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Effect of Chemical Composition of Clay on Physical-Mechanical Properties of Clay Paving Blocks

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Abstract: Clay paving blocks made of natural, environmentally friendly materials have their own originality and colorful authenticity, but due to the complex technological production process, they are also relatively expensive products; therefore, their environmental resistance properties are strictly defined and controlled by the standards. The physical and mechanical properties of clay paving blocks are the key factors aiming to ensure the longevity of products and their long-term success in the market. Therefore, ensuring high physical and mechanical properties of clay paving blocks have become a most crucial challenge for the manufactures. This article considers the parameters of the technological production process of clay paving blocks manufacturing and evaluates the influence of the chemical composition of three different types of clay on the physical and mechanical properties of the paving blocks. Water absorption, linear shrinkage, freeze/thaw, acid resistance, and transverse breaking load of clay paving blocks are investigated. This study reveals the importance of raw material selection in the production process of clay paving blocks and provides the concept of main quality factors of clay.

Keywords: clay paving blocks; physical-mechanical properties; clay structure; building materials

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1. Introduction

Various clays have been used as a building material in the construction industry from ancient times to the present day due to their exceptional properties and usage possibilities, durability, economy, and environmental friendliness. Clay is one of the main ingredients applied in the production of clay paving blocks. Due to its functionality, authenticity of color range, and durability, clay paving blocks have been and are one of the most attractive building materials in the small architecture sector [1–3]. The industrial use of clay minerals is quite wide. The ceramic industry uses various types of clay for pottery making and construction ceramic (bricks, tiles, etc.) [4]. Clay bricks as well as clay paving blocks are commonly used outdoors and, therefore, are exposed to various environmental factors such as humidity, cold, heat, friction, and compression. Therefore, in order to ensure the resilience of these products to the effect of environment factors, the technological process of their production is constantly monitored and improved. The results of research confirm that great attention must be paid to the quality of the raw materials, that is why scientists and staff of manufacturing companies make daily efforts to determine the optimal chemical, mineralogical, granulometric, and other compositions of the raw materials. A number of existing studies are related to the properties of bricks using various additives such as fly ash [5–7], waste glass powder [8], sugarcane bagasse ashes [9,10], sawdust, tobacco residues [11], spent grains [12], valorized sludge [13], waste micro cellulosic fiber [14], industrial slags, such as steel slag, ferrochromium slag, or granulated blast furnace slag [15], charcoal [16], agricultural waste, such as hazelnut shells [17], waste rice husk [9,18,19], or rice straw [20,21], paper sludge [22], calamine processing wastes [23], and some other industrial and agricultural wastes. Depending on the additives, the properties of clay bricks change, in particular porosity, compressive strength, water absorption, texture, soundness, color, and some others. Requirements in terms of physical-mechanical properties for clay bricks used in masonry work and clay paving blocks differ. When various additives are mixed into the clay brick mixture, the organic components burn during the firing phase and larger or smaller pores are formed in the brick structure [24]. In this way, due to the reduced density, the thermal conductivity decreases. The incorporation of relevant waste in the production of fired clay bricks can significantly change the thermal conductivity and heat resistance properties of the building material, so the building will lose less heat in winter and will not heat up too much during the summer. An undesirable additive is iron oxide, which degrades the color after firing and reduces resistance to heat [4]. The thermal resistance is increased by amount of aluminum oxide Al_2O_3 and kaolinite $Al_2Si_2O_5(OH)_4$ [25].

Some additives, such as rice husk waste, reduce linear shrinkage [26], and sugarcane bagasse ash reduces the flexural and compressive strengths at a certain percentage by mass (these additives accounted for 5 to 15 percent of the total clay mass during brick manufacturing) [27]. Such changes in properties have a negative effect on the durability and other performance of the clay paving blocks. The addition of mining and construction waste as additives to the brick clay improves the plasticity, reduces the working moisture, the shrinkage of drying and firing, and the drying sensitivity which is positive because it facilitates brick processing due to reduced costs and the risk of breakage [28]. However, these additives reduce the mechanical strength of bricks, both dry and fired. These effects are associated with the addition of degreasers, but this is not appropriate for some properties such as water absorption or porosity. As the content of fly ash and glass cullet in the clay mass and firing temperature increases, an increasing linear firing shrinkage is observed [29]. The thermal properties of bricks can be improved by introduction of grapevine twig dust and poplar dust by forming pores in the brick mud after firing [30].

Mineralizing additives such as chalk, sodium chloride NaCl, lithium carbonate Li_2CO_3, and potassium carbonate K_2CO_3 affect the color of the bricks [31]. In addition, some mineral substances not only give an interesting color but also increase the strength of the bricks, optimize water absorption and frost resistance, and reduce energy consumption during firing. Glass additives reduce brick shrinkage, porosity, and hereby improve the water absorption and strength [29,32–34]. On the other hand, the addition of biosolids increases water absorption [35,36]. Water absorption also is affected by the firing temperature [37]. At firing temperatures above 1000 °C, the volume of the clay pores decreases and, at the same time, the rate of water absorption. To prevent water ingress, the structure of clay paving blocks should be of high densification. The use of some additives such as hydroxyapatite $Ca_5(PO_4)_3(OH)$ for surface treatment of bricks increases resistance to acids [38]: dimethyl carbonate $C_3H_6O_3$—increases durability [39], or vermiculite $(Mg,Fe^{2+},Fe^{3+})_3[(Al,Si)_4O_{10}](OH)_2{\cdot}4H_2O$—increases compressive strength [40]. Bricks containing expanded perlite, natural zeolite, and ground granulated blast furnace slag have lower thermal conductivity if compared to conventional bricks and, therefore, can be used as heat insulation materials [41]. For bricks containing grated polystyrene, iron slag, and waste glass combined with small amounts of cement, the effects of aggressive environmental influence are reduced [42].

It is worth noting that although there are numerous works on the use of various additives in the production of clay bricks, there is a relatively small number of research on the mechanical and physical properties of clay paving blocks depending on the chemical composition of the clay and its plasticity due to the water content. An analysis of the literature has shown that there are very few studies revealing the technology of the clay paving blocks manufacturing process and the sequence of operations together with the technological parameters.

