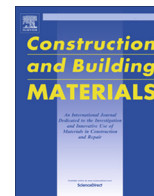




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Utilisation of wood ash from biomass for the production of ceramic products

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HIGHLIGHTS

- 5–60% of the additive of ash can be used for the production of ceramic products.
- The main elements of ash composition are C, O, Si, Ca and Fe (more than 88%).
- Additive of ash reduces shrinkage, density, heat conductivity of ceramic bodies.
- The amount 20% amount of wood ash influences of the ceramic body as natural pigment.
- Calcium pyroxene (CaFe(Si,Al)₂O₆) brightens the ceramic body.

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ABSTRACT

Utilisation of biomass as fuel is very attractive from the ecological point of view. Such way of energy extraction is ecologically clean (zero CO₂ emission) and biomass is renewable and theoretically inexhaustible local energy source. However, during the utilisation of secondary raw materials of biomass for energy production, higher amount of ash accumulates. Wood ash analysed in the research consists of two types of particles: first type – nonflammable mineral particles consisting of the following main elements: Si, Ca, Fe, O and C (86%) and the main elements of the particles of second type are C and O (98%). The aim of the research is to analyse the possibility to utilise wood ash from biomass for the production of ceramic products. Formation masses are prepared by incorporating from 5% to 60% of wood ash additive and burned at various temperatures. It is identified that this additive influences the properties of ceramic body, i.e. reduces drying and burning shrinkage, density, compressive strength, thermal conductivity, increases water absorption and porosity. Additive of wood ash from biomass influences the ceramic body as a natural pigment that brightens the ceramic body. Colour change is determined by sufficient amounts of calcium pyroxene in the composition thereof iron is inserted.

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1. Introduction

In recent years, the main topics of discussions are related to the utilisation of biomass, renewable energy sources and the utilisation of secondary materials. In other words, it is the transition from linear economy to the circular one, where all new products created could be reused several times, and the amount of waste materials left would be as least as possible. Before 2020, EU aims to produce 20% of energy from the renewable energy sources – wind, sun, waterpower, as well as biomass. The usage of renewable energy could allow EU to reduce the amount of greenhouse gases and become less reliant on the imported energy. Lithuania commits

to stimulate the constant increase of the utilisation of renewable energy sources in energetics and transport in order to reach higher than 15% of total balance of initial energy until 2020.

Vegetal biomass dedicated for energy purposes is very important source of renewable energy. Biomass resources cover not only agriculture and forestry production, but also its treatment and processing waste, i.e. waste of disforest as well as chemically untreated wood industry. When prices of natural resources grow, in the countries, where fossil fuel resources are limited, more and more attention is being paid to the biomass. It is considered that the utilisation of biomass for energy production does not increase carbon dioxide emissions to the atmosphere. By growing, all plants remove carbon dioxide from the atmosphere during photosynthesis process. When the amount of newly created biomass corresponds to the amount of biomass used to produce energy, it

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is considered that the biomass transformation to the energy process is neutral in respect to the carbon dioxide. In this respect the utilisation of biomass resources for energy production does not influence climate change, and, in addition, it is one of the ways to utilise biomass waste.

After the beginning of the utilisation of biomass waste for energy production, the cost of energy production decreases. However, high amounts of ash accumulate during the burning process. Ash of biomass cause serious environmental problems. Nowadays, in Lithuania the major part of such waste (25,000–30,000 tonnes of wood ash per year) is transported to the dump areas. Although the researches carried out show that such waste can be effectively utilised in the industry of construction products [1–7].

However, in order to utilise ash from biomass, it is essential to know and evaluate the composition of ash from biomass. This composition might depend on various factors, such as region and conditions of growth, type of soil and biomass, burning parameters and etc. Therefore, the composition might vary in a very wide range [8–11].

It is suggested to use ash from biomass for concrete production [12–14]. The researches implemented show that ash from biomass can partially replace cement during concrete production. In this way expensive binding material can be saved and cost price of such product reduced. Additive of ash from biomass influences the rheological properties of concrete mixture, concrete's physical and mechanical properties (shrinkage, water absorption, density, compressive strength), product's porosity, frost resistance and thermal conductivity.

In addition, ash from biomass can be used for the production of multi-purpose ceramic products [15–17] introduce up to 50% of ash from biomass into the formation masses. After ash introduction, the density and compressive strength of ceramic products burned at temperature of 950 °C decrease, shrinkage and water absorption increase. The authors state that the optimal amount of ash to be utilised for the production of ceramic products shall not exceed 20%. Other authors state that from 5% to 10% of ash from biomass additive decreases the density of ceramic products by 210 kg/m³, thermal conductivity up to 0.13–0.16 W/(m·K) and increases water absorption by 7%. However, these ash must be additionally rinsed before the utilisation for the production of ceramic products, thus impeding and increasing its production costs [18]. In addition, there is data confirming that the additive of ash from biomass changes colour of ceramic products [16].

During the recent years, the utilisation of biomass for energy production in Lithuania has increased considerably in both, private

and industry sectors. However, the problem of the utilisation of ash from biomass has not been solved so far. It can be assumed that waste of ash from biomass can be effectively utilised for the production of ceramic products, thus saving the expensive traditional raw materials.

The research has been carried out in order to improve knowledge as well as to estimate the possibilities of the utilisation of wood ash from biomass for the production of ceramic products. Therefore, the objective of the research is to determine the influence of wood ash on physical and mechanical properties, structural parameters and microstructure of the ceramic body burned at 950 °C and 1000 °C temperatures. In addition, it is assumed that wood ash can influence the ceramic body as natural pigment that dyes it in brighter colours.

