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Waste sludge from washing dolomite aggregates as a raw material for brick production

Introduction

Dolomite deposits are very popular mineral resources in Poland used in various industries (Radwanek-Bąk and Nieć 2015) and the important role of meeting the needs of society. Their resources should be therefore rationally used. It requires in particular safeguarding access to deposits areas. The way to indicate the valuable deposits to protect them seems to be their valuation, using uniform criteria. The authors present a simple method of mineral

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deposits value ranking. It consists of deposits ranking value in four, separate domains: quality and size (resources). Dolomite mining for crushed stone production amounted to $3 \cdot 10^6$ Mg in 2017 (PIG-PIB 2018). They are often used as concrete aggregates and as road aggregates. Aggregates are washed to remove clayey pollutants. During this process, waste sludge containing clay minerals and fine-grained dolomite are produced (Naziemiec 2015). Their properties and amount depends on the type of raw material and type of washing technology (Naziemiec 2015). Washing generates between 10–15% sludge of total aggregate production.

The utilization of waste from washing aggregates is a common problem and has not been sought out yet (Naziemiec 2015). The possibilities of the application of different sludge were considered (Careddu and Dino 2016; González-Corrochano et al. 2016; Moreno-Maroto et al. 2017). Research on the usefulness of sludge from washing limestone aggregates as an additive to ceramic masses are presented in two authors earlier works (Kłosek-Wawrzyn et al. 2017; Kłosek-Wawrzyn and Małolepszy 2016). The influence of pure dolomite on the brick body was also determined (Kłosek-Wawrzyn and Bugaj 2016). Dolomite quarry waste as sand replacement in sand brick was considered in Nur Fitriah's work (Nur Fitriah et al. 2016).

According to (Kłosek-Wawrzyn and Bugaj 2018) there are indications that sludge from washing dolomite aggregates can be used in ceramic building technology. In the aforementioned article, authors compared the physico-chemical properties of two sludges: from dolomite and limestone aggregates washing. Due to the instability of fired material in the presence of water vapor, sludge from limestone aggregates washing was disqualified as the main raw material for building ceramic masses. Fired lumps of the waste sludge from washing dolomite aggregates washing after 4 hours of steam subjection were unchanged. Compared to typical Polish clay raw materials for building ceramics (Kłosek-Wawrzyn 2016) dolomite sludge has a similar grain size composition and grinding during ceramics production is not necessary. Characteristic temperatures (beginning of sintering – 790°C, reaction sintering – 900°C, liquid phase sintering – 1165°C, softening – 1205°C, melting – 1215°C and flow – 1220°C) are also suitable for the production of building ceramics (Kłosek-Wawrzyn and Bugaj 2018). This paper presents technological properties, phase composition and microstructure of materials made of waste from washing dolomite aggregates. The usefulness of this waste as the main raw material for building ceramic masses was considered.

1. Materials and methods

1.1. Materials

Devonian dolomite washing sludge from the dolomite mine and stone processing plants located in the Central-Eastern part of Poland washing aggregates sludge were examined.