This research was directed towards investigating the performance of the manufacturing process of clay paving blocks made of different types of clay, evaluating the mechanical and physical parameters of the final product that would meet the requirements established by the EN 1344:2013/AC:2015 standard [43]. The parameters investigated included characteristics such as freeze/thaw resistance, acid resistance, water absorption, tensile bending strength, linear firing shrinkage after drying, and firing of the specimens.

2. Materials and Methodology

2.1. Raw Materials

Necessity to investigate the chemical composition of clays and the influence of chemical composition of clays on physical and mechanical properties of clay paving blocks arose from the need to understand which clay is the most suitable for the production of them. All raw clay samples in this study were obtained from clay deposits located in different countries and extracted with bucket chain excavators and ordinary wheel loaders. Fist clay sample was obtained from a reservoir in eastern Germany, the clay sample being yellowish in color, further (S1). Another sample was obtained from the deposit in north-eastern Estonia and was grey in color, further (S2). The third sample of clay was obtained from a clay deposit in western Lithuania and was dark brown and red in color, further (S3).

After the taking of clay samples, 100 g of each sample was separated, and the chemical composition was determined. To determine the chemical compositions of clays, all samples were dried in an electric oven at 60 °C temperature for about 12 h and after shredded to 2 mm particles. Further, the drying process of the clay samples was continued for another 6 h at the same temperature of 60 °C. Once the clay drying process was finished, the clay samples were milled to a powder and submitted for chemical analysis. The chemical composition of all clay samples was determined by applying energy dispersive x-ray spectroscopy method with an x-ray spectrometer "Spectro XLAB 2000 XRF". Samples of powdered clay before the determination of the chemical analysis are presented in Figure 1.

Figure 1. Clay samples in powder form: S1–clay raw material from a deposit in Germany, S2–clay raw material from a deposit in Estonia, S3–clay raw material from a deposit in Lithuania.

All clays were essentially composed of the same chemical oxides as Al_2O_3 (aluminum oxide), SiO_2 (silicone oxide), Fe_2O_3 (iron III oxide), TiO_2 (titanium oxide), MgO (magnesium oxide), and others; however, the amount of mentioned oxides in the clays was different. The x-ray spectroscopy results of clays S1, S2, and S3 are provided in Table 1.

Table 1. Chemical composition and plasticity of clay specimens S1, S2, and S3.

Clay	Chemical Oxides (%)								Plasticity
	SiO_2 Silicone Oxides	Al_2O_3 Aluminum Oxides	TiO_2 Titanium Oxides	Fe_2O_3 Iron III Oxides	CaO Calcium Oxides	MgO Magnesium Oxides	K_2O Potassium Oxides	Na_2O Sodium Oxides	H_2O (%)
S1	70.05	19.52	1.35	5.35	0.30	0.50	2.30	0.30	23.51
S2	62.20	19.35	2.39	6.30	1.25	2.33	5.41	0.16	18.54
S3	48.15	12.21	3.23	4.45	7.50	4.01	4.23	4.82	20.42

2.2. Experimental Methodology

The technological production process of clay paving blocks specimens was designed and developed in accordance with the requirements of the following standards: EN 17450-1:2011 requirements for production of masonry units [44]; EN 12620:2002+A1:2008 requirements for concrete aggregates [45]; SIST EN 1008:2003 requirements and test methods for water sampling and suitability [46]; ISO 3310-1:2016 requirement and test methods for sieves from metal wire mesh [47]. During the preparation of the raw material, water was added to clay mass twice: once of 12% of the clay mass before the clay maturation process and a second time during the final clay mass mixing, when the required plasticity of clay mass had to be ensured before the shaping and cutting operations. During the experiment, specimens of size 200 × 100 × 52 mm were manufactured.

All manufactured specimens of clay paving blocks were tested in accordance with requirements of EN 1344:2013/AC:2015 standard, which defines the physical and mechanical properties of clay paving blocks. According to the mentioned standard, the physical properties of the clay paving blocks were determined by performing freeze–thaw resistance and acid resistance tests. For determination of mechanical properties of specimens of clay paving blocks, the transverse braking load test was selected.

2.2.1. Linear Firing Shrinkage and Deviations

Dimensional deviations of specimens were measured by the procedures described in the standard EN 1344:2013/AC:2015. To find out the shape and size of the clay paving blocks, 10 specimens of blocks from each type of clay were randomly selected and their overall dimensions after firing were determined. The length, width, and thickness of the clay paving blocks were measured with an accuracy of 0.5 mm. Differences in the dimensions of the clay paving blocks of the same production batch must not exceed established norms.

2.2.2. Freeze/Thaw Resistance

Aiming to determine freeze/thaw resistance of clay paving blocks, selected specimens were placed in an oven and dried at 105 ± 5 °C temperature till constant mass. A constant mass of the specimens is considered when the mass of the specimens weighed over a period of 24 h does not differ by more than 0.2%. All specimens were cooled down at ambient temperature before weighing.

Once constant mass of the specimens was achieved, all specimens were immersed into the water at room temperature. Further, water temperature was raised till 80 ± 3 °C gradually, over a period of 2–5 h and then specimens remained in water for another 24 h. Later, heating of water was stopped, and the water temperature cooled down to room temperature gradually.

After determination of constant mass and water absorption, 10 specimens from each clay were assembled into the frames for freeze/thaw test. The temperature during the freezing periods was −15 ± 3 °C, the first freezing period took 6 h and other freezing periods lasted for 120 min. During the thawing process, the temperature was increased gradually from −15 ± 3 °C to 20 ± 3 °C between 15 and 20 min. After thawing, water was sprayed on the surface of specimens for 120 ± 10 s and the process was repeated.

