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The impact of primary sludge from paper industry on the properties of hardened cement paste and mortar

Jurgita Malaiskiene *, Olga Kizinievic, Viktor Kizinievic, Renata Boris

Vilnius Gediminas Technical University, Institute of Building Materials, Laboratory of Composite Materials, Lithuania

HIGHLIGHTS

- Increasing content of PS in cement paste, the process of hydration is retarded.
- Increasing content of PS, density of mortar and the cement content were decreasing.
- PS waste may be used for mortar by replacing up to 5% of cement or fine filler.

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ABSTRACT

In the paper, the impact of paper production waste (primary sludge, or PS) on the properties of cement pastes and mortars is discussed upon. Some specimens were formed with fine PS fraction that replaced a part of cement content in them (5%, 10%, 15%, or 20%); in other specimens, coarse fraction of sludge (2/4) was used and a part of content of fine filler (sand) was replaced by PS. The properties of mixtures, such as water content required for cement paste of normal consistence, the setting time, exothermic temperature, flow diameter and plunger penetration, were established. In addition, the properties of hardened mortar, such as its density, flexural strength and compressive strength, were established. The microstructure of hardened specimens was examined as well. It was found that PS retarded hydration processes and reduced exothermic temperature; in addition, it reduced the flow diameter and the plunger penetration. While assessing the properties of hardened mortar, it may be stated that 5% PS waste might be used for replacement of a part of cement content.

1. Introduction

In course of paper production, different kinds of waste are formed in each phase of production. Most frequently, waste from paper production is divided into 4 parts: rejects, deinking sludge, primary sludge and secondary (or biological) sludge [1]. The waste from paper production is mostly presented by primary sludge. Because paper production industry is developed world widely, very large quantities of the said waste are generated. "This sludge is generated in the clarification of process water by kidney treatments, e. g., dissolved air flotation. The sludge consists of mostly fines and fillers depending on the recovered paper being processed and it is relatively easy to dewater" [2].

Primary sludge distinguishes itself for high humidity and high content of cellulose as well as variable chemical composition [3]. In the work [4], chemical and mineral composition of dried primary sludge waste is presented: cellulose ($C_{12}H_{20}O_{10}$), calcite ($CaCO_3$), muscovite $(Al_2O_3)_3(SiO_2)_6K_2O(H_2O)_2$, talc $(Mg_3Si_4O_{10}(OH)_2$, kaolinite $Al_2O_32SiO_22H_2O$, and quartz SiO_2. It was established that waste from paper production is classified as non-hazardous waste [5–8].

This waste may be used for production of various building materials, such as cement, ceramics, zeolite synthesis, waterproofing, lightweight matrices et cetera. It was found that primary sludge activated at a high temperature (500–900 °C for 2–5 h) distinguishes for high pozzolanic properties, and kaolin presented in it transforms into metakaolin [4,9]. It was established that if a part of cement content (10–20%) is replaced by the said thermally processed waste, the compressive strength remains practically the same; however, frost resistance of hardened cement mortar increases considerably [10]. The authors [11] found that paper production waste (primary sludge) might be used for cement production. The scientists [12,13] established that if a part of cement content in mortars is replaced by primary sludge ash, the compressive strength of hardened mortar decreases after 28 days; however,

* Corresponding author.
E-mail address: jurgita.malaiskiene@vgtu.lt (J. Malaiskiene).

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it begins increasing again after 90 days. The increasing starts when 10% the waste is used [13]; in addition, the authors [14] found that if 10% of waste (where the content of the predominating oxide CaO is 46.2% and the loss of ignition (L.O.I.) – 38.5%) is added to the mortar instead of cement, the strength remains the same as of the reference specimen; however, waste utilization takes place. The same authors also established the impact of primary sludge waste on the strength of hardened concrete. It was found that the strength had decreased in all the cases: in case of 10% content of waste, the strength decreased approximately by 20% and in case of 20% content of waste – about 35%.

It was established that if PS waste has been held at 650 °C for 2 h, it may be used in construction industry [15].

Scientists from India [16] used paper sludge ash (primary sludge after thermal processing at a high temperature) in production of concrete replacing the said waste 5%, 10%, 15%, 20% content of cement. The predominating chemical elements of waste included: Si – 60.6%, Ca – 14.9% and O – 15.8%. In course of the research, it was found that increasing the waste content in the mortar required higher content of water; in addition, the slump decreases, the density becomes less, the water infiltration rate grows and the compressive strength, when 5% of cement content is replaced by the waste, increases by about 15%. However, on further increasing the waste content, the compressive strength was decreasing. In addition, the impact of such a waste on the properties of wood-wool cement boards had been examined [17] and 5–20% content of waste was recommended for use.

In their paper, the authors from UK [18] provided the following chemical composition (the predominating oxides include: CaO – 61.2%, SiO_2 – 21.2% and Al_2O_3 – 12.6%) and mineral composition (gehlenite, calcite, lime and mayenite) of primary sludge ash. The said scientists replaced 2%, 4%, 6%, 8%, or 16% of cement content with the above-mentioned waste and found that the density and the compressive strength of hardened concrete were decreasing with increasing the waste content in the mortar; however, water tightness of concrete increases dramatically.

The scientists [19] investigated opportunities of using paper production waste in asphalt production and found that ash obtained of primary sludge might be used for cold asphalt production; however, dregs because of their properties did not show any positive changes of the properties of asphalt.

