry products are represented by the following mixtures:

- clay minerals (determining viscous - plastic properties of mixture)
- carbonates, oxides of iron and other minerals, which are specific fluxes materials, and
- Quartz /sand, non plastic material stabilizing filler for the ceramic body, as dominant mineral creation being present in all clay raw materials.

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Technological features of heavy clay products will depend on relation in participation of the above mentioned silicates in raw composite mixtures.

Mechanical activation[3, 4] is the process of increasing reactive capability of materials where activated material remains chemically unchanged. By mechanical activation material is brought in excited condition when it might be also potentially capable of switching into some other, more stabile state. Manner and way of developing change from state of excited to stabile state depend on, in addition to everything else, manner of mechanical activation, nature of activated material as well as other chemical components the activated materials possess an chemical affinity to.

EXPERIMENTAL RESEARCH

Representative sample of masonry clay was used in experiment. The analyzing of raw material was done by determining its chemical, mineral and grain composition.
Masonry clay was treated using classical processing procedure; firstly, it was dried at 60°C and then milled down in lab perforated rolls mill /kollerwalzwerk/. Then, sample was moisten and milled in lab differential mill first at gap of 3mm and then of 1mm. In further examination so prepared sample is treated as inactivated sample which is marked with "Sample A".

Part of masonry clay representative sample is mechanically treated by milling in „Fritsch" mill involving time of mechanical activation of 30 minutes (Sample B), 60 minutes (Sample C), 90 minutes (Sample D) and 120 minutes (Sample E).
Examination of technological features essential for process of forming, drying and firing of masonry products was conducted on inactivated and mechanically activate samples.
Features being essential for raw material processing comprise the determination of: mean content of remains at 10.000 apert. /cm^2 sieve and mineralogical definition of remains. Remains at sieve is determined by wet sieving and mineralogical defining remains by microscopic examination.

The following essential parameters for process of forming were determined: qtty. of water required for plastic processing and plasticity after Feferkorn (coefficient and plasticity criterion, angle ctg.)[5].

The sensitivity at drying of examined samples was determined by recording Bigot curve on Barelotograph. Furtheron, test samples were prepared in the shape of cube by hand shaping in 30x30x30mm mold. Bodies were dried on air and then dried up in lab dryer at 105°C up to achieving constant mass. Dry test samples were fired in lab furnace in accordance with prescribed firing regime up to maximal temperature of 900°C. Fired test samples were determined for total shrinking, compressive strength, loos of mass at firing and water absorption.

Characterization of raw materials

Characterization of raw materials were carried out: by determining chemical composition, using X-ray diffraction, DTA and TG analyse, dilatometric analyse and determining granulometric composition.

Table 1. below represents results of chemical silicate analyse and Fig. 1 shows histogram of masonry clay granulometric composition.

Table 1. Results of chemical analyse

Parametre	u %
mass loss at 1000°C	11.71
SiO_2	53.23
Al_2O_3	13.64
Fe_2O_3	5.34
CaO	7.50
MgO	3.59
SO_3	0.00
S^{2-}	0.00
Na_2O	1.24
K_2O	3.42
MnO	0.091
TiO_2	0.60
Zbir:	100.36
Insolluble content	71.13
water loss	2.04

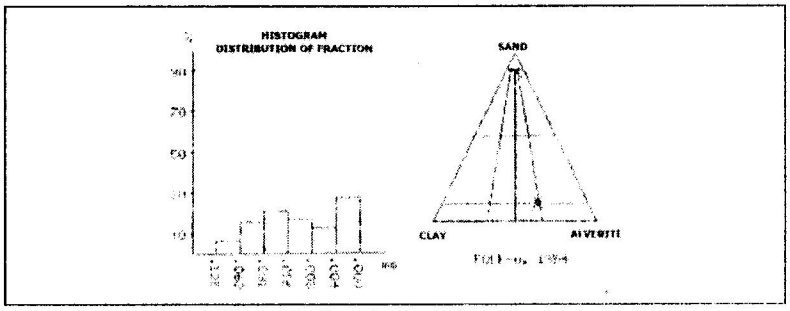

Figure 1. Granulometric histogram

The results of masonry clay examination, show low content of Al_2O_3 (12.88%), content of SiO_2 of 49.97 and content of Fe_2O_3 abt. 5.3%.

Based on results from granulometric composition analyse it can be clearly seen that alevrite (dusty) fraction is dominant having the content of clay component of abt. 30% and sandy fraction of 10.60%.

Fig. 2 shows differential-thermic curve and thermal-gravimetric curve of raw material and Fig. 3 shows dilatometric curve at warming up to 1000°C.

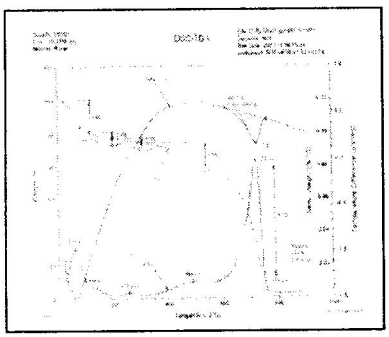

Figure 2. DTA and TGA of raw material

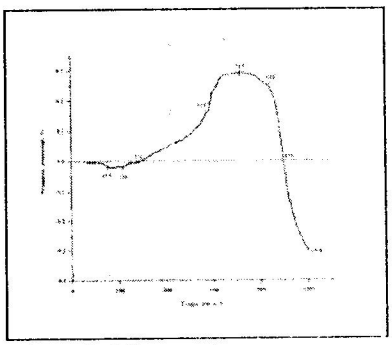

Figure 3. Dilatometric curv

Figs. 4 and 5 show X-ray curves of raw material.