2.2.3. Acid Resistance

The service life of the clay paving blocks may be reduced due to an influence of the aggressive media. One of such important threats to the blocks are the effects of acid. The sulfuric acid H_2SO_4, nitric acid HNO_3, and deionized water were used as reagents in the acid exposure study. At the beginning of the test, five specimens were randomly collected. Specimens were crushed to a particle size of 10–12 mm, then grained and retained through 800 µm sieve. The same process was repeated, and the specimens sifted through 500 µm sieve, followed by washing with deionized water to a clean 500 µm fraction. Clean fraction of specimens was dried at 110 °C temperature to a constant mass. A constant mass was considered, when during the drying process, the loss of mass between the two determinations during the two subsequent weightings with an interval of 24 h did not exceed 0.1%. After drying, a specimen mass of 100 ± 5 grams' mass was weighed with the accuracy of 0.01 g. The mass of the specimen was transferred to laboratorial flask and charged with 75 mL of 10% sulfuric acid and 25 mL of 10% nitric acid. Using the reagents, the samples were boiled for 60 ± 2 min by immersing the flask in a bath of hot oil. After acid treatment and boiling, the whole mass of specimens was poured on a 150 µm sieve, washed with deionized water, and dried at 110 °C temperature to a constant mass. After the drying, the specimens were weighed with the accuracy of 0.01 g and the loss of mass was calculated and recorded.

2.2.4. Tensile Bending Strength

For the transverse braking load test, 10 specimens of clay paving blocks were selected and immersed into the water for 48 h at 20 ± 5 °C according to the standard [43]. Then the clay paving blocks were removed from the water and cleaned. Prior to applying external force on the clay paving blocks, they were measured as required and all measurements were recorded. Then the clay paving blocks were placed into the test apparatus. Two ends of clay paving block were supported, and an external force was applied in the middle of the block. The external force was applied to the specimen gradually, not exceeding 5 N/mm load per 5 s, until fracture occurrence and clay paving blocks broke in half.

3. Manufacturing of Specimens

In order to determine the effect of different raw materials on the physical and mechanical properties of clay paving blocks, the specimens of clay paving blocks were produced under the same production conditions and the same production parameters. The specimens' production process covered four main steps, which are presented in Figure 2.

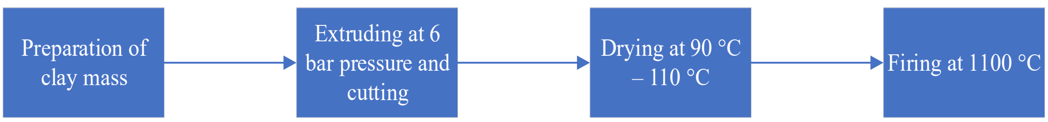

Figure 2. Production process of specimens.

The raw materials obtained from the quarries were not suitable for the production of clay paving block samples, as they were in the form of pieces of various sizes and had to be prepared according to the technological requirements.

3.1. Preparation of Clay Mass

All three different samples of clay, without mixing them together, were crushed and blended using the crushing machine. During the crushing process, additional additives such as water (12%), sand (10%), and barium carbonate (1%) were added to the clay mass.

In order for the moisture to be evenly distributed, the clay raw material must be matured. During the maturing process, the clay raw material softens and acquires plasticity. The maturation time of clay depends on various factors; however, the most important factors are the type of clay and its granulometric composition.

3.2. Extruding and Cutting of Specimens

The prepared clay mass was delivered to the clay shaping machine by belt conveyors for the shaping operation. During the shaping and extruding processes (Figure 3a,b), clay bars of size 200 × 100 mm were produced.

Specimens of clay paving blocks with a size of 200 × 100 × 52 mm made of each type of clay S1, S2, and S3 were cut by an automatic cutting machine.

(a)

(b)

Figure 3. Clay extrusion process: (**a**) extruded bar of clay S2, (**b**) extruded bar of clay S3.

3.3. Drying and Firing of Specimens

Specimens were dried in the drying chamber (Figure 4) at temperature 90–110 °C for 68 h and then delivered to the firing site. If clay paving blocks are dried improperly, the cracks and splits may appear during the firing process, thus damaging the products. After clay paving block specimens were dried, they were placed into the tunnel firing chamber (Figure 5). After preheating, the firing process was performed at 1100 °C temperature.

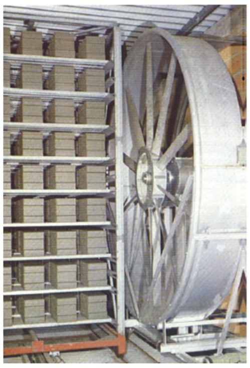

Figure 4. Clay paving blocks in drying chamber.

Figure 5. Tunnel firing chamber.

It was observed that before the clay maturation process, the same amount of water (12% by mass of the clay) that was added into the different types of clay mass, during the maturation process was absorbed differently. This is shown by the fact that the clay raw material S2 had a higher plasticity compared to the S1 and S3 raw clay. The specimens of clay paving blocks produced are shown in Figure 6.

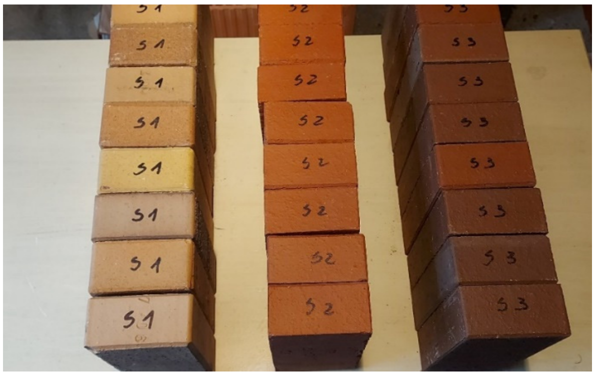

Figure 6. Specimens of clay paving blocks**.**

At the beginning of the laboratory tests to determine physical and mechanical properties of clay paving block specimens, all manufactured specimens were thoroughly visually inspected and no fissures or external damages was founded. A good quality clay paving blocks after the firing process must meet standard dimensions.

4. Results and Discussion

The physical and mechanical properties of specimens were determined by executing laboratorial tests according to the requirements of EN 1344:2013/AC:2015 standard [43]. The aim of this experiment was not only to determine the influence of chemical composition of clay on physical and mechanical properties of clay paving blocks, but also to study the technological process of production and evaluate the impact of manufacturing parameters on product properties.