2. Materials and methods

Low-melting illite clay obtained in bags from the factory of ceramic products is used for the research. This clay has been shredded and sieved through 0.63 mm sieve. Ash is produced after the burning of various waste materials of biofuel. It is easy to insert ash into formation mass, as well as to easily mix it with the clay. Initially, dry mixture of components is mixed and the obtained mixture is watered till the humidity reached the level suitable for the formation. This formation mass is kept for three days at (95 ± 5)% of humidity in order to achieve even distribution of water in the formation mass. After three days (70 × 70 × 70) mm sized samples are formed from formation masses. 12 specimens are prepared for each forming mixture. 6 of them are burned at 950 °C temperature and other 6 specimens – at 1000 °C temperature. Composition of formation masses (% by mass) is provided in Table 1.

Formed semi-finished products are initially dried at natural laboratory conditions and later additionally dried in an oven for 2 more days. First day, samples are dried at temperature of (60 ± 5) °C and second day – at temperature of (105 ± 5) °C. Burning of the samples is carried out at temperatures of 950 °C and 1000 °C, overall burning period is 34 h by keeping at the highest burning temperature for 4 h.

Chemical compositions of the clay and ash used in the research are determined through the classical methodologies of chemical analysis of silicate materials and by employing Oxford Instruments INCA penta FET×3.

Compressive strength of the ceramic body is determined according to LST EN 772-1:2003, net dry density based on the requirements of LST EN 772-13:2003, water absorption (Wh) in

Table 1
Formation masses.

Raw materials	Formation masses (%)					
	A0	A5	A10	A20	A40	A60
Clay	100	95	90	80	60	40
Ash	0	5	10	20	40	60

Table 2
Chemical and granulometric composition of clay, %.

Clay	Chemical composition of clay, %						
C	SO ₂	Al ₂ O ₃ + TiO ₂	Fe ₂ O ₃	CaO	MgO	K ₂ O	Loss on ignition
	48.70	18.43	8.52	6.80	2.50	4.52	10.53
	Granulometric composition of clay, %		Amount of dust fraction (0.05–0.005 mm of particles)			Clay fraction (<0.005 mm of particles)	
	Amount of sandy fraction (>0.05 mm of particles)						
	8.5		22.4			69.1	

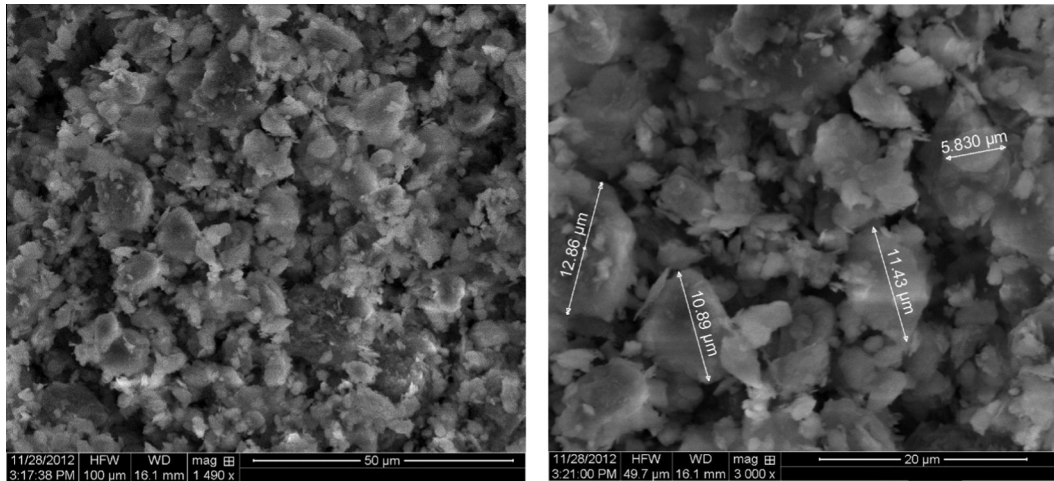
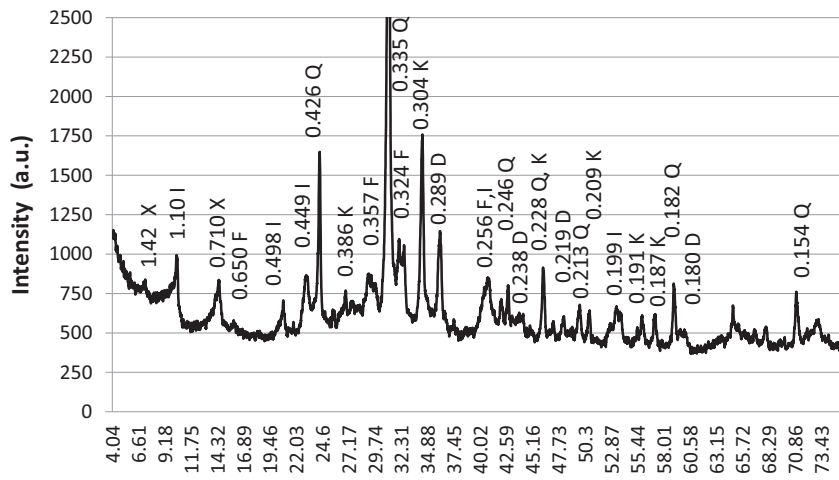
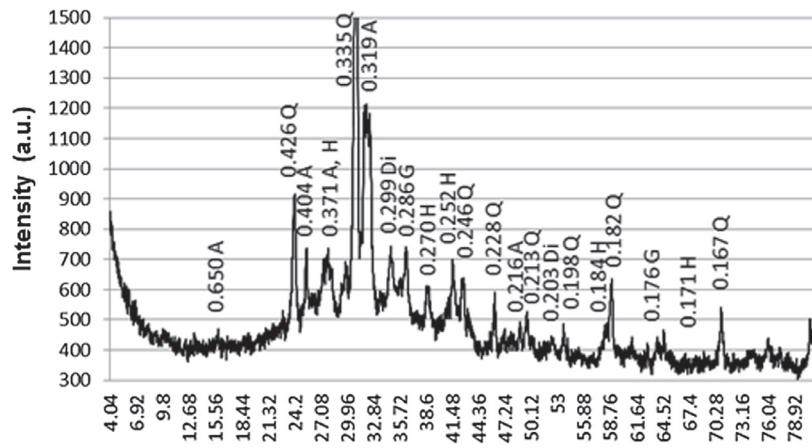


Fig. 1. SEM images of clay.