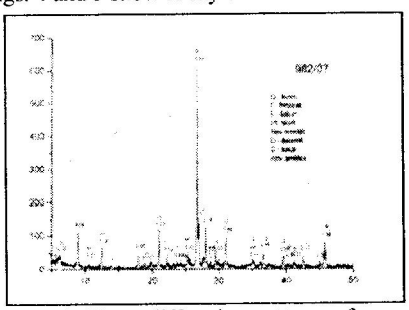

Figure 4. X-ray diffraction patterns of raw material

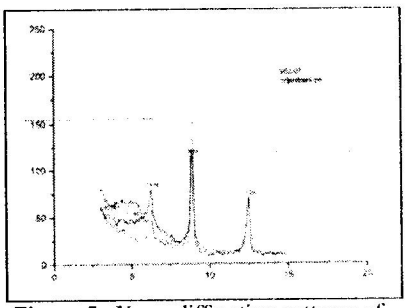

Figure 5. X-ray diffraction patterns of directed raw material

DTA and TG analyses indicate presence of iron hydroxide and some organic matters.

The result of dylatometric examination confirms presence of the mentioned minerals and defines presence of free quartz at abt. 39%.

X-ray examination shows quartz as the most representative mineral. Mica, chlorite and some smectite appear as „stratified"silicates. Feldspars within group of Plagioclase and minerals within the group of carbonates (calcite and calcite dolomite) are present. Amphibole appears in traces.

Technological features of examined samples

As for Sample A., mean content of remain at 10.000 apert. /cm^2 sieve is determined to 3.76%. Microscopic identification showed that remain is composed by quartz as dominant component, then by mica, calcite and ball-like gathered carbonate matters. There was not remaining at sieve from mechanically activated samples (Samples B, C, D and E).

Table 2. Contains the rewiev of basic properties of examined samples which are essential for process of shaping.

		Test sample A	Test sample B	Test sample C	Test sample D	Test sample E
The quantity of water for plastic treatment (%)		20.64	30.71	31.12	32.32	28.39
Feferkorn plasticity	Coefficient of plasticity	27.2	35.38	38.96	40.34	33.94
	Plasticity criteria	Good plastic	Highly plastic	Highly plastic	The most plastic	Highly plastic
	ctg angle	0.36	0.35	0.46	0.20	0.19

Table 3. Contains the rewiev of basic properties of examined samples which are essential for process of draying.

		Test sample A	Test sample B	Test sample C	Test sample D	Test sample E
Compressive strength (Mpa)		11.68	15.37	15.20	13.43	12.79
Shrinkage at draying process		6,41	8.17	7.31	8.55	6.84
Bigot-curve	Shrinkage at critical point K_1(%)	5.12	7.67	5.99	7.70	4.73
	Loss of water at critical point K_1(%)	9.62	17.37	16.75	18.36	17.37
	Drying sensitivity	Sensitive	Highly sensitive	Highly sensitive	Highly sensitive	Highly sensitive

Table 4. Contains the rewiev of basic properties of examined samples obtained after process of firing.

	Test sample A	Test sample B	Test sample C	Test sample D	Test sample E
Compressive strength (MPa)	35.44	51.19	48.79	58.22	47.92
Loss of mass at firing (%)	10.66	10.64	10.61	10.56	10.77
Total shrinkage (%)	5.9	8.81	8.65	10.02	9.28
Water absorption (%)	16.82	17,75	19.04	18.87	18.44
Color of fired test samples	red	red	red	red	red
Harmful influence of carbonate	Rear balls	-	-	-	-

Analyse and discussion of results

Detected content of particles larger than 63 microns for inactivated tested sample (A) is 3.76%. As in the fraction carbonate minerals are present, they have negative influence on quality of fired products thus causing the appearance of rare carbonate balls and carbonate skim.

Mechanically threated samples (B, C, D and E) do not contain particles larger than 63 microns, so negative influence of carbonate on fired samples is eliminated.

After Feferkorn method, inactivated sample (A) belong to group of well plastic raw materials. It is stated that mechanical activation obtained by milling leads to higher value of plastic coefficient. It shows a growing tendency with increasing time of activation up to 90 minutes (samples B through D) and than significant drop of plastic coefficient takes place. (Sample E). Latter can be explained with fact that within mechanical activation fine reducing of particles occurs what leads to increase of plasticity. Following further increasing of activation time, and after certain particle size is reached, the positive effect of milling stops thus enabling agglomeration of particles as well as lowering of plasticity. This is in accordance with different „packing" of particle along the process of forming, what is further on in correlation with measured values of compressive strength in dry state. The increse of compressive strength is clearly noticed within test samples B and C, and than the decrease of compressive strength with further increasing of activation time (test samples D and E).

It is also determined that the drying sensitivity is changing within mechanical activation, as well as that test samples become more sensitive on draying. Linear dependence vs activation time can not be established neither for position of critical point at Bigot curve nor for shrinkage of activated samples at drying.

The compressive strength of fired samples indicate increasing tendency of mechanical properties with increasing of mechanical activation time up to 90 minutes a than also the decreasing of mechanical properties being likely connected with activation energy and particle „packing".

CONCLUSION

Based on conducted research it can be stated that mechanical activation and energy engaged in the process of milling exerts the influence on increasing of the following: plasticity of raw composite and increasing mechanical properties of ready products after firing.

There are justified assumptions that by applying mechanical activation in case of "non plastic" raw materials the plasticity can be increased as well as mechanical properties of final products improved. The results obtained indicate justification for continuation of research of mechanisms being of influence, throughout the process of mechanical activation, on change in technological features of raw materials. Next step is to design raw material mixtures of inactivated and activated raw materials as well as optimization of technological parameters in process of making masonry products.

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