The scientists [20,21] investigated opportunities of using paper production waste in production of ceramics. It was found that upon using paper production waste where the predominating oxide of chemical composition is CaO (approximately from 40% to 56%), and L.O.I. is from 41% to 46%, it would be possible to produce ceramic bricks and such a production would be attractive both in the technological and the environmental aspects, because the produced bricks distinguish for improved mechanical properties; in addition, use of natural resources (particularly, clay) is reduced and waste processing takes place [20]. Other authors [21,22] established that paper production waste might be used in production of ceramic bricks, thus saving clay resources; however, such ceramic bricks distinguish for lower compressive strength, higher porosity and higher water infiltration rate. When porosity increases and the density decreases, the thermal conductivity coefficient decreases as well. So, the said waste may be used in production of energy-efficient blocks.

In the research [23], it was investigated whether paper production waste was usable in gypsum products. Different compositions and different mixture preparation ways (i.e. using dried waste or using waste mixed with water) were chosen. The best results were obtained when dried waste was used; first of all, the dry waste was mixed and the water was added.

Analysis of literature shows that the most researches were performed with PS waste, which was burned at higher than 500 °C temperature and were analysed its impact on cement, ceramics, zeolite synthesis, waterproofing, lightweight matrices and et cetera properties.

The goal of our work was to examine the impact of paper production waste (primary sludge), which hasn‘t been heated at high temperature, on the physical and mechanical properties of cement mortar and to analyse possibilities of utilization of PS waste in cement mortar.

2. Materials

In the research, cement CEM I 42.5 R was used; its chemical composition is presented in Table 1.

Mineral composition of the cement: C_3S – 62.5%, C_2S – 16.9%, C_3A – 7.1%, C_4AF – 11.5% and 2% others (alkaline sulphates and CaO). The fine aggregate – sand conforms to the requirements of EN 12620. Fraction 0/2. Granulometric composition of sand is presented in Fig. 1.

It was determined, that PS bulk density of 0/0.63 fraction is 420 kg/m^3, and the density of coarse fraction 2/4–390 kg/m^3. According to the research methodology provided in [24], it was found that PS was an active additive of fraction 0/0.63. After seven days of hardening with lime in air and three days of soaking in water, the specimens remained hardened. In the beginning, PS was dried at the temperature of 75 °C for 48 h in laboratory stove SNOL, then crumbled by a gill crusher and screened through a sieve of an appropriate fineness. The waste was put in the mixture in a form of dry powder. The photo of PS is provided in Fig. 2.

In the paper, crumbled fraction 4/8, fraction 2/4 and fraction 0/0.63 of paper production waste are discussed upon.

In one case, a part of cement content in cement mortars was replaced with the waste (the mark C) of the fraction 0/0.63, while in the other case, a part of sand content was replaced with coarser waste, fraction 2/4 (mark S). While choosing the compositions, the results of investigations carried out by other scientists, namely, that the maximum content of PS waste can be 20%, were taken into account. The compositions of cement mixtures are provided in the

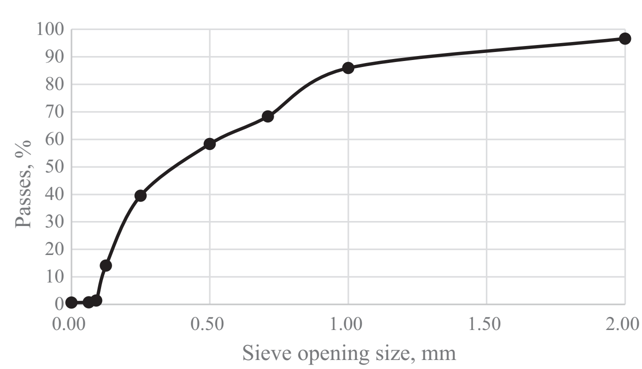

Fig. 1. Granulometric composition of sand.

Table 1
Chemical composition of cement.

CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	K_2O	Na_2O	SO_3	Cl	L.O.I.
63.2	20.4	4.0	3.6	2.4	0.9	0.2	3.1	0.05	2.15

Fig. 2. Photo of different PS fractions.

Table 2
The compositions of cement mixtures.

Batch	PS,%	PS, kg	Cement, kg	Water, kg	Superplasticizer, kg	Sand 0/2, kg
K	0	0	586	456	5.86	1758
Replaced a part of cement						
C1	5	29	557	456	5.86	1758
C2	10	59	527	456	5.86	1758
C3	15	88	498	456	5.86	1758
C4	20	117	469	456	5.86	1758
Replaced a part of sand						
S1	5	88	586	456	5.86	1670
S2	10	176	586	456	5.86	1582
S3	15	264	651	521	6.51	1494
S4	20	352	651	521	6.51	1406

Table 2. During mortar preparation, first of all, dry raw materials (PS waste was used in a form of dry powder as well), then 80% of the required water content was added and after it – the superplasticizer and the remained water were added.

3. Methods

The water content for cement paste of the standard consistence and the setting time were established according to the standard EN 196-3 (2017). The consistence of prepared mortars was established according to the flow diameter established according to EN 1015-3 and the plunger penetration established according to EN 1015-4. The specimens were kept in forms under a film for one day and in water for 27 days. The density of the specimens was established according to EN 772-13. The flexural strength and the compressive strength were established according to EN 196-1.