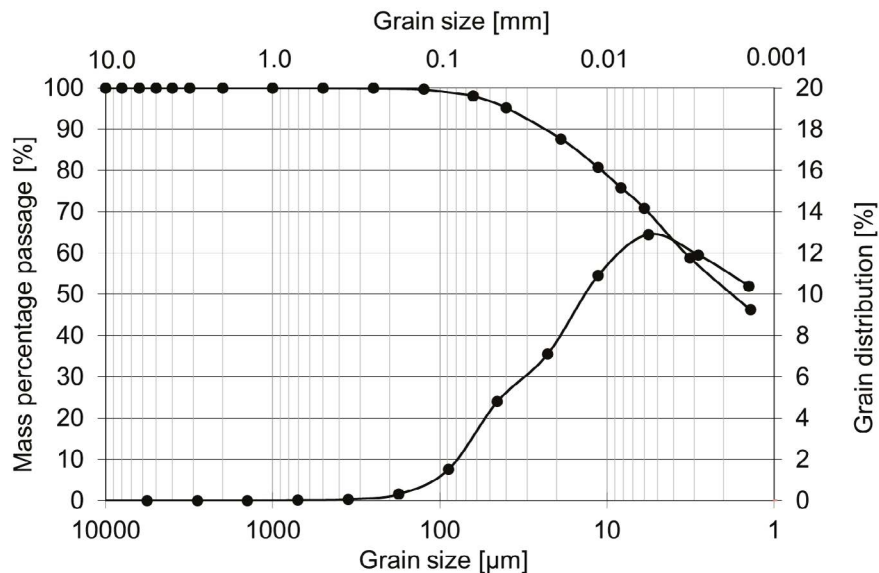


Fig. 1. Grain size of waste sludge from washing dolomite aggregates

Rys. 1. Skład ziarnowy odpadu z płukania kruszywa dolomitowego

The washing of aggregates was carried out in a log washer, vibrating screens, hydrocyclone and filter press. The average water content of the delivered samples of sludge was: $21.7 \pm 1.2\%$. According to the sieve analysis, the amount of grains with a diameter smaller than $63 \mu\text{m}$ is 98%. The results of the areometric analysis were presented in Figure 1. They indicate a monomodal grain size distribution of waste sludge from washing dolomite aggregates. The most frequently occurring diameter is $5.66 \mu\text{m}$. The fine-grainedness of the raw material results from its mineral composition and high content of clay minerals. According to the semiquantitative analysis, the crystalline parts consist of about 45% of clay minerals and about 40% of carbonates (Table 1). An amount of carbonates calculated on the basis of thermogravimetric analysis (Fig. 2) is about 41–44%. According to Figure 2, during the heating of waste sludge from washing dolomite aggregates the following process occurs:

- ◆ during I stage: dehydration of clay minerals,
- ◆ during II stage: firing of organic matter, dehydration of goethite, dehydroxylation of clay minerals,
- ◆ during III stage: two-step decomposition of dolomite to $\text{MgO} + \text{CO}_2$ and $\text{CaO} + \text{CO}_2$, one-step decomposition of calcite to CaO and CO_2 ,
- ◆ during IV stage: destruction of the structure of clay minerals and crystallization of new phases (first peak on DTA curve is an evidence for high illite content and second for kaolinite content in waste sludge).

Table 1. Semiquantitative mineral composition of waste sludge from washing dolomite aggregates

Tabela 1. Analiza półilościowa składu mineralnego odpadu z płukania kruszywa dolomitowego

Group of minerals	Mineral Name	Chemical Formula	Semiquantitative concentration (Weight %)
Carbonates	Dolomite	$\text{CaMg}(\text{CO}_3)_2$	33
	Calcite	CaCO_3	6
Clay minerals	Illite	$\text{KAl}_2(\text{Si}_3\text{Al})\text{O}_{10}(\text{OH})_2$	37
	Kaolinite	$\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$	8
Additional minerals	Quartz low	SiO_2	13
	Goethite	$\text{FeO}(\text{OH})$	3

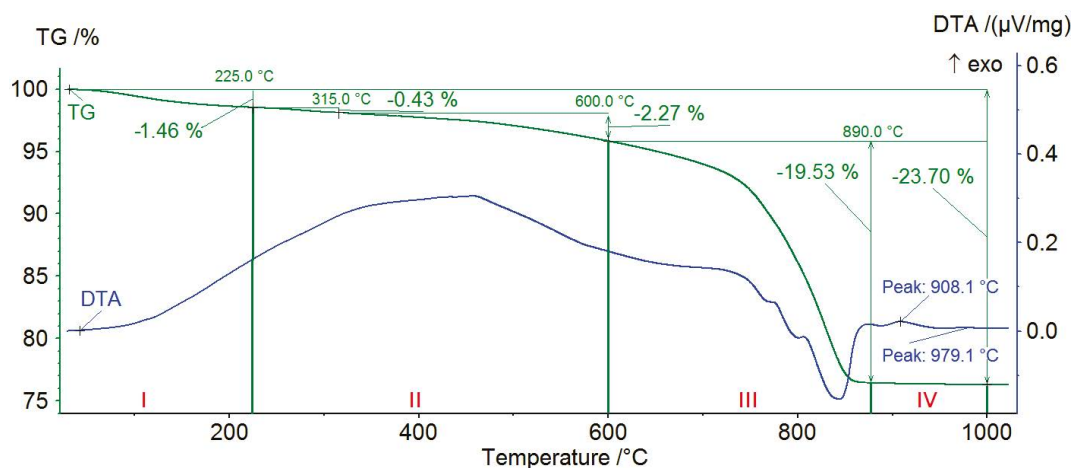


Fig. 2. Results of thermal analysis DTA/TG of waste sludge from washing dolomite aggregates

Rys. 2. Wyniki analizy termicznej DTA/TG odpadu z płukania kruszywa dolomitowego

Wider characteristics of the average sample of the sludge used in this work were described in the paper (Kłosek-Wawrzyn and Bugaj 2018).