4.1. Results of Linear Firing Shrinkage and Deviations Tests

The linear firing shrinkage of the clay paving blocks is caused by the loss of H_2O, which results in close movement of solid particles relative to each other. Linear shrinkage is one of the most important properties of clay paving blocks during sintering. Shrinkage and expansion were determined by measuring the dimensions of the specimens before and after firing in accordance with standard EN 1344:2013/AC:2015 [43]. The measuring results are presented in Table 2.

Table 2. Assessment of dimensional deviations.

Dimension's Deviation Test		Total Samples: 30 Units	Standard: EN 1344:2013/AC:2015	
Type of Clay	No.	Length d (mm)	Width d (mm)	Thickness d (mm)
		200	100	52
		Measuring Point: Middle of the Area		
S1	1	201.4	100.5	52.1
	2	201.0	100.6	52.2
	3	200.1	99.6	52.0
	4	201.6	100.7	51.4
	5	201.6	100.9	51.8
	6	201.7	100.9	52.4
	7	201.8	100.8	51.7
	8	201.1	100.5	52.2
	9	201.8	100.8	52.0
	10	200.1	99.5	52.1
Average value:		201	100	52
Allowed deviation value: from: d−(0.4· $\sqrt{d}$)/to: d+(0.4· $\sqrt{d}$)		194 to 206	96 to 104	49 to 55
Smallest value:		200	100	51
Greatest value:		202	101	52
Class R0		No requirement	No requirement	No requirement
Class R1 0.6· $\sqrt{d}$		8	6	4
Result:		Assigned to class	R1	
S2	1	197.2	99.0	50.2
	2	198.5	98.4	51.5
	3	200.5	99.3	50.6
	4	198.3	98.4	52.3
	5	199.2	97.9	50.3
	6	198.2	98.2	49.9
	7	198.6	97.8	50.3
	8	199.1	98.5	51.6
	9	197.8	99.1	50.8
	10	198.5	97.3	52.2
Average value:		199	98	51
Allowed deviation value: from: d−(0.4· $\sqrt{d}$)/to: d+(0.4· $\sqrt{d}$)		194 to 206	96 to 104	49 to 55
Smallest value:		197	97	50
Greatest value:		200	99	52
Class R0		No requirement	No requirement	No requirement
Class R1 0.6· $\sqrt{d}$		8	6	4
Result:		Assigned to class	R1	
S3	1	195.8	97.3	50.1
	2	196.2	96.8	50.3
	3	196.5	96.3	50.6
	4	195.1	97.1	50.0
	5	197.4	97.3	49.3
	6	196.7	96.4	49.7
	7	195.1	96.1	49.9
	8	196.4	96.3	50.6

	9	196.7	97.1	50.4
	10	196.6	96.8	49.2
Average value:		196	97	50
Allowed deviation value: from: d−(0.4· $\sqrt{d}$)/to: d+(0.4∗ $\sqrt{d}$)		194 to 206	96 to 104	49 to 55
Smallest value:		195	96	49
Greatest value:		197	97	51
Class R0		No requirement	No requirement	No requirement
Class R1 0.6∗ $\sqrt{d}$		8	6	4
Result:		Assigned to class	R1	

The results showed that all specimens are assigned to class R1 as its dimensional differences does not exceed established norms required by the standard [43]. However, despite the fact that all specimens of clay paving blocks were the same dimensions after shaping and cutting operations, it was notable that dimensions after the firing operation changed. Chart of dimensional deviations of all tested specimens is presented in Figure 7.

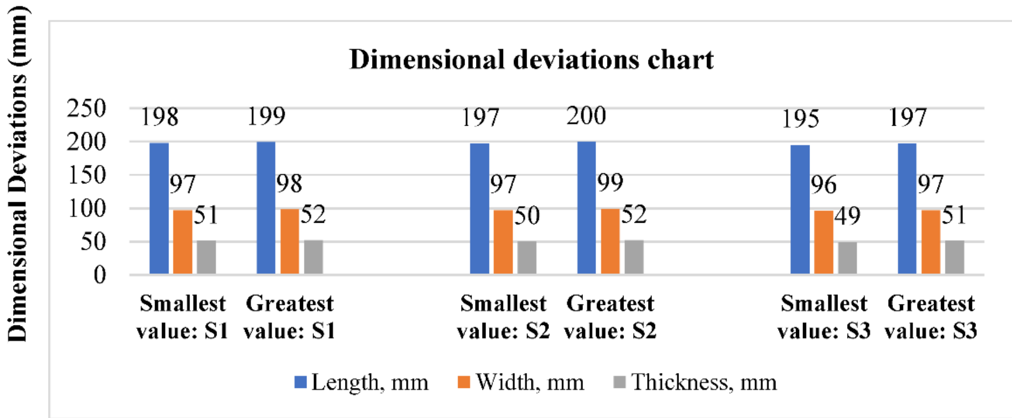

Figure 7. Results of dimensional differences and deviations.

The highest dimensional deviations were recorded in the specimens manufactured from clay type S3 and the most resistant to expansion and shrinkage were specimens from clay type S1.

4.2. Results of Freeze/Thaw Resistance Tests

Prior to freezing and thawing, the clay paving blocks were weighed, and the results obtained were recorded in order to determine water absorption. Determination of constant mass of specimens is presented in Table 3.

Table 3. Determination of constant mass.