For exothermal tests of mixtures, the specimens were prepared as follows: cement mortar mixtures (the total weight 900 g) were poured into disposable plastic cups and a thermocouple in a glass pipe was immersed in the cup. The prepared mixture with the form was immediately placed into a metal box with internal 50 mm thick foam plastic insulation. Four mixtures with different contents of waste were simultaneously tested. Temperature was measured and its values were uninterruptedly fixed until heat emission processes took place in the specimen. The exothermal tests were carried out at the room temperature (20 ± 1) °C, the temperature of the used raw materials and water was the same, i.e. (20 ± 1) °C. This test was provided in semi-adiabatic conditions.

For an analysis of the microstructure, scanning electron microscope JEOL JSM-7600F was used. Its resolution was 1.5 nm, magnification – from 25 to 10,000,00 times; voltage of 10.0 kV was used in the tests; the surface of specimens under testing was covered with gold.

X-ray diffraction analysis was carried out by diffractometer DRON-7. The X-ray pictures were encoded according to the reference tables provided in literature where the values of the distance d between planes and the values of relative intensity I were assessed.

A statistical analysis of the researched indicators was carried out as per literature [25–27]. Regression analysis of the lowest squares was used to process the data.

4. Analysis of the results

The X-ray image of waste from paper production is provided in Fig. 3 and its microstructure – in Fig. 4. It can be seen from the X-ray image that the principal components of PS waste are calcite and cellulose. The same is shown by SEM analysis of PS waste. Moreover, it can be seen from the Fig. 4 that PS waste includes abundant open pores of various sizes (up to 10–15 μm) and capillaries that may absorb water.

The impact of PS on the water content required for obtaining cement paste of standard consistence was examined. The results of the research are provided in Fig. 5.

It can be seen from Fig. 5 that the used waste considerably increases the need in water for obtaining cement paste of the stan-

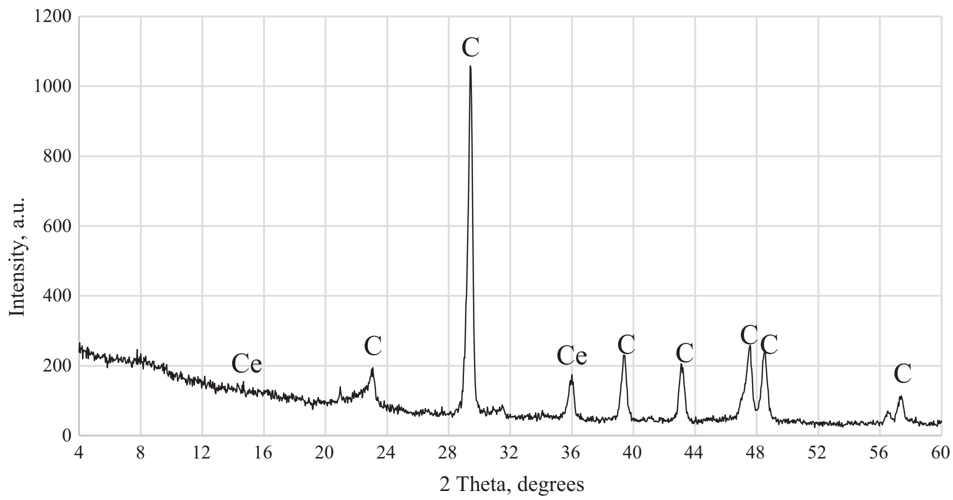

Fig. 3. X-ray diffractometric analysis of the paper sludge: C – calcite, Ce – cellulose.

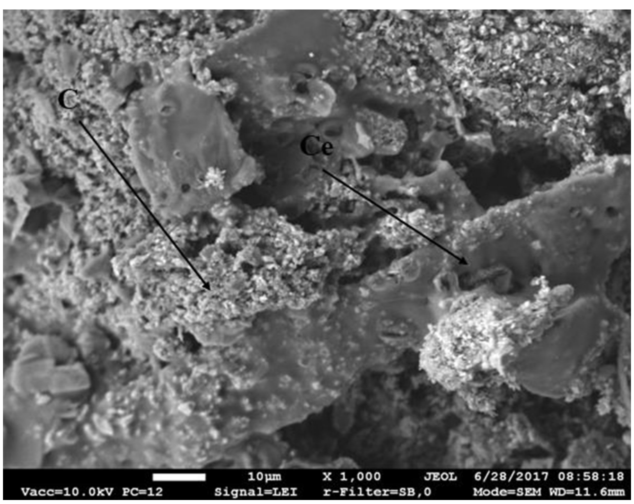

Fig. 4. Microstructure of PS waste (C – calcite, Ce – cellulose).

dard consistence, as compared to the reference specimen: when 20% of waste was used, the need in water increased by about 40%. It may be explained as follows: the dry waste put in the mixture distinguishes itself for high porosity, so it absorbs water rapidly. The waste causes linear increase of the need in water for obtaining cement paste of the standard consistence. In the provided empirical equation, the plus signs before variables show the positive correlation, i.e. the need in water for obtaining cement paste of the standard consistence increases with the increase of the waste content. The determination coefficient is sufficiently close to one, so the trends of the obtained dependence are considered reliable.