1.2. Methods

The research was divided into three parts: technological properties analysis, phase composition analysis and microstructure analysis.

Samples made of waste sludge from washing dolomite aggregates were formed in the laboratory clay brick vacuum extruder and fired at 900°C, 1000°C and 1100°C (temperature range for reaction sintering). For final materials, apparent density, open porosity, water absorption, compressive strength and durability were examined (according to methods described in the standard for masonry units ([European Standard PN-EN 771-1+A1:2015-10](#))).

The color of the materials was examined by spectrophotometer Konica Minolta CM700d, by providing the values of L, a, b in the CIELab system. Markings of color indicators were as follows:

- ◆ L – luminance (brightness),
- ◆ a – color from green (negative values) to red (positive values),
- ◆ b – color from blue (negative values) to yellow (positive values).

The phase composition was investigated by the XRD method. The Seifert FPM XRD 7 analyzer was used. The parameters were as follows:

- ◆ measuring range $2\theta = 5\text{--}60^\circ$,
- ◆ detector – scintillation counter,
- ◆ Cu anode, anode current – 29 mA,
- ◆ lamp voltage – 35 kV,
- ◆ counting time – 3 s,
- ◆ counter movement – $0,05^\circ$.

The microstructure of polished sections of materials was observed using a scanning electron microscope FEI Nova 200 Nanos with EDS attachment. Backscattered-electron (BSE) imaging was used.

2. Results and discussion

2.1. Technological properties

The average values of dried and fired materials properties are presented in Table 2. The bulk density of dried material was $1.92\pm 0.03\text{ g/cm}^3$. The average drying shrinkage was $9.7\pm 0.3\%$, which is similar to typical Polish clay raw materials for building ceramics. The value of drying shrinkage depends on amount of mixing water. Samples were formed from waste sludge from washing dolomite aggregates with the water content as was supplied (without the addition of water or drying).

Differences in the materials properties depend on the firing temperature. Significant differences are detected for material fired at 1100°C. Firing the shrinkage of samples from the temperature of 1100°C is 50% smaller than samples fired at 900°C and 1000°C. It is caused by a high reaction rate between clay minerals and CaO connected with the crystallization of the new phases: gehlenite, akermanite, diopside, hedenbergite (Fig. 3). The dominant process of reaction sintering (driven by the difference in the concentration of substrates)

Table 2. Average values of properties of fired materials made of waste sludge from washing dolomite aggregates

Tabela 2. Właściwości wypalonych tworzyw z odpadu z płukania kruszywa dolomitowego

Firing temperature (°C)	Dried material		Fired material								
	B (g/cm ³)	D (%)	F (%)	A (%)	P (%)	ρ (g/cm ³)	R _c (MPa)	Dur (-)	CI		
									L	a	b
900	1.92±0.03	9.7±0.3	2.6±0.7	16.9±0.8	29.2±0.9	1.72±0.01	34.0±1.5	*	62	13	19
1 000			2.6±0.2	18.8±0.6	30.5±1.1	1.62±0.01	37.2±1.8	*	78	6	15
1 100			1.3±0.3	20.1±0.5	31.8±0.8	1.58±0.01	52.7±1.9	*	74	0	22

B – bulk density; D – drying shrinkage; F – firing shrinkage; A – Water adsorption; P – Open porosity; ρ – Apparent density; R_c – Compressive strength, Dur – Durability, * – meets the requirements (European Standard, PN-EN 771-1+A1:2015-10; Polish Standard, PN-B-12012:2007, n.d.), CI – colour indicator.