Type of Clay	No.	Mass after Drying at 105 ± 5 (°C)			Loss of Mass < 0.2%	
		After 24 h		After 48 h		
		1 (kg)	2 (kg)	3 (kg)	After 24 h	After 24 h More
S1	1	2.308	2.300	2.300	0.35	0.0
	2	2.319	2.313	2.313	0.26	0.0
	3	2.312	2.308	2.308	0.17	0.0
	4	2.293	2.284	2.284	0.39	0.0
	5	2.301	2.292	2.292	0.39	0.0
	6	2.321	2.312	2.312	0.39	0.0
	7	2.312	2.303	2.303	0.39	0.0

	8	2.297	2.284	2.284	0.57	0.0
	9	2.305	2.296	2.296	0.39	0.0
	10	2.315	2.309	2.309	0.26	0.0
	Constancy of mass achieved				Not achieved	Achieved
S2	1	2.301	2.279	2.279	0.97	0.0
	2	2.311	2.281	2.281	1.32	0.0
	3	2.321	2.295	2.295	1.13	0.0
	4	2.305	2.286	2.286	0.83	0.0
	5	2.322	2.302	2.302	0.87	0.0
	6	2.326	2.315	2.315	0.48	0.0
	7	2.300	2.278	2.278	0.97	0.0
	8	2.304	2.295	2.295	0.39	0.0
	9	2.321	2.315	2.315	0.26	0.0
	10	2.299	2.272	2.272	1.19	0.0
	Constancy of mass achieved				Not achieved	Achieved
S3	1	2.193	2.189	2.189	0.18	0.0
	2	2.201	2.198	2.198	0.14	0.0
	3	2.185	2.183	2.183	0.09	0.0
	4	2.198	2.195	2.195	0.14	0.0
	5	2.188	2.187	2.187	0.05	0.0
	6	2.190	2.186	2.186	0.16	0.0
	7	2.205	2.201	2.201	0.18	0.0
	8	2.210	2.209	2.209	0.07	0.0
	9	2.197	2.195	2.195	0.11	0.0
	10	2.206	2.204	2.204	0.10	0.0
	Constancy of mass achieved				Achieved	Achieved

When a constant mass of the specimens was reached, a water absorption test was started. The total immersion time of specimens into the water was 48 h. Measurements were performed in accordance with the requirements of standard [43]. Results are presented in Table 4.

Table 4. Determination of water absorption.

Type of Clay	No.	Mass in Air after Immersion in Water (g)	Mass under the Water (g)	Specimen Density (kg/m³)	Specimen Mass after Drying (g)	Water Absorption (%)
S1	1	2308.2	1274.9	2.23	2300	0.36
	2	2320.8	1282.4	2.23	2313	0.34
	3	2314.7	1298.7	2.27	2308	0.29
	4	2292.0	1264.8	2.22	2284	0.35
	5	2300.0	1269.0	2.22	2292	0.35
	6	2321.5	1281.4	2.22	2312	0.41
	7	2310.6	1276.6	2.23	2303	0.33
	8	2293.0	1259.5	2.21	2284	0.39
	9	2303.8	1269.2	2.22	2296	0.34
	10	2316.4	1304.1	2.28	2309	0.32
	Average value:			2.23	0.35	
S2	1	2325.3	1273.9	2.15	2279	2.03
	2	2331.5	1280.6	2.18	2281	2.21
	3	2353.3	1285.6	2.14	2295	2.54
	4	2364.2	1275.4	2.15	2286	3.42

	5	2348.5	1284.2	2.14	2302	2.02
		2338.7	1290.9	2.13	2315	1.02
	7	2348.1	1280.7	2.14	2278	3.08
	8	2345.5	1270.2	2.12	2295	2.20
		2366.3	1283.2	2.15	2315	2.22
	10	2337.6	1286.0	2.13	2272	2.89
	Average value:			2.14	2.36	
S3	1	2356.3	1298.5	1.95	2189	7.64
	2	2361.1	1302.3	2.02	2198	7.42
	3	2335.2	1295.4	1.96	2183	6.97
	4	2388.6	1318.6	1.95	2195	8.82
	5	2345.5	1294.3	1.95	2187	7.25
	6	2335.5	1292.3	1.96	2186	6.84
	7	2362.8	1301.1	1.95	2201	7.35
	8	2375.5	1304.1	1.97	2209	7.54
	9	2361.2	1296.8	2.01	2195	7.57
	10	2375.2	1312.6	2.03	2204	7.77
	Average value:			1.98	7.52	

Water absorption directly affects the durability of clay paving blocks. As can be seen from the results of water absorption in Table 4, specimens manufactured from clay S3 can be characterized as the most moisture absorbing, because the average percentage value of difference between dry and moistened specimens was determined to be 7.52%, which is higher than maximum value of 6% established by the standard EN 1344:2013/AC:2015. Due to the determined moisture absorption value, the use of such clay paving blocks in outdoor conditions would not be recommended.

After 100 cycles of freezing and thawing, the specimens were analyzed with a special device. If defects were observed on the clay paving blocks, they were recorded. The results are presented in Table 5.

Table 5. Determination of freeze/thaw resistance.

Type of Clay	**Description of Damage**	**Type**	**No.**									
			1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
S1	Changes or damages	0	0	0	0	0	0	0	0	0	0	0
	Chalking crater	0	-	-	-	-	-	-	-	-	-	-
	Hairline crack ≤ 0.15 (mm)	0	-	-	-	-	-	-	-	-	-	-
	Crack	0	-	-	-	-	-	-	-	-	-	-
	Surface crack > 0.15 (mm)	0	-	-	-	-	-	-	-	-	-	-
	Continuous crack	0	-	-	-	-	-	-	-	-	-	-
	Chipping	0	-	-	-	-	-	-	-	-	-	-
	Shuttering	0	-	-	-	-	-	-	-	-	-	-
	Flake off	0	-	-	-	-	-	-	-	-	-	-
	Fracture	0	-	-	-	-	-	-	-	-	-	-
	Flaking	0	-	-	-	-	-	-	-	-	-	-
	Result:	Assigned to class				FP 100				Freeze/thaw resistant		
S2	Changes or damages	0	0	0	0	0	0	0	0	0	0	0
	Chalking crater	0	-	-	-	-	-	-	-	-	-	-
	Hairline crack ≤ 0.15 (mm)	0	-	-	-	-	-	-	-	-	-	-
	Crack	0	-	-	-	-	-	-	-	-	-	-