The dependence of the setting time of a mixture on the content of waste is provided in Fig. 6. When 10% of cement content in the mixture is replaced with PS waste, the initial setting time procedure elongates almost twice – from 165 min (when PS 0%) to 350 min (when PS 10%) and is further growing with increase of the content of waste. The obtained equation that's correlation coefficient is 0.928, determination coefficient is 0.861 and standard error 47.9 min. The trends of changes of the terms related to the final setting time procedure are analogous: for example, when

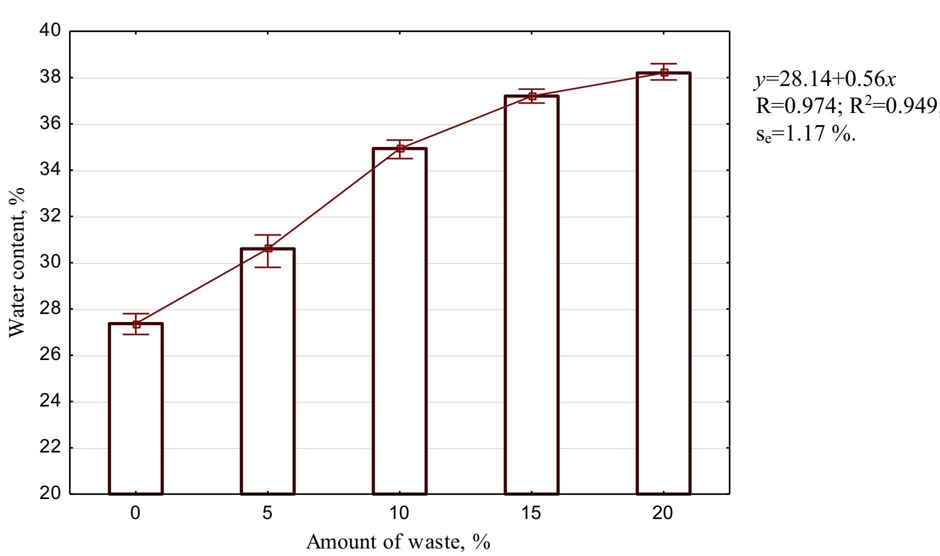

Fig. 5. The results of analysis of the needs in water for obtaining cement paste of the standard consistence dependently on the content of the waste in the mixture.

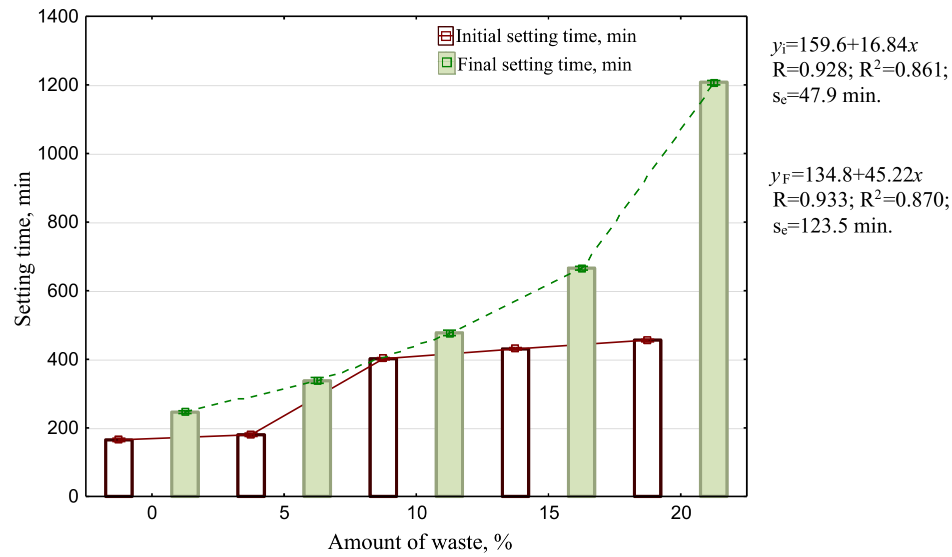

Fig. 6. The dependence of the setting time on the content of waste in the mixture: yi – initial setting time; yF – final setting time.