2 Theta
(a)



2 theta
(b)

Fig. 2. X-ray curve of clay (nm): a) non-burned clay – chlorite X, illite I, silica Q, feldspar F, dolomite D, calcite K; b) burned clay at temperature of 1000 °C – silica Q, anorthite A, diopside Di, hematite H.

accordance with EN 772-21:2011 and thermal conductivity by using the method of LST EN 1745:2002. Effective and total open porosities of the ceramic bodies are determined using methodology [19].

X-ray analysis is implemented by using diffraction meter DRON-7 (Co anode and Fe filter). Investigation of microstructure is carried out by employing SEM “Quanta” 250 with SE detector. Differential thermal analysis is implemented by using derivatograph Q 1500D.

3. Results and discussion

Chemical and granulometric composition of low-melting illite clay used during the research is provided in Table 2. According to the chemical composition, clay is characterised as semi-acid ($\text{Al}_2\text{O}_3 + \text{TiO}_2 = 18.43\%$) carbonaceous clay with high amount of dyeing oxides ($\text{Fe}_2\text{O}_3 = 8.52\%$). Considering the granulometric composition, clay is dispersive because the amount of particles smaller than 0.005 mm accounts for approximately 70%. Clay's

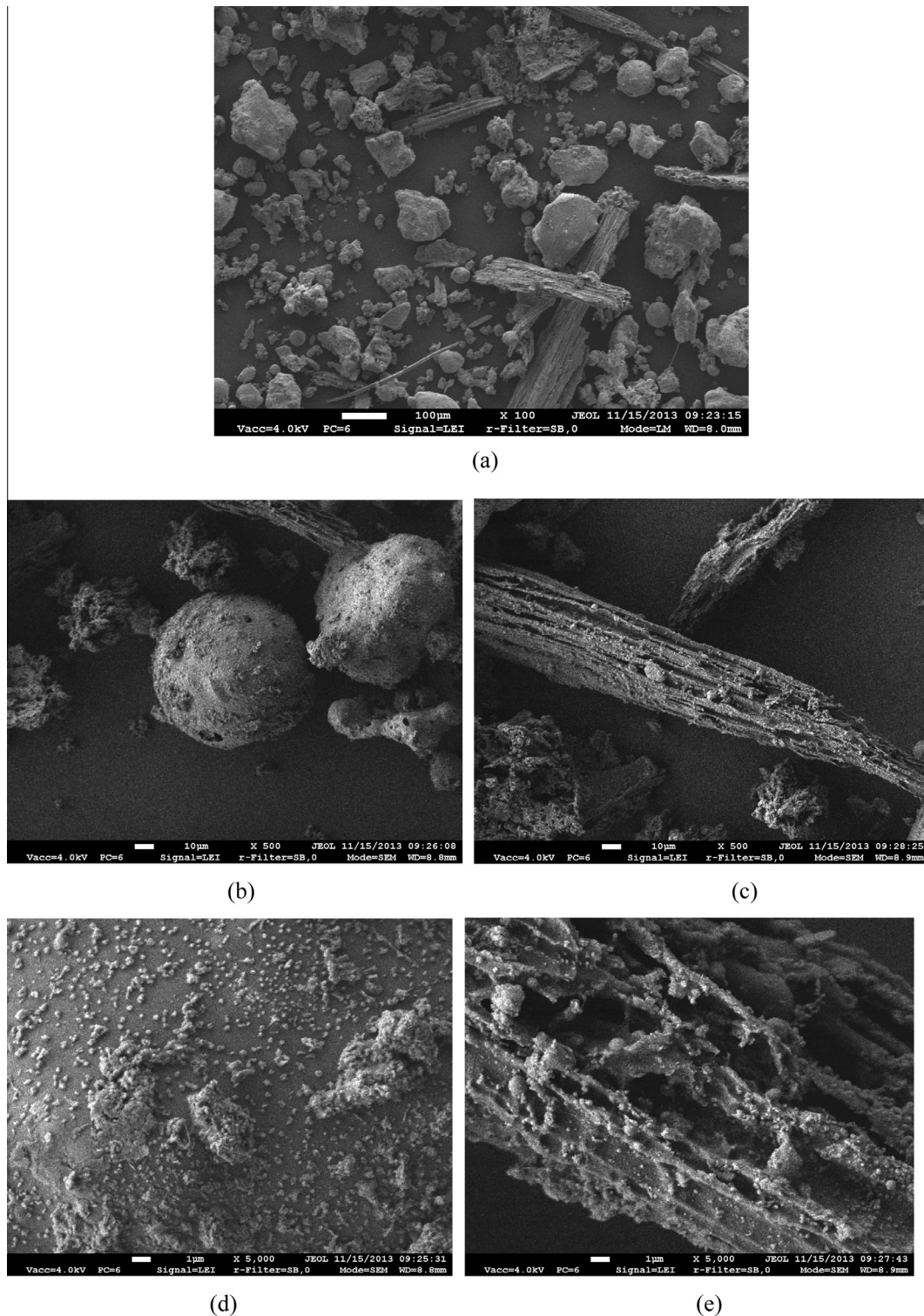


Fig. 3. SEM photo of wood ash: a) structural view of wood ash; b) and d) nonflammable mineral particles of the first type; c) and e) hollow porous particles of the second type.

plasticity is especially dependent on its granulometric composition. Finer clay is more plastic because the contact surface of particles and cohesion among them are higher. Data of clay plasticity show that clay is characterised as semi-plastic clay (plasticity index $P = 19$).