	Surface crack > 0.15 (mm)	0	-	-	-	-	-	-	-	-	-	-
	Continuous crack	0	-	-	-	-	-	-	-	-	-	-
	Chipping	0	-	-	-	-	-	-	-	-	-	-
	Shuttering	0	-	-	-	-	-	-	-	-	-	-
	Flake off	0	-	-	-	-	-	-	-	-	-	-
	Fracture	0	-	-	-	-	-	-	-	-	-	-
	Flaking	0	-	-	-	-	-	-	-	-	-	-
	Result:	Assigned to class				FP 100			Freeze/thaw resistant			
S3	Changes or damages (units)	6	0	0	0	0	0	2	0	3	1	0
	Chalking crater	1	-	-	-	-	-	-	-	-	+	-
	Hairline crack ≤ 0.15 (mm)	1	-	-	-	-	-	-	-	+	-	-
	Crack	0	-	-	-	-	-	-	-	-	-	-
	Surface crack > 0.15 (mm)	0	-	-	-	-	-	-	-	-	-	-
	Continuous crack	0	-	-	-	-	-	-	-	-	-	-
	Chipping	0	-	-	-	-	-	-	-	-	-	-
	Shuttering	0	-	-	-	-	-	-	-	-	-	
	Flake off	1	-	-	-	-	-	+	-	-	-	-
	Fracture	1	-	-	-	-	-	-	-	+	-	-
	Flaking	2	-	-	-	-	-	+	-	+	-	-
	Result:	Assigned to class				FP 0			No requirement			

(-) No changes or damages. (+) Occurrence of the violation. Clay paving blocks of class FP 100 must not show any damage of the type or higher after 100 freeze/thaw cycles.

From the results presented in Table 5, it should be noted that specimens of clay paving blocks made of clay S3 do not meet the minimum requirements of standard [43], which is 100 freezing and thawing cycles. First violations were observed after cycle 82. Later, specimens started to collapse, many cracks and fracture damages, flaking, and chipping were detected. Other specimens of clay paving blocks made from clay S1 and S2 met the requirements of the standard perfectly. Specimens made from clay S2 and S1 were assigned to class FP 100, which is resistant to freeze and thaw, and specimens made from clay S3 assigned to class FP 0 as not freeze and thaw resistant. A graphical chart of the results of freeze/thaw resistance test is presented in Figure 8.

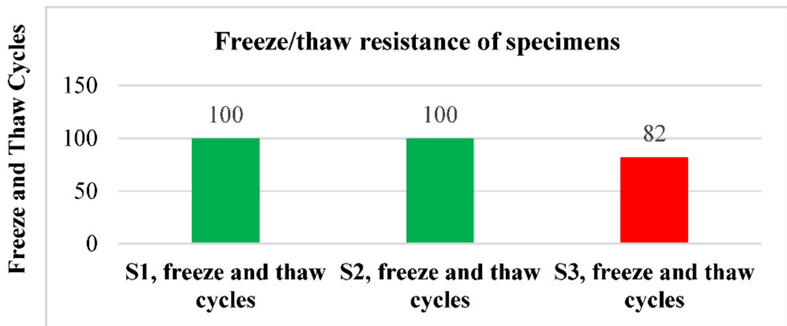

Figure 8. Results of freeze/thaw resistance.

As it was mentioned, resistance to freezing and thawing is one of the most important properties of clay paving blocks as they are designed for outdoor usage.

4.3. Results of Acid Resistance Tests

Acid resistance, as well as freeze and thaw resistance, are attributed to the physical properties of clay paving blocks. The acid resistance of clay paving blocks is very important when pavers are used outdoors during the winter, as most of the roads and yards are treated with various salts or acids to melt ice or snow. Moreover, clay paving blocks can be used near to the plants of chemical components or materials and some of them reach the surface of the pavers and cause damage. For these reasons, clay paving blocks must meet the requirements of the standard for acid resistance properties. According to the standard [43], the loss in mass after acid treatment of the specimens must not exceed 7%. The results of acid resistance are presented in Figure 9.

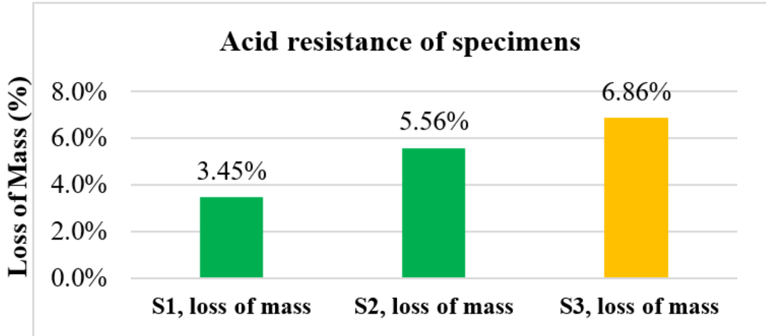

Figure 9. Results of acid resistance.

As it is visible from the results presented in Figure 9, all specimens made of clay S1, S2, and S3 met the requirements of the relevant standard.

4.4. Results of Tensile Bending Strength Tests

The transverse breaking load property is very important because during the exploitation of clay paving blocks they are affected by various external forces and have to withstand certain loads defined by the standard.

After determination of breaking loads of specimens, calculations of transverse breaking load and tensile bending strength were performed. The results are presented in Table 6.

Table 6. Results of transverse braking load.

Type of Clay	No.	Width (mm)	Thickness (mm)	Distance between End Supports (mm)	Breaking Load (N)	Transverse Breaking Load (N/mm)	Tensile Bending Strength (N/mm²)
S1	1	100.5	52.1	170	12,582	132	13
	2	100.6	52.2	170	10,306	107	10
	3	99.6	52.0	170	12,062	126	12
	4	100.7	51.4	170	12,474	133	13
	5	100.9	51.8	170	11,549	119	12
	6	100.9	52.4	170	13,088	138	13
	7	100.8	51.7	170	12,513	132	12
	8	100.5	52.2	170	12,551	133	12
	9	100.8	52.0	170	12,216	128	12
	10	99.5	52.1	170	12,967	137	13
Average value:						129	12
Minimum value:						107	10