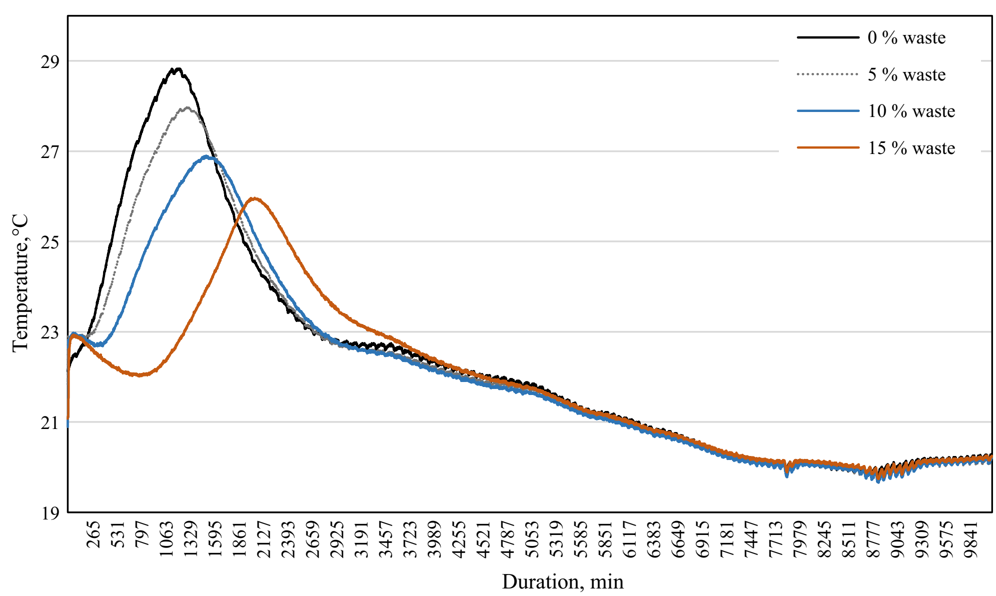

Fig. 7. The dependence of exothermal temperature on the content of waste.

10% of cement content in the mixture is replaced with PS waste, the final setting time procedure elongates from 246 min (when PS 0%) to 477 min (when PS 10%) and is further growing with increase of the content of waste. So, PS considerably prolongs the duration of setting time. It may explained by presence of cellulose in PS composition (Figs. 3 and 4) that retards cement hydration. In addition, large quantities of ettringite were formed in the mixtures (Figs. 11 and 12).

The performed exothermal tests of mortar mixtures also showed that (Fig. 7) that increasing the PS content in the mixture caused retarding the hydration process and reduction of the hydration temperature. The maximum hydration temperature for a mixture without the waste was 29 °C, while for mixture with 10% PS – about 27 °C and for mixture with 15% PS – fell down to about 26 °C. So, the said waste should be useful for working with mortars when the weather is hotter. The setting time of the mortar is retarded by

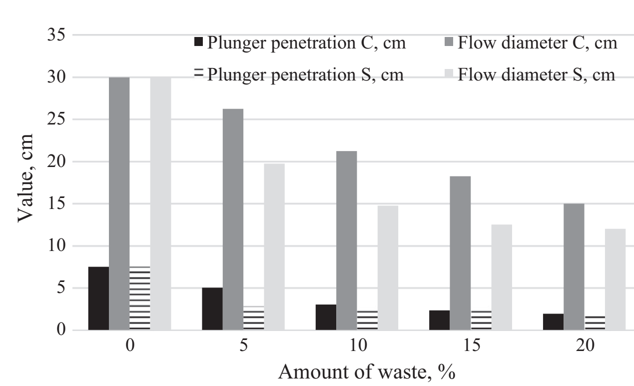

Fig. 8. The dependence of the changes of mortar consistence on the content of waste in the mixture.

about 1000 min as compared to the reference specimens, if 15% PS is added to the mortar. When 5% PS is added to the mixture, no considerable impact is observed; however, the regularities related to the drop of temperature and the increase of the duration of the hydration process remains.

The results of the tests on the mortar consistency are provided in Fig. 8. The consistency had been examined in two ways: according to the flow diameter and according to the plunger penetration. On replacing a part of the content of cement or the content of sand by PS, the flow diameter decreased considerably. When 10% of the content of cement in the mortar was replaced by PS, the flow diameter decreased by about 30%, and when the same percentage of sand was replaced by PS – about 50%. If the maximum 20% content of the waste was added to mixture, the flow diameter decreased by about 50% in case of cement replacement and by about 60% – in case of sand replacement.

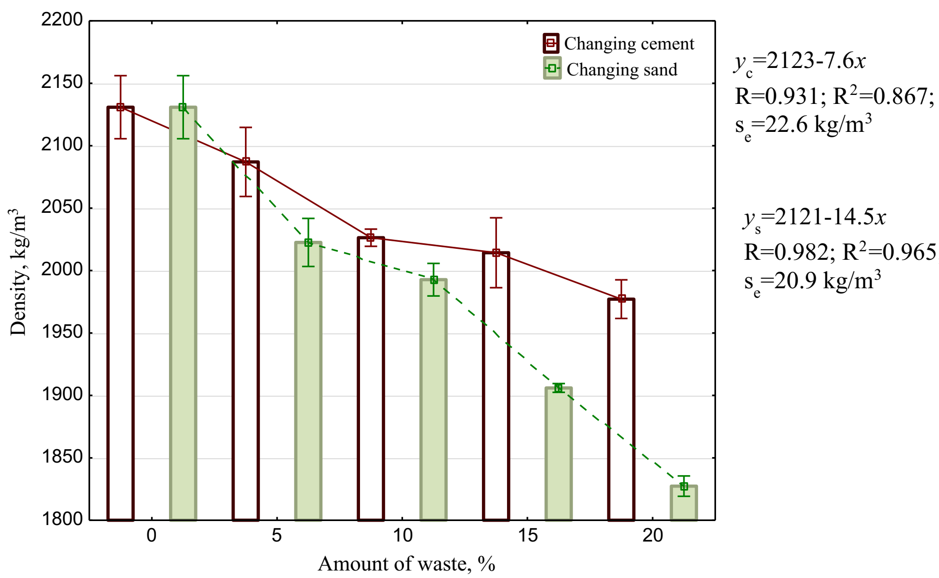

Fig. 9. The dependence of the density of dried mortar on PS content: yc – changing cement, ys – changing sand.