During visual investigation of microstructure, it is noticed that plate-like particles of clay distribute unevenly and do not form a solid clay matrix. Clay particles contact with other particles via thin bridges of clay particles which are also considered as coagula-

tion contacts. The size of plate-like particles varies from 5 μm to 13 μm . Among the plates of clay, fragments thereof exist (Fig. 1). Clay with such microstructure most often consists of initial minerals, such as silica, feldspar, illite and etc. This is also confirmed through X-ray analysis of the clay (Fig. 2a). It is identified that the following minerals dominate in the non-burned clay: silica, feldspar, illite, chlorite, dolomite, calcite. After the burning process of clay at temperature of 1000 °C, X-ray curve identifies the minerals as follows: silica, anorthite, diopside and hematite (Fig. 2b).

Table 3
Elemental composition of wood ash.

Raw	Elements								
	C	O.	Na	Mg	Al	Si	K	Ca	Fe
Ash (std. deviation)	5.80 (2.19)	49.21 (2.06)	2.47 (0.42)	2.11 (0.61)	3.59 (1.71)	15.65 (2.87)	3.67 (1.92)	10.28 (2.81)	7.23 (2.84)
Particles of the first type (std. deviation)	8.14 (1.38)	44.86 (4.37)	3.32 (0.66)	1.84 (0.73)	1.79 (1.01)	13.77 (5.79)	6.12 (1.48)	11.04 (2.32)	9.12 (2.56)
Particles of the second type (std. deviation)	88.84 (0.87)	9.79 (0.66)	1.21 (0.26)	0.16 (0.05)	–	–	–	–	–

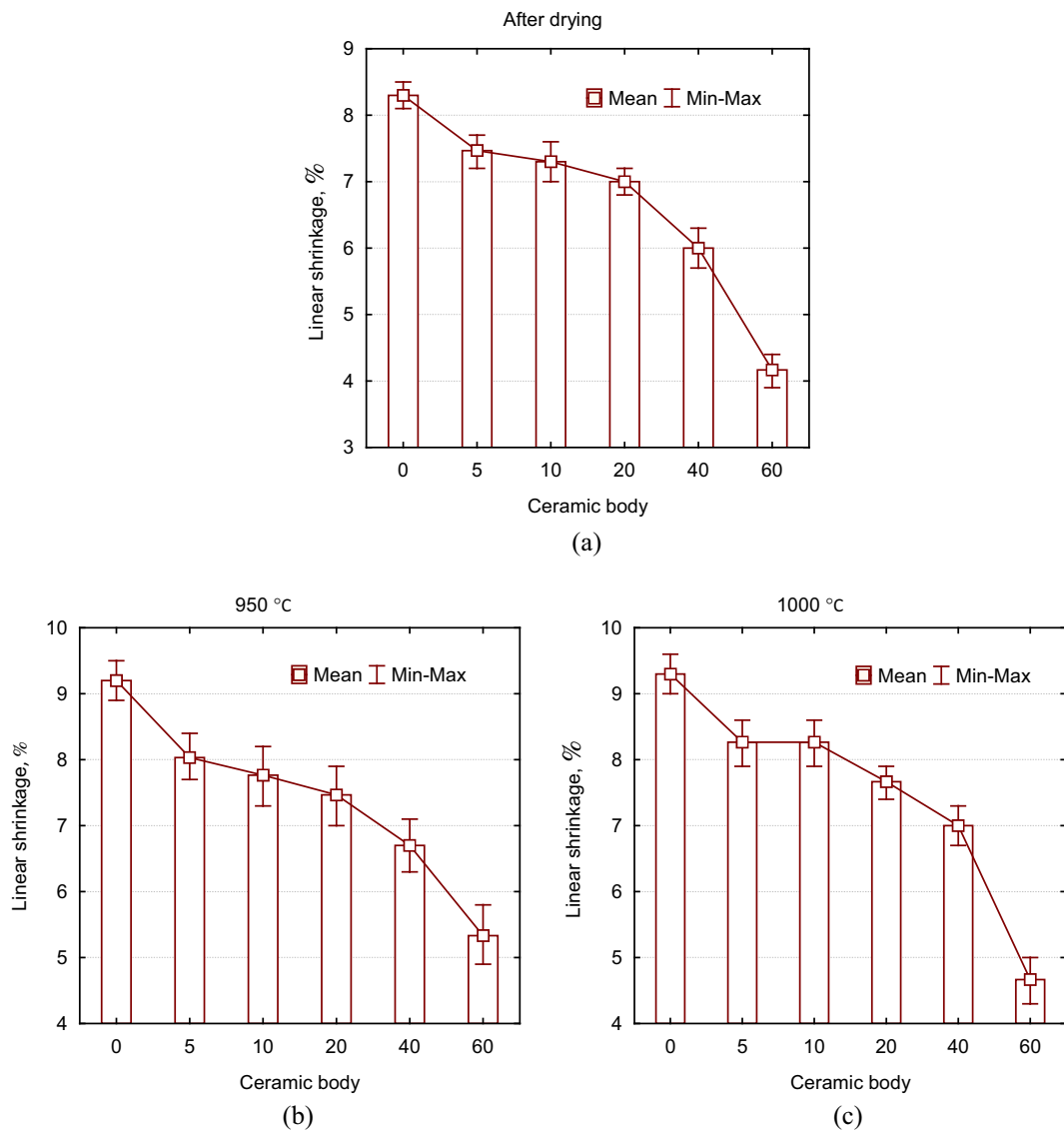


Fig. 4. Shrinkages of formation masses after drying (a) and after drying and burning at temperatures of 950 °C (b) and 1000 °C (c).