			Assigned to class:			T4	
S2	1	99.2	50.6	170	8685	88	9
	2	98.1	51.3	170	8253	84	8
	3	99.5	50.7	170	9253	93	9
	4	98.7	52.4	170	8173	83	8
	5	97.5	50.2	170	8789	90	9
	6	98.6	49.4	170	8078	82	9
	7	97.9	50.5	170	7987	82	8
	8	98.4	51.7	170	8347	85	8
	9	99.5	50.9	170	8574	86	9
	10	97.4	52.6	170	8465	87	8
			Average value:			86	9
			Minimum value:			82	8
			Assigned to class:			T4	
S3	1	97.3	50.1	170	4568	47	5
	2	96.8	50.3	170	3864	40	4
	3	96.3	50.6	170	3989	41	4
	4	97.1	50.0	170	4130	43	4
	5	97.3	49.3	170	4677	48	5
	6	96.4	49.7	170	4324	45	5
	7	96.1	49.9	170	4257	44	5
	8	96.3	50.6	170	3745	39	4
	9	97.1	50.4	170	3961	41	4
	10	96.8	49.2	170	4337	45	5
			Average value:			43	5
			Minimum value:			39	4
			Assigned to class:			T2	

The results in Table 6 show that specimens of clay paving blocks manufactured from clay types S1 and S2 were assigned to class T4, as the calculated average value of the transverse breaking load is higher than 80 N/mm and the minimum value is higher than 65 N/mm. However, specimens of clay paving blocks made of clay type S3 were assigned to class T2, as the average value of the transverse breaking load is set at 43 N/mm and the minimum value is 39 N/mm. After identification of the values of breaking loads of the specimens, the transverse braking loads and tensile bending strength were calculated according to the standard [43]. The results show that insufficient silicon and aluminum oxides content reduces the strength of the blocks. A column chart of the results of specimens' transverse breaking loads values is presented in Figure 10.

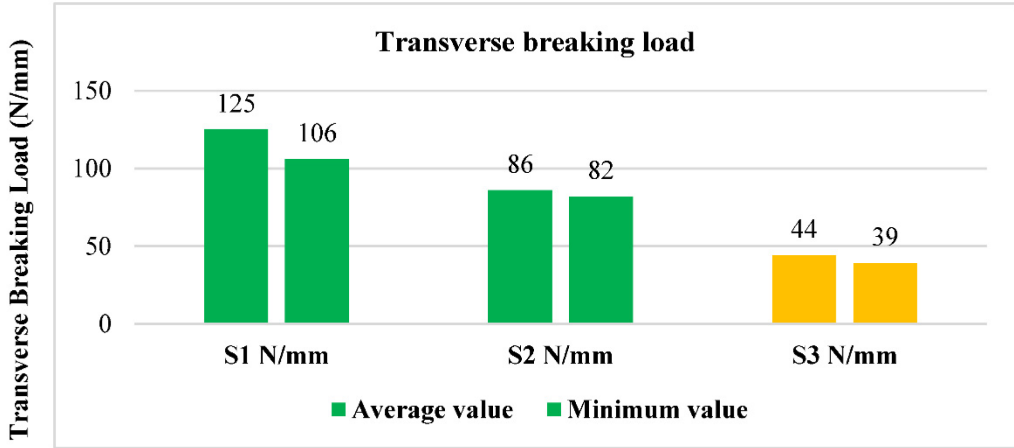

Figure 10. Results of transverse braking load.

From the results presented in the column chart, it is visible that all specimens of clay paving blocks are within the range of the requirements of the standard [43].

4.5. Summary of Results

The main goal of this experiment was to determine how different parameters of technological production process correlate with each other and how they affect the final physical and mechanical properties of clay paving blocks. As it is observed during the specimens' production process, there are many production parameters that affect the quality of the final products. As the experimental results have shown, one of the most important factors is the raw material. The results in Table 7 show that the specimens manufactured from clay S1 and S2, which contained higher amounts of silicon oxides and aluminum oxides, had better physical and mechanical properties.

Table 7. Summary of experiment results.

Summary of Results	Content		
Chemical oxide	Clay type S1	Clay type S2	Clay type S3
SiO_2	High	7.85% lower	21.95% lower
Al_2O_3	High	0.17% lower	7.31% lower
		Properties	
Freeze and thaw resistance	High resistance	High resistance	18.0% lower from minimum value
Acid resistance	High resistance	61.15% higher in loss of mass	98.8% higher in loss of mass
Transverse breaking load [N/mm]	High	31.2% lower	65.6% lower

However, in order to confirm that lower values of the physical and mechanical properties of the specimens are always caused by the lower content of silicon oxides and aluminum oxides, additional studies are necessary, as this may be due to poor selection of other technological process parameters, e.g., incorrect selection of the compression force of the material during the extrusion-shaping operation, improper firing temperature and atmosphere in the firing chamber, improper plasticity of the raw material. When using different clays, the particle size, melting point, and plasticity of the clays differ, so for these reasons it is always necessary to determine the optimal parameters of technological production process. Based on the production methodology of clay paving blocks specimens and the obtained results of physical and mechanical properties of the specimens, it can be presumed that positive correlations of technological process parameters were found for S1 and S2 type clay specimens, as for those clay specimens, paving blocks had physical and mechanical properties, which fully comply with the requirements of the EN 1344:2013/AC:2015 standard. Specimens of clay paving blocks S1 and S2, manufactured according to the production methodology and under parameters of technological process presented in Figure 11, withstood 100 freeze and thaw cycles, were resistant to acids, and their transverse breaking load values and dimensional deviations did not exceed the limits specified in the relevant standard.

The structure of clay raw materials often differs, so the same parameters of technological production process may not be the optimal solution. The suitability of the raw material must be determined before starting the production process. The structure of clay, the melting point, the particle size and plasticity of the clay, and the firing temperature are essential factors that directly affect the physical and mechanical properties of clay paving blocks.

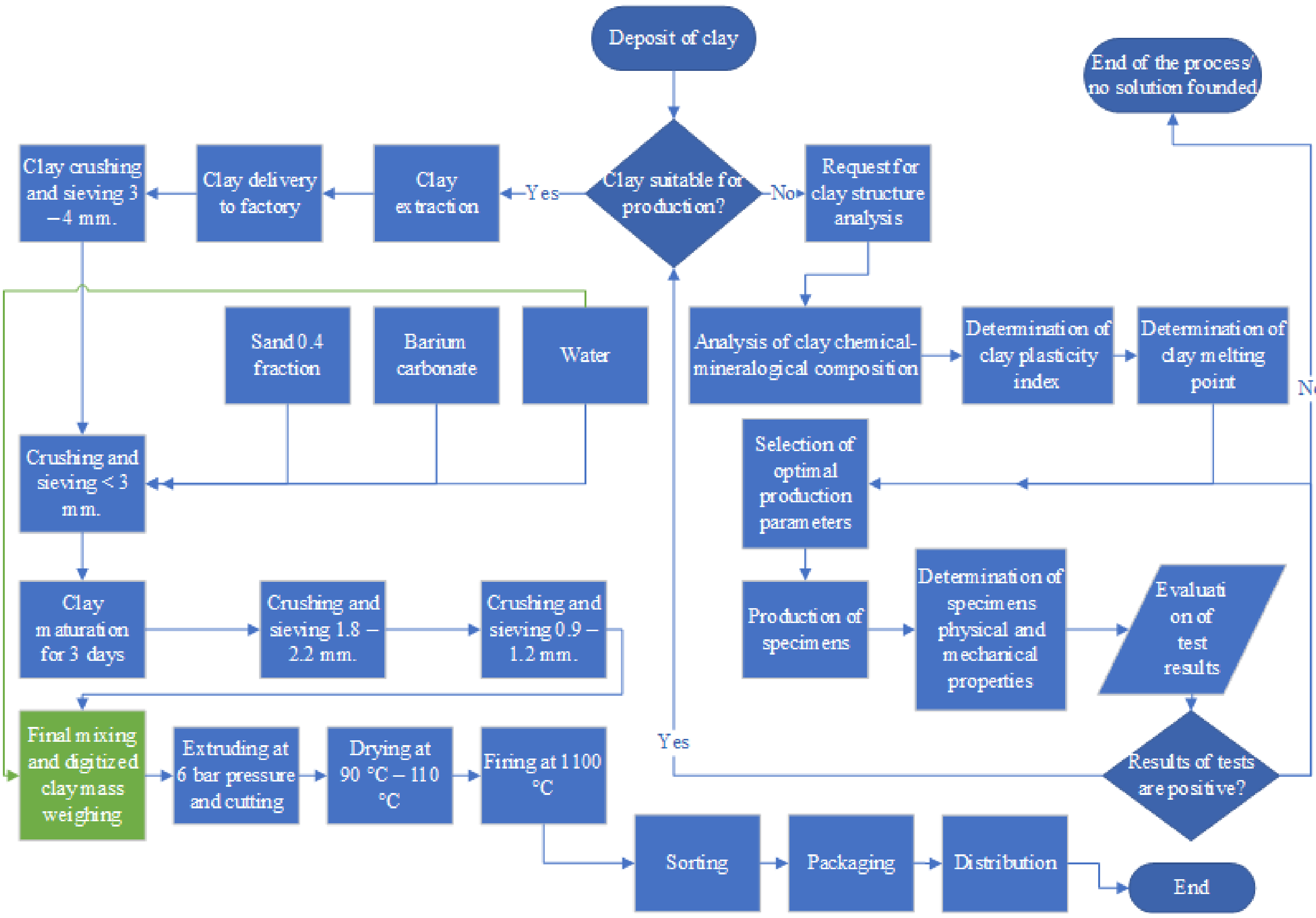

Figure 11. Proposed technological production process of clay paving blocks.

The experiment analysis showed that accurate determination of the clay mass in the clay paving block production process is vital to calculate the additional amount of water that needs to be added to obtain optimal clay plasticity during the final clay mixing operation. As shown in Figure 11 (green square), it is recommended to install digitized clay mass scalers in the final mixing hopper to determine the clay mass. Based on the exact clay mass obtained, the computer software program based on programmed logarithms automatically calculates the required amount of water to be added to the clay mass before extruding.

The modernized technological production process of clay paving blocks covers all stages of clay pavers manufacturing and specifies all the necessary parameters of technological process required for the production of high-quality clay paving blocks.

5. Conclusions

The main conclusions have been drawn based on the research results.

The x-ray spectroscopy results showed that all three clay samples in powder form consisted predominantly of silicone, aluminum, titanium, iron III, calcium, magnesium, potassium, and sodium oxides. From a mineralogical point of view, silicone and aluminum oxides are the main constituents, while other oxides are present as minor constituents.

Although the manufacturing fundamentals of clay paving blocks remain unchanged over time, some changes in the technological process have led to an overall improvement in product quality. The properties of the different clays resulted in different water absorption. It was determined that in order to provide required plasticity to some clays, it is necessary to add an additional amount of water just before the shaping operation, during the final clay mass mixing process. It was proven that specimens manufactured from clay type S3 were most water absorbing, as water absorption percentage was determined of 7.52%. Water absorption for specimens manufactured from clay types S1 and S2 was determined of 0.35% and 2.36%, respectively.

Based on the results of experiments, it can be stated that the performance of clay paving blocks can be improved using waste material additives such as silicon oxides and aluminum oxides. This could be a cost-effective solution to increase the durability of outdoor clay paving blocks. The results of the freeze and thaw tests showed that the specimens made of clay S1 and S2, which contain more chemical oxides SiO_2 and Al_2O_3, were 21.95% more resistant to freezing and thawing compared to the specimens made of clay S3. Specimens made of S1 and S2 types of clay withstood 100 freeze-thaw cycles; however, the violations on specimens made of clay type S3 were noted after 82 freeze/thaw cycles.

The acid resistance test showed that the mass loss of the clay paving blocks made of S1 type clay with the highest acid resistance properties after the acid resistance test was 3.45%. Specimens made from type S2 clay showed a loss in mass of 5.56% and type S3 clay a loss in mass of 6.86%. All the samples met the minimum requirement of EN 1344: 2013/AC: 2015, which is 7%.

The results of the transverse breaking load test confirmed that specimens made of clay type S1 could withstand higher loads compared to the specimens made of clay types S2 and S3 due to higher content of silicon and aluminum oxides and less of calcium oxides. The transverse braking load values of the S2 and S3 clay specimens were lower by 31.2% and 65.5% respectively, if compared to the S1 clay specimens.

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