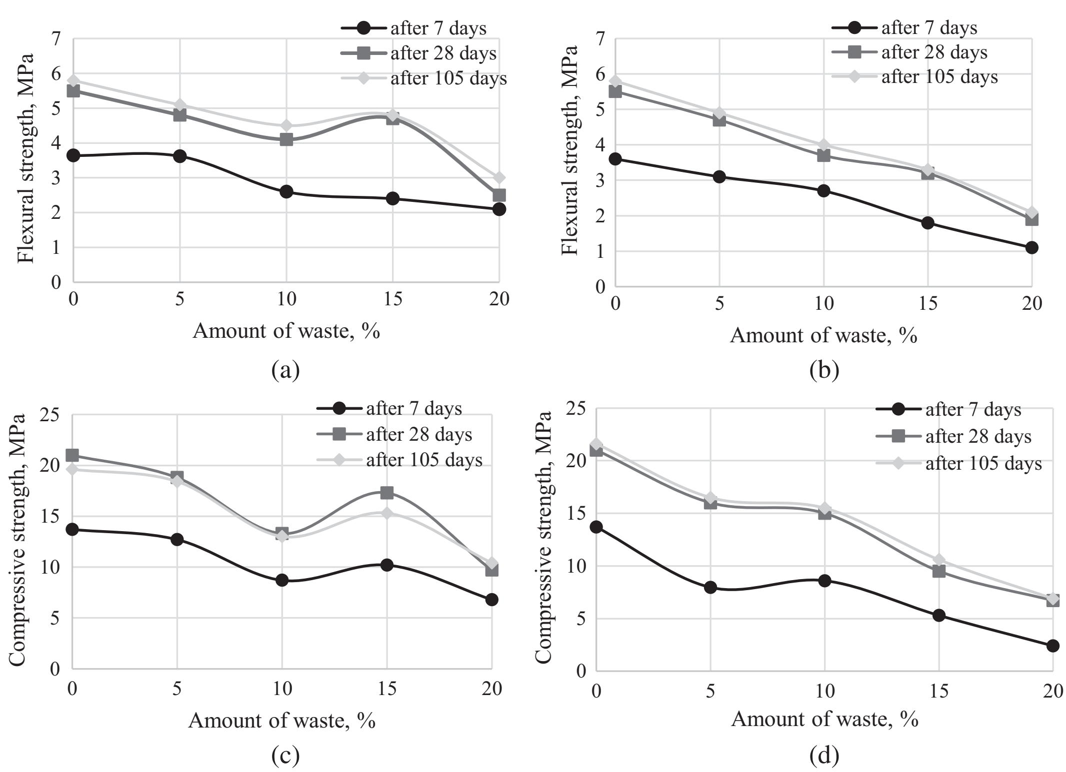

Fig. 10. The dependence of the flexural and compressive strength on the waste content: a) flexural strength, when a part of cement content is replaced by PS waste; b) flexural strength, when a part of sand content is replaced by PS waste; c) compressive strength, when a part of cement content is replaced by PS waste; d) compressive strength, when a part of sand content is replaced by PS waste.

The plunger penetration depth was decreasing in a similar way both on replacing a part of the cement content and a part of the sand content by PS. When 10% of the content of cement in the mortar was replaced by PS, the plunger penetration decreased by about 60%, and when the same percentage of sand was replaced by PS – about 67%. If the maximum 20% content of the waste was added to mixture, the flow diameter in case of cement replacement decreased by about 75%, and in case of sand replacement – about 72%. Such an effect of PS may be explained by its high needs in water (Fig. 5) for mixture formation. PS easily and rapidly absorbs water; if its content is increased to 15–20%, formation of specimens becomes more complicated, a longer duration of vibration is required and so on.

The dependence of the density of dried mortar on PS content is provided in Fig. 9.

Because PS density is several times less than the density of Portland cement and sand, the density of the mortar decreases with increasing PS content in the mixture. When a part of cement content is replaced by PS waste, the density decreasing becomes lower, because a smaller part of the mass is replaced, as compared to the case, when a part of sand content is replaced by PS waste. When 10% of the content of cement was replaced by PS waste,