Biofuel wastes are burned in plants at temperature range of (900–1000) °C. Microscopic investigation of wood ash from biomass shows (Fig. 3a) that it consists of two types of particles. Particles of the first type consist of nonflammable mineral particles, as well as their fragments. These particles have round shape, grey colour with rounded edges and sufficiently even surface (Fig. 3b and d). Particles of the second type have oblong shape, black colour, are of different size, hollow and porous. Surface of these particles is uneven, grained and formed from a lot of external, open, fine and interconnected pores (Fig. 3 c and e). Black particles (of the second type) are carbon particles.

Bulk density of wood ash is 900 kg/m³, loss on ignition during the burning at temperature of 1000 °C is 4%. Before the use, ashes have been sieved through 0.63 mm mesh.

The examined biomass wastes are taken from the same plant. Elemental composition has been checked twice per month for 6 months. Elemental composition of the ash is shown in Table 3. As it can be seen, the main elements of ash are C, O, Si, Ca and Fe (more than 88% of overall elemental composition of the ash). In addition, Na, Mg, K elements are found. The main elements of

the particles of the first type are Si, Ca, Fe, O and C. Overall amount of these elements is higher than 86%. The main elements of the particles of the second type are C and O. Overall amount of these two elements is higher than 98%.

After X-ray analysis of the ash, two main minerals, i.e. amorphous silica (SiO₂) and calcite (CaCO₃), are identified. This is as well confirmed by investigations of other authors [20].

Results of the investigation of the physical, mechanical properties and structural parameters of the ceramic bodies burned at temperatures of 950 °C and 1000 °C are provided in Figs. 4–8.

It is identified that the amount varying from 5% to 60% of wood ash additive decreases the shrinkage of formation masses after the drying from 7.5% to 4.2%. The shrinkage of ceramic bodies with 5% of ash additive (A5 ceramic body) and burned at temperature of 950 °C decreases by 1.2%, the shrinkage of the burned one at temperature of 1000 °C decreases by 1.0% comparing with the reference ceramic body A0 burned at corresponding temperatures. When the amount of ash in the formation mass is increased up to 60% (A60 ceramic body) and

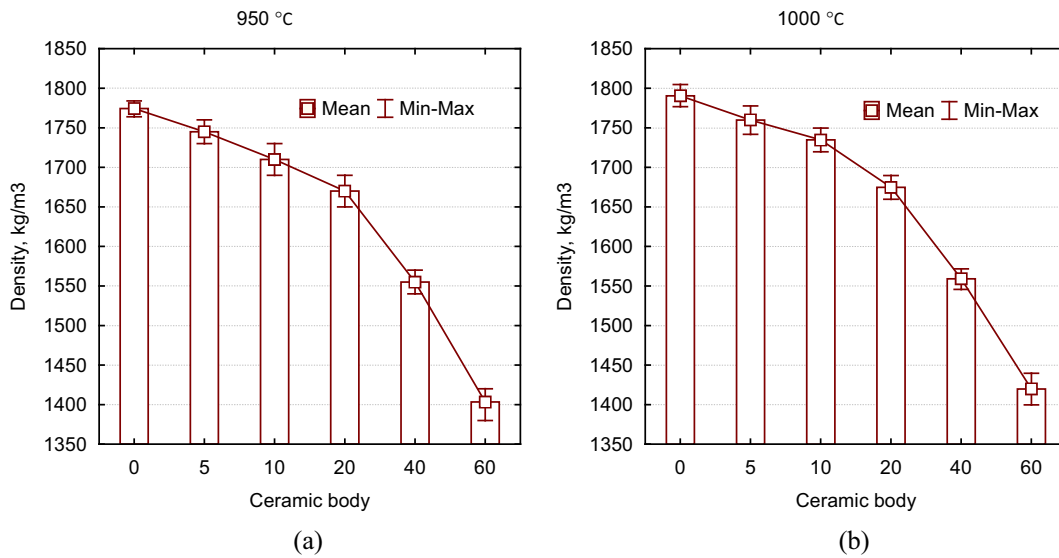


Fig. 5. Density of ceramic bodies burned at temperatures of 950 °C (a) and 1000 °C (b).

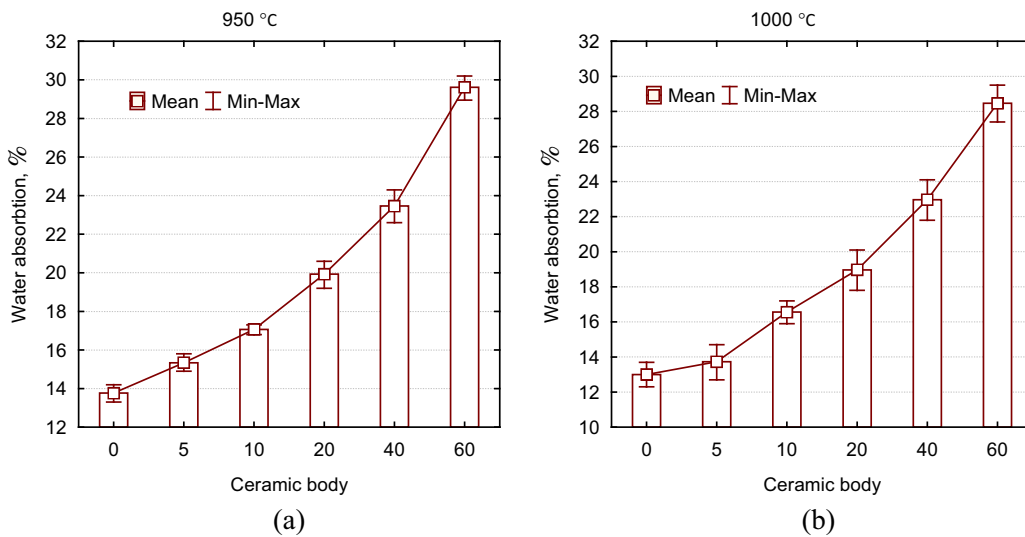


Fig. 6. Water absorption of the ceramic bodies burned at temperatures of 950 °C (a) and 1000 °C (b).