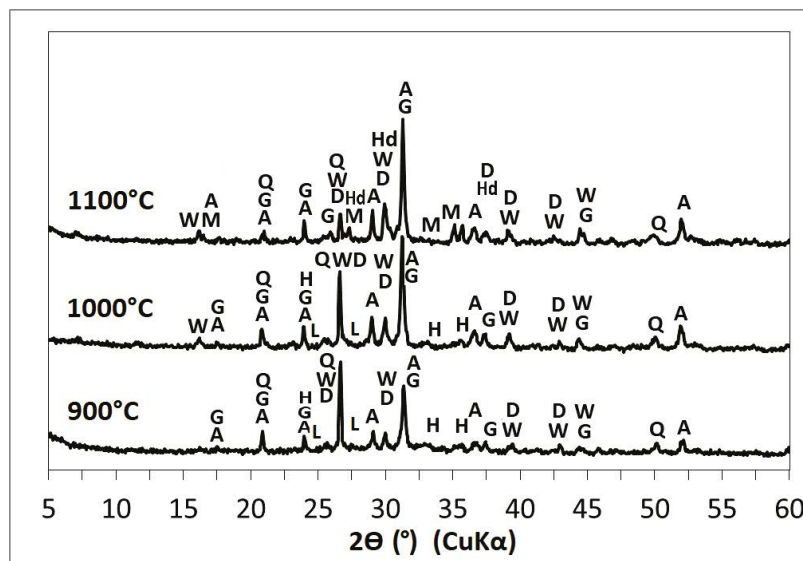


Fig. 3. XRD analysis of materials obtained from waste sludge from washing dolomite aggregates at three temperature
 A – akermanite, D – diopside, G – gehlenite, H – hematite, Hd – hedenbergite, M – mullite, L – leucite, Q – quartz, W – wollastonite

Rys. 3. XRD tworzyw otrzymanych z odpadu z płukania kruszywa dolomitowego wypalonych w trzech temperaturach
 A – akermanit, D – diopsyd, G – gehlenit, H – hematyt, Hd – hedenbergit, M – mullit, L – leucyt, Q – kwarc, W – wollastonit

and the crystallization of new phases do not cause the effective thickening of the material or even its expansion. This was confirmed by dilatometer tests (Kłosek-Wawrzyn and Bugaj 2019). For the same reason, as the temperature rises, the density of the material decreases and its porosity increases. The formation of a liquid phase, that dissolves after the material counteracts the rapid increase in porosity. Water absorption also increases with the increasing firing temperature. The water absorption of materials fired at 1000°C and 1100°C is respectively 11% and 19% higher than the water absorption of material fired at the lowest temperature (values in relative percent). The relative percentage differences in open porosity between materials fired at 1100°C and 900°C is 9% and between materials fired at 1000°C and 900°C is 4%. The increase of firing temperature caused a 6% and 8% decrease in apparent density respectively for materials fired at 1000°C and 1100°C. Compared to the materials fired at 900°C, the compressive strength increased by 9% and 35% respectively for materials fired at 1000°C and 1100°C, which is the result of a higher firing temperature and high rate of reaction sintering. The mentioned materials changes in density, porosity, water absorption and compressive strength were mainly caused by the reaction of CaO and/or MgO with SiO₂ and/or Al₂O₃, which were a volume expansion process with the formation of the new phases.

The durability (frost resistance) of all the examined materials presented in Table 2 meets the requirements for applicability to elements subjected to passive exposure (European Standard, PN-EN 771-1+A1:2015-10; Polish Standard, PN-B-12012:2007). According to durability results, all materials can be used for masonry protected against water penetration and without contact with soil and ground water and also for masonry subjected to passive exposure (European Standard, PN-EN 771-1+A1:2015-10).

Increasing firing temperature caused changes in the color of the materials (Table 2). According to CIE76, the color difference coefficient (ΔE_{ab}^*) is about 18 (between the colors of materials fired at 900°C and 1000°C and also between the colors of materials fired at 900°C and 1100°C). Color difference coefficient (ΔE_{ab}^*) between colors of materials fired at 1000°C and 1100°C is about 10. These values ΔE_{ab}^* of point to noticeable differences in the colors of all materials. Based on visual observations, it was found that materials fired at 900°C are reddish and materials fired at 1100°C are yellowish. Color changes are caused by smaller amount of hematite and also its reaction with CaO and dehydrated clay minerals to new phases e.g. hedenbergite (Fig. 3).

2.2. Phase composition analysis

Figure 3 presents results of phase composition analysis (XRD method). The characteristic crystalline phases for the materials made of dolomite sludge are: akermanite – Ca₂MgSi₂O₇, diopside – MgCaSi₂O₆, hematite – Fe₂O₃, leucite – KAlSi₂O₆, gehlenite – Ca₂Al₂SiO₇, wollastonite – CaSiO₃, quartz – SiO₂ (for all materials) and also hedenbergite – FeCaSi₂O₆ and mullite – Al₆Si₂O₁₃ (for material fired at 1100°C).