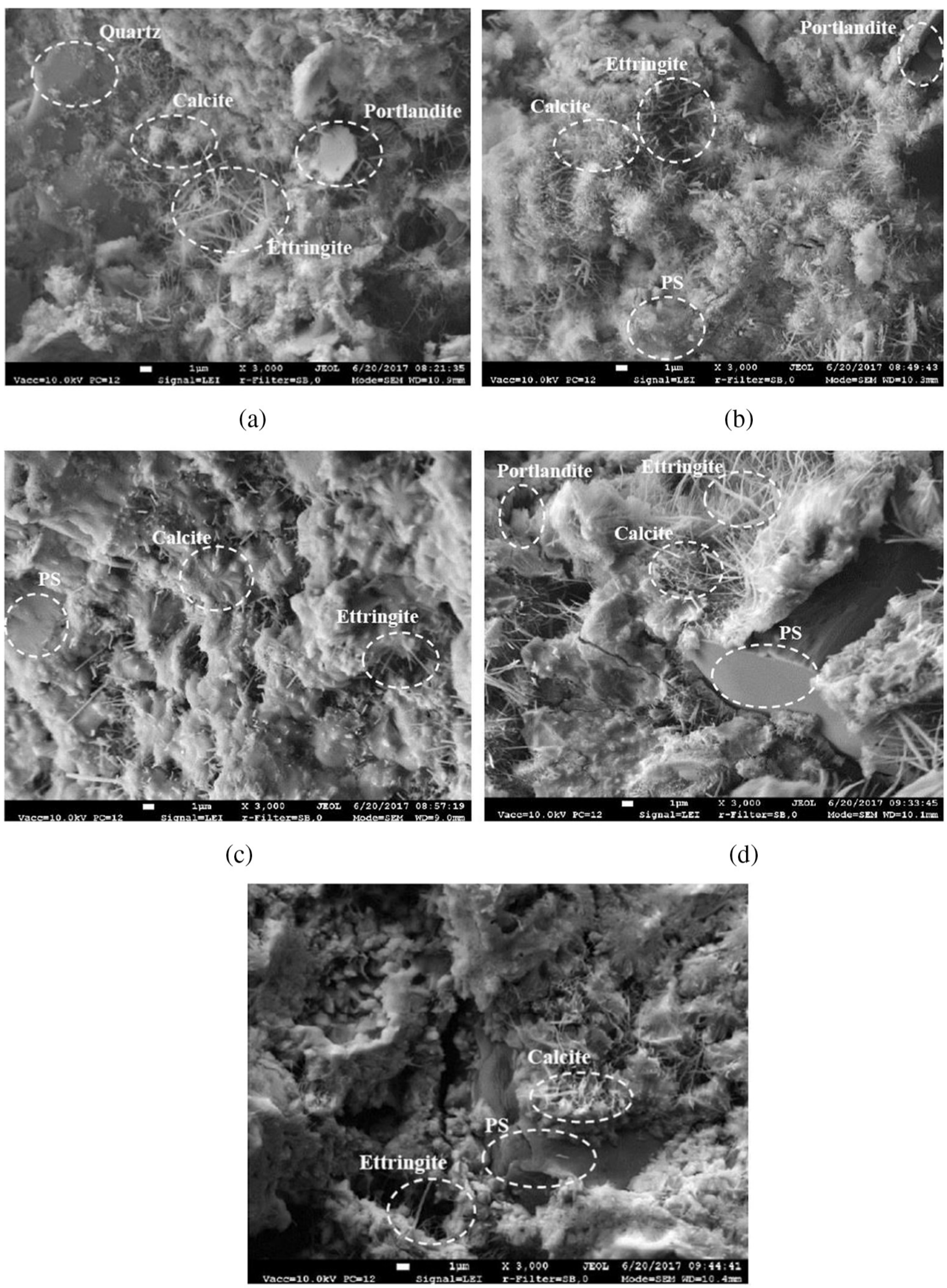

Fig. 11. The dependence of the microstructure on the waste content (×3000): a) without waste, b) when 15% of cement content is replaced with PS waste, c) when 20% of cement content is replaced with PS waste, d) when 10% sand content is replaced with PS waste, , e) when 20% of sand content is replaced with PS waste.

the density decreased by 6%, and when 20% was replaced – by 8%. When 10% of the content of sand was replaced by PS waste, the density decreased by 10%, and when 20% of cement was replaced – by 22%. The formed dependences show that on increasing the waste content, the decreasing of density is linear. The minus sign before the variable *x* shows that the density decreases when the content of waste is increased. The dependences are reliable and the link between the indicators is very strong, because the correlation coefficients in both equations are 0.931 and 0.982, and the coefficients of determination are equal to 0867 and 0.965.

The results of measuring the flexural strength and compressive strength of the mortar are provided in Fig. 10. Variation coefficient of flexural strength results was 0.1–3.8%, of compressive strength – 0.3–2.3%. If a part of sand content of fraction 2/4 is replaced by PS waste, it can be seen that both the flexural strength and the compressive strength are decreasing almost linearly (Fig. 10b, d). If the optimum 5% PS content is used, the flexural strength after 7, 28 and 105 days is gradually decreasing by about 15%, as compared to the reference specimen. The compressive strength decreased by about 40% after 7 days; however, after 28 and 105 days, the decreasing of the compressive strength was about 20% only. When 10% PS was added instead of sand, both flexural strength and compressive strength decreased about 30%.

When a part of cement content (Fig. 10a, c) is replaced by PS, more complicated dependences of the strength on the waste content are obtained. The model of strength variation may be described as a polynomial. When 5% PS waste was added, the flexural strength remained the same after 7 days; however, it decreased by about 12% after 28 days. The observed changes of the compressive strength were inconsiderable as well: when 5% PS waste was added, the compressive strength decreased by about 7%; however, the density of the mortar and the cement content decreased. When 10% PS was added, the strength decreased; however, when PS content was increased to 15%, the strength increased again. While comparing the results in cases of replacement of cement and sand by PS, it may be seen that a higher strength was obtained for specimens where a part of cement content was replaced. Thus, a finer PS waste fraction is more useful when production of a stronger mortar is required; however, PS fraction 2/4 decreases the density to a larger extent and increases the porosity.

In both cases, 20% PS very negatively impacts the properties of the mortar (the strength decreases by about 60%), so its content over 15% in mixtures is not recommended.

The changes of the strength may be explained by the formed different microstructure of the mortar shown in Fig. 11.