ceramic body is burned at temperature of 950 °C, the shrinkage decreases by 3.9%. When it is burned at temperature of 1000 °C, the mentioned parameter decreases by 4.6%. The shrinkage of the ceramic bodies decreases by the existence of silica particles in the ash composition. When the ash additive with the sufficient amount of silica particles is incorporated into formation masses, their plasticity decreases, and this effect influences the decrease of shrinkage after the drying and burning.

The additive analysed decreases the density of ceramic bodies. Addition of 5% of ash additive decrease the density of ceramic body burned at temperatures of 950 °C and 1000 °C by approximately 1.7%, and the maximum amount of additive, i.e. 60%, – by 20.5% compared to the reference ceramic body A0 (Fig. 5). Additionally, when the amount of ash additive increases in the formation masses, water absorption of the burned ceramic bodies increases evenly. When the highest amount of ash, i.e. 60%, is inserted, water absorption of ceramic body reaches 29.7% after the burning at temperature of 950 °C and 28.5% after the burning at temperature of

1000 °C (Fig. 6). The highest compressive strength is identified for reference ceramic body A0. After its' burning at temperature of 950 °C, the compressive strength 17 MPa, and after the burning at temperature of 1000 °C it is 20 MPa. It can be stated that wood ash additive reduces the compressive strength of ceramic bodies (Fig. 7).

Decrease in density and increase in the amount of absorbed water are determined by the general volume, dimensions and arrangement of pores formed in a ceramic body due to wood ash additive. This is confirmed by the investigations of total open porosity and microstructure analysis of ceramic bodies (Figs. 8 and 9).

Total open porosity of ceramic bodies increases with the increase in the amount of wood ash in formation masses burned at different burning temperatures (Fig. 8). Total open porosity of reference ceramic body (A0 ceramic body) is 32% after burning at temperature of 950 °C and is decreased up to 29.5% after burning at temperature of 1000 °C. The amount of 5% of wood ash slightly

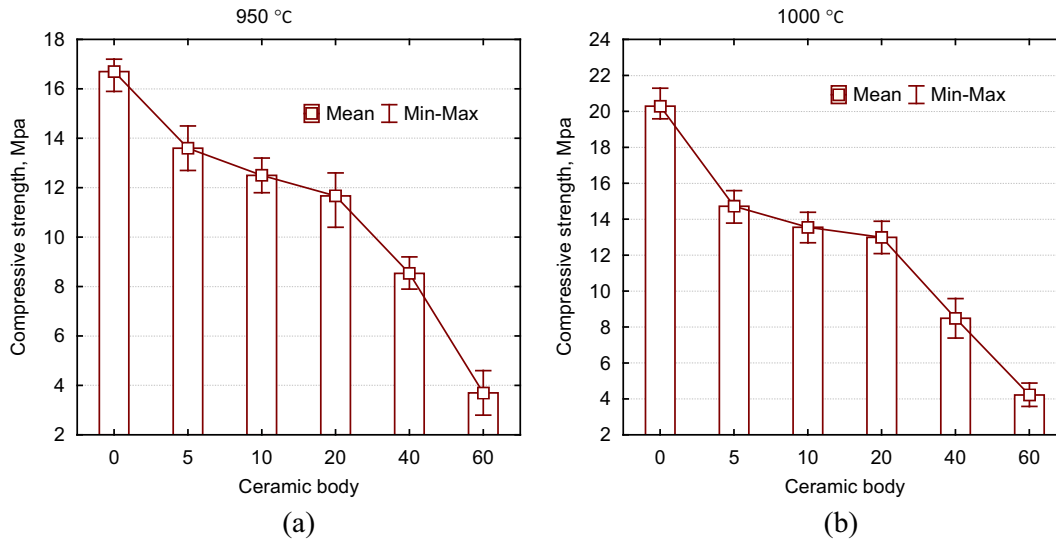


Fig. 7. Compressive strength of the ceramic bodies burned at temperatures of 950 °C (a) and 1000 °C (b).

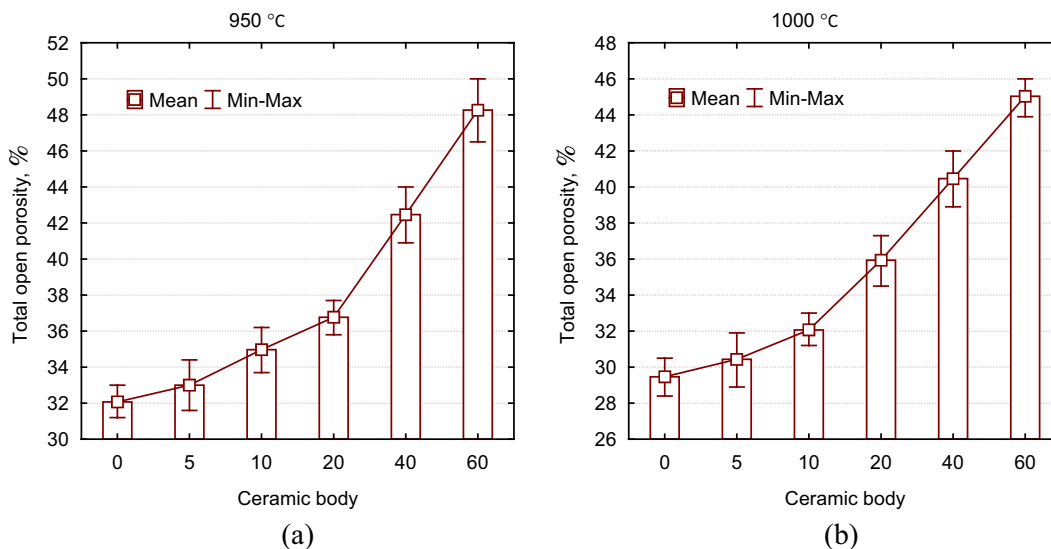


Fig. 8. Total open porosity of the ceramic bodies burned at temperatures of 950 °C (a) and 1000 °C (b).