The tested system is in a state of chemical imbalance. With increasing firing temperature the following changes occur:

- ◆ the amount of leucite decreases due to its dissolution and melt formation,
- ◆ the total amount of following phases: akermanite, gehlenite, diopside and wollastonite increases due to solid-state reactions, melting and crystallization from the melt,
- ◆ quartz is consumed for the formation of new phases during its reaction with other minerals present in the ceramic body and its dissolution in the melt,
- ◆ hematite disappears at 1100°C but a new phase containing iron is formed – hedenbergite,
- ◆ a small amount of mullite appears in the materials fired at 1100°C, probably formed during crystallization from the melt.

According to (Rashin 1965) the composition of melilite crystallizing from a melt is a strong function of temperature. The highest-temperature melilite formed from the melt is relatively aluminum-rich (gehlenite) and becomes progressively magnesium-rich with falling temperature (akermanite). On this basis it can be concluded that the longer the cooling time (and the higher the firing temperature), the more formed gehlenite will transform into akermanite. Therefore, the gehlenite phase dominates in material fired at 900°C and the akermanite phase dominates in material fired at 1100°C. Mg-rich pyroxene (diopside) are transformed to Fe-rich pyroxene (hedenbergite) at a higher temperature. As a result of a smaller amount of hematite and the presence of hedenbergite, material fired at 1100°C has a yellowish color (Table 2). The presence of a small amount of mullite an evidence for the existence of kaolinite in the raw material and a large amount of liquid during the sintering phase (Fig. 4).

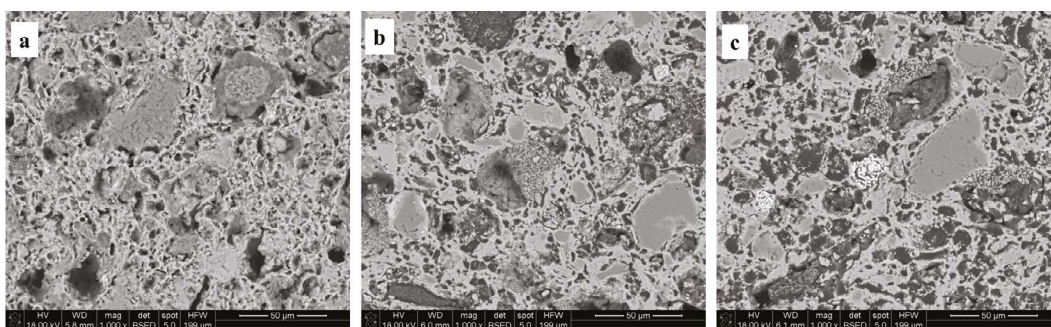


Fig. 4. BSE microphotographs of materials polished sections obtained from waste sludge from washing dolomite aggregates fired at a – 900°C, b – 1000°C, c – 1100°C

Rys. 4. Mikrofotografie BSE zglądów tworzyw z odpadu z płukania kruszywa dolomitowego wypalonych a – 900°C, b – 1000°C, c – 1100°C

2.3. Microstructure analysis

The microstructure (obtained by the BSE method) of the polished sections of fired materials are shown in Figure 4. All materials are highly porous due to the decarbonation of dolomite and calcite. There are two types of pores: micro-pores trapped in a silicate matrix and large pores surrounded by a porous matrix. The first type of pores was probably formed by trapping CO₂ in the softening structure of the material and/or the decarbonation of very fine carbonates. The second type of pores was formed during the decarbonation of coarse carbonates. In their interior new calcium-aluminosilicate and calcium-magnesium-aluminosilicate phases and melts were formed. High temperature solid-state reactions between CaO and/or MgO with SiO₂ and/or Al₂O₃, melt forming and crystallization from the liquid phase did not reduce the total porosity. This is due to small viscosity and the high ability of the crystallization of the melt. With an increasing firing temperature, changes typical for the progress of sintering are observed: an increase in the amount of the glassy phase and an increase in the amount of larger pores with the simultaneous disappearance of smaller pores. An increase in compressive strength may result from the presence of high amounts of the glassy phase in materials fired at higher temperature and new crystalline phases.