It can be seen from Fig. 11 that high content of ettringite (stick- and needle-shaped mineral) was formed in all specimens and the largest aggregates of it are observed at cavities of various shapes. Formation of ettringite regulates the process of the paste setting process: the ettringite films around cement grains slow down hydration of other minerals of cement and simultaneously retard setting of the paste [28]. It may be stated that lower strength of specimens with PS waste was predetermined by the slowdown of cement hydration process caused by formation of ettringite films. In absence of PS waste, the microstructure of the specimen is dense, and in course of gradual increasing the waste content, the pores and their number are increasing. If 20% PS is used instead of cement (Fig. 11 c), abundant interconnecting large pores (10–20 μm) are formed, the structure of the cement stone is coalesced in some points and ettringite appears as needles of various diameters. The specimen is quite fragile.

If 10% of sand content is replaced by PS waste, the structure of the cement stone remains dense. In the photo, PS waste can be seen (while adding PS to the mixture, the waste is dry and porous, so it absorbs much water): its ability to accumulate water causes an appearance of crystals on it. The cohesion between the waste and the cement stone is good, no cracks are observed. If 20% of the sand content is replaced by PS, the structure of the cement stone changes: the size of pores achieves 30–40 μm. The structure is highly porous, it includes abundant interconnecting pores and capillaries; the cohesion between the components is low. Adding more than 15% of the PS waste to the mixture does not mention portlandite minerals, which are in the form of hexagonal plates. Therefore, it can be assumed that when more than 15% of the PS waste content is released, hydration is significantly slowed down. All samples shown calcite, which is shaped like a flower.

The X-ray images of the most typical batches are provided in Fig. 12.

It can be seen from the above-provided X-ray images that in all the batches, the same minerals were formed: ettringite ($3CaO{\cdot}Al_2O_3{\cdot}3CaSO_4{\cdot}31H_2O$), quartz ($SiO_2$), portlandite ($Ca(OH)_2$), dolomite ($CaMg(CO_3)_2$, and calcite ($CaCO_3$), only the peaks of their intensity differ. The highest intensity was found for the curves of ettringite, quartz and dolomite (for specimens free of PS waste); for specimens with PS waste, the highest intensity of peak was found for calcite, when calcite was a predominating element of PS waste

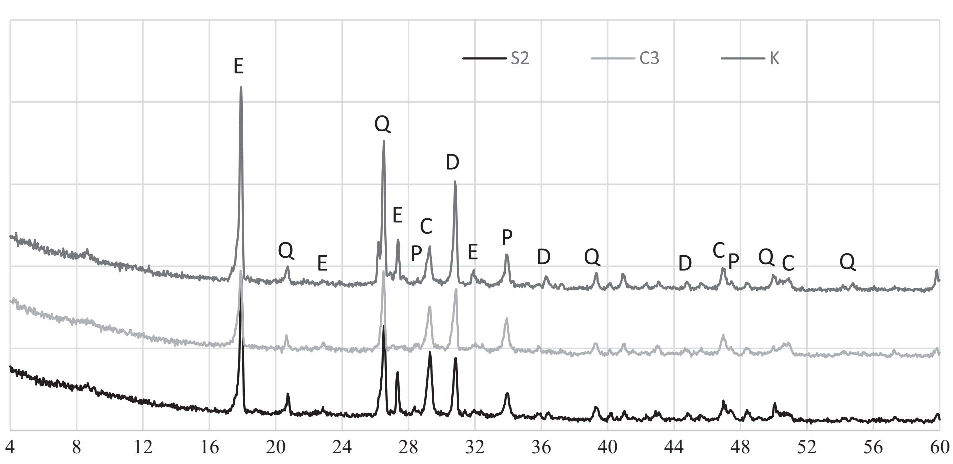

Fig. 12. The X-ray images of the most typical batches (E – ettringite, Q – quartz, P – portlandite, D – dolomite, C – calcite).

(Fig. 3). The results show that the chosen W/C ratio ensures the normal run of the hydration process.

5. Conclusions

The principal components of dried PS waste include cellulose and calcite. PC waste distinguishes for high open porosity and ability to absorb water. When the content of waste in the mixture was increased from 0% to 20%, the need in water for cement paste of normal consistence increased by about 40%. The initial setting procedure and the final setting procedure became about 3 times longer. When 10% of cement content in the mixture was replaced by PS waste, the duration of the final setting procedure increased from 246 min to 477 min. The performed exothermal tests also showed that on increasing the content of PS waste in the mixture, the process of hydration is retarded and the hydration temperature decreases. 20% PS waste dramatically (in half) reduces the flow diameter and the plunger penetration. On increasing the content of PS waste, the decreasing of density of the mixture is linear. The correlation coefficients of linear dependence are about 0.9.

When 5% of cement content was replaced by PS waste, inconsiderable changes of the compressive strength (the compressive strength reduced by about 7%) were observed. The strength of this mortar after 28 days is high enough: the flexural strength was about 5 MPa and the compressive strength was about 19 MPa. Such a mixture may be used for construction works. 20% content of PS waste negatively impacts the properties of the mortar (the strength decreases by about 60%), so its content over 15% in mixtures is not recommended.

In course of SEM tests, it was found that on gradual increasing the content of PS waste, pores and their quantity are increasing. When 20% of cement content was replaced by PS waste, abundant large interconnecting pores were formed