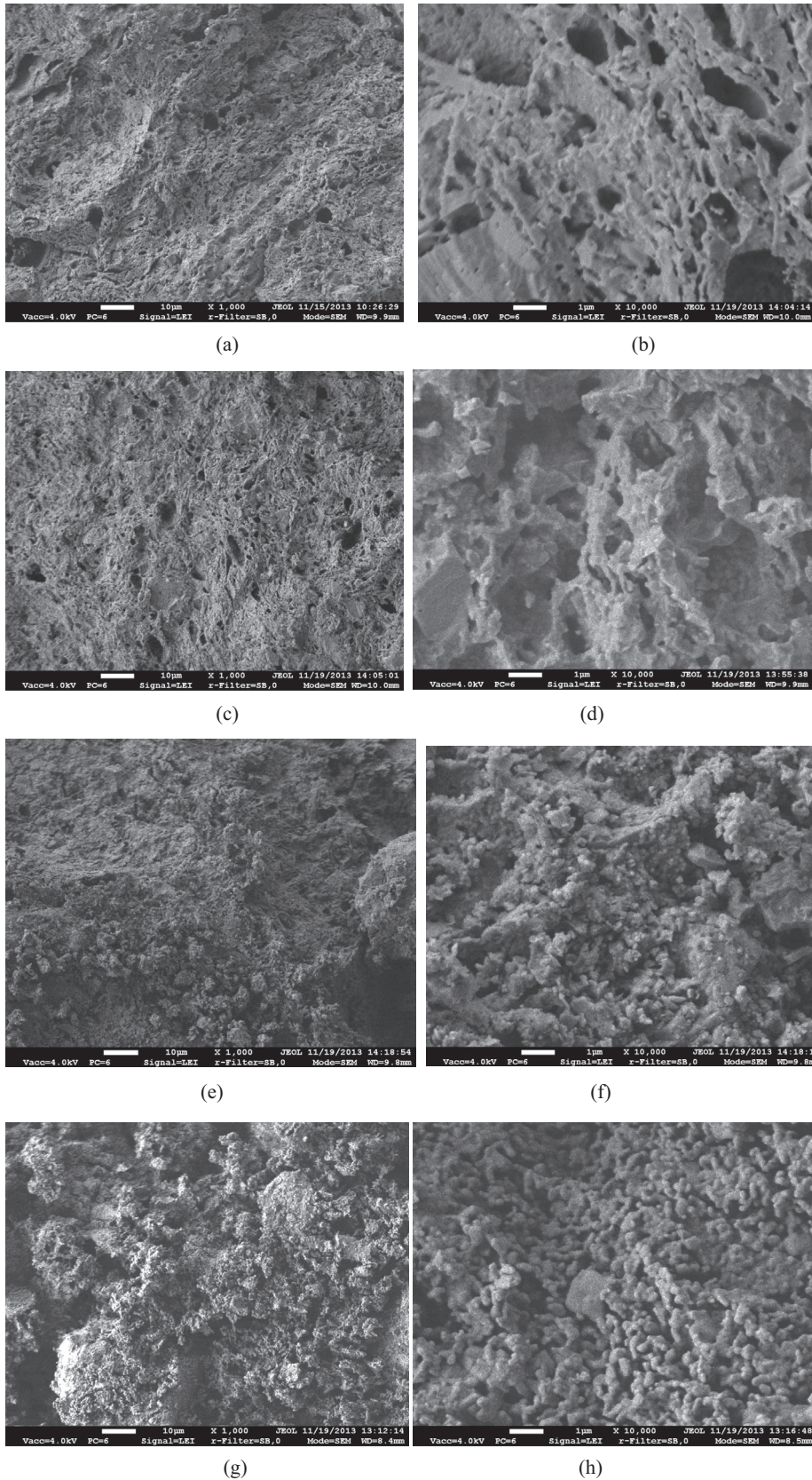






Fig. 9. SEM photo of ceramic body: reference ceramic body A0 a) and b), ceramic body A5 c) and d), ceramic body A20 e) and f), ceramic body A60 g) and h).

increases total open porosity in the ceramic body after burning at temperature of 950 °C, i.e. up to 33%, and up to 30.5% after burning at temperature of 1000 °C. The addition of 60% of wood ash (A60

ceramic body) increases total open porosity up to 48.3% after burning ceramic body at temperature of 950 °C and up to 45.2% after burning it at temperature of 1000 °C.

Table 4
Elemental composition of ceramic bodies burned at temperature of 1000 °C.

Ceramic body (colour)		Elements								
		C	O	Na	Mg	Al	Si	K	Ca	Fe
A0 	Mean (Std. deviation)	4.22 (0.19)	51.50 (1.57)	0.79 (0.09)	2.58 (1.50)	9.22 (0.69)	17.04 (0.75)	2.12 (1.3)	4.27 (1.08)	8.26 (1.31)
	A5 	Mean (Std. deviation)	4.64 (0.11)	49.89 (1.74)	0.64 (0.14)	2.20 (1.60)	9.47 (0.81)	17.78 (0.92)	2.45 (1.57)	4.99 (1.40)
A20 	Mean (Std. deviation)	5.64 (1.29)	36.61 (5.55)	0.67 (0.21)	1.62 (0.57)	10.93 (1.27)	24.50 (2.86)	4.34 (1.55)	11.12 (3.78)	4.56 (2.58)
A60 	Mean (Std. deviation)	6.09 (0.68)	37.25 (6.24)	0.31 (0.22)	0.28 (0.24)	10.4 (0.83)	25.90 (1.76)	4.41 (2.20)	14.04 (1.64)	1.32 (0.49)

When the amount of ash additive is increased in formation masses, the microstructure of ceramic body apparently becomes more porous (Fig. 9). Distribution of the pores is sufficiently even, however, the total volume of the pores and their amount increase. The slight increase in the burning temperature from 950 °C to 1000 °C has almost no influence on the microstructure of ceramic body.

SEM images of ceramic body with 5% of wood ash additive (Fig. 9c, d) burned at temperature of 1000 °C show that the microstructure has almost no difference from the one of reference ceramic body A0 (Fig. 9a and b), it is dense and has even surface. The interconnection between minerals is sufficient, there are not many spherically shaped pores, most of them are of closed nature. Total open porosity of this ceramic body is not high and it is 30% (Fig. 8), density is 1760 kg/m³ (Fig. 5) and compressive strength is 14.8 MPa (Fig. 7). When 20% of wood ash additive are incorporated into the formation mass (Fig. 9e and f), the view of microstructure changes, surface becomes uneven, higher amount of pores can be observed. Microstructure of the ceramic body with 60% of wood ash additive (Fig. 9g and h) is completely different and it can be distinguished by the porous structure. A characteristic feature of the ceramic body is high amount of small pores which are hollow with various sizes and shapes, although, their distribution in the entire ceramic body is even. Connection among minerals' particles is limited. Total open porosity of this ceramic body is higher than 40% (Fig. 8), density is approximately 1400 kg/m³ (Fig. 5) and compressive strength is higher than 4.0 MPa (Fig. 7).

During the investigation, it is noticed that higher than 10% amount of wood ash additive influences the colour of ceramic bodies (Table 4). Colour change of ceramic bodies analysed demonstrates that wood ash additive participates in and influences physical and chemical processes in ceramics during the burning process. Implemented investigation of elemental composition of the ceramic bodies burned at temperature of 1000 °C (Table 4) shows that the amounts of elements C, O, Al, Si, K, Ca increase and Fe, Na, Mg decrease when the amount of wood ash additive increases.

It is known that various red hues in ceramic products appear due to relatively high amount of iron in ceramic body. Red hues might be reduced when iron penetrates to the crystalline structure of some compounds faster than free iron oxide – hematite is formed [21,22]. In order to brighten the colour of ceramic bodies, it is possible to introduce additives with Ca compounds into the formation mixtures. Calcite itself does not brighten the red colour, therefore, it is necessary for calcite to react with clay minerals after

fragmentation, so the calcium pyroxenes could form where iron and melilite could be inserted therein [23]. These compounds are characterised by colouring features, consequently, much lighter ceramic body is formed.

In this case, when the amount of wood ash is increased in the formation mass and it is burned at temperatures of 950 °C or 1000 °C, the amount of iron in ceramic body decreases proportionally and the amount of calcium increases. After the incorporation of 5% of wood ash additive into the ceramic body, the amount of iron decreases up to 7.93% compared to reference ceramic body A0 having 8.26% of iron, and the amount of calcium increases from 4.27% to 4.99%. Colours of reference ceramic body A0 and ceramic body A5 do not differ.

20% of wood ash additive decrease the amount of iron in the ceramic body up to 4.56% and the amount of calcium up to 11.12%, resulting into the brighter ceramic body. 60% of additive decrease the amount of iron even up to 1.32% and the amount of calcium increases up to 14.04%, accordingly the colour of ceramic body becomes pale yellow. It as a result, we may be assumed that during the burning process of ceramic bodies with the amount of wood ash higher than 20%, sufficiently high amounts of calcium pyroxenes with inserted iron are formed and, as a result, they brighten the ceramic body.

The identified thermal conductivity of reference ceramic body A0 burned at temperature of 1000 °C is 0.55 W/(m·K), for ceramic body A5 it is 0.50 W/(m·K) and for ceramic body A60 it is 0.34 W/(m·K). It is supposed that if ceramic products with such properties are formed with hollows in a plant, it would be possible to decrease thermal conductivity of ceramic products much more by keeping density and compressive strength sufficiently high.

4. Conclusions

Analysed influence of wood ash additive from biomass on physical and mechanical properties and microstructure of the ceramic bodies allows the conclusion that such secondary raw material is suitable for the production of ceramic products.

The investigations carried out show that after incorporation from 5% to 60% of wood ash additive into the formation masses and burning them at temperatures of 950 °C and 1000 °C, the ceramic bodies with the density varying from 1410 kg/m³ to 1745 kg/m³, water absorption from 13.8% to 29.7%, burning shrinkage from 4.7% to 8.0%, compressive strength from 3.7 MPa to 13.6 MPa and total porosity from 30.5% to 48.3% are prepared. Additive analysed reduces shrinkage, density, compressive strength, thermal conduc-

tivity and increases water absorption as well as overall porosity of ceramic bodies.

Through the investigations, it is proved that the addition of 20% of the additive of wood ash from biomass influences the ceramic body as natural pigment that brightens the ceramic body. Colour change is determined by sufficient amount of calcium pyroxene in the composition thereof iron is inserted. Such additive of wood ash from biomass can be used as natural, economical, environmentally friendly pigment for volumetric brightening of ceramic bodies.

Additive of wood ash from biomass can be used for the production of multi-purpose ceramic products by partially or fully replacing natural sand.

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