Conclusion

In this paper research on the possibility of using waste from washing dolomite aggregates in the building ceramics technology was presented. The results of technological research suggest the possibility of its application in building ceramics technology as brick materials. According to the durability results, all materials can be used for masonry protected against water penetration and without contact with soil and ground water and also for masonry subjected to passive exposure.

Waste from washing dolomite aggregates can be used without processing as a raw material for building ceramics masses. Without any additional technological operations, a material with satisfactory properties was obtained.

Due to the different sludge properties, which depend on the method of their production (type of washers, screens, drainage devices and filter presses and also their parameters) and the properties of the washed aggregates (material, grain size, clay minerals content) it is necessary to verify the obtained results each time using sludge in the production of building ceramics.

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WASTE SLUDGE FROM WASHING DOLOMITE AGGREGATES AS A RAW MATERIAL FOR THE BRICKS PRODUCTION

Keywords

properties, ceramic materials, dolomite sludge, building ceramics,
waste sludge from washing dolomite aggregates

Abstract

The amount of waste from washing dolomite aggregates increases continuously. Aggregates are washed to remove clayey pollutants. They consist of a large amount of clay minerals and carbonates. Their properties and amount depends on the type of raw material and type of washing technology. Utilization of waste from washing aggregates is common problem and has not been sought out yet. Their usage as the raw material in ceramics might be environmentally friendly way to utilize them.

This paper presents technological properties, phase composition and microstructure analysis of materials made of waste sludge from washing dolomite aggregates. Research was divided into three parts: technological properties analysis, phase composition analysis and microstructure analysis.

Samples made of waste dolomite sludge were formed in laboratory clay brick vacuum extruder and fired at 900, 1000 and 1100°C. For final materials, apparent density, open porosity, water absorption, compressive strength and durability were examined.

Results of technological research suggest the possibility of the application of the waste sludge from washing aggregates in building ceramics technology as bricks materials. Waste sludge from washing dolomite aggregates can be used as the main raw material of building ceramics masses. Without any additional technological operations (e.g. drying or grinding), the material with satisfactory properties was obtained. According to durability results all obtained materials can be used for masonry protected against water penetration and without contact with soil and ground water and also for masonry subjected to passive exposure (F0 – according to the standard EN 771-1).

**ODPAD Z PŁUKANIA KRUSZYWA DOLOMITOWEGO
JAKO SUROWIEC PODSTAWOWY W PRODUKCJI CERAMIKI BUDOWLANEJ**

Słowa kluczowe

właściwości, szlam dolomitowy, ceramika budowlana, materiały ceramiczne,
odpad z płukania kruszywa dolomitowego

Streszczenie

Ilość odpadów z płukania kruszyw dolomitowych stale wzrasta. Kruszywa płucze się w celu usunięcia zanieczyszczeń ilastych. Odpady z płukania charakteryzują się wysoką zawartością minerałów ilastych oraz węglanów. Ich właściwości zależą od charakterystyki płukanego surowca oraz zastosowanej technologii płukania. Problem utylizacji szlamów z płukania kruszyw wapiennych nie został rozwiązany do dzisiaj. Ich zagospodarowanie w ceramice budowlanej może przynieść korzyści ekologiczne i ekonomiczne.

W pracy przedstawiono właściwości technologiczne, skład fazowy i analizę mikrostruktury materiałów wykonanych z odpadu z płukania kruszywa dolomitowego. Badania podzielono na trzy części: analizę właściwości technologicznych, analizę składu fazowego i analizę mikrostruktury. Próbkę wykonaną z odpadowego szlamu dolomitowego formowano w laboratoryjnej próżniowej prasie ślimakowej i wypalono w 900, 1000 i 1100°C. Dla otrzymanych tworzyw wyznaczono: gęstość pozorną, porowatość otwartą, absorpcję wody, wytrzymałość na ściskanie oraz trwałość.

Wyniki badań technologicznych sugerują możliwość zastosowania odpadu z płukania kruszywa dolomitowego jako surowca mas w technologii wytwarzania ceramiki budowlanej. Bez zastosowania dodatkowych operacji technologicznych w przygotowaniu surowca uzyskano materiał o zadowalających właściwościach. Stwierdzono, że otrzymane tworzywa mogą być stosowane w murach zabezpieczonych przed przenikaniem wody, niemających kontaktu z glebą i wodą gruntową, a także w warunkach obojętnych (wyrobach kategorii F0 zgodnie z normą EN 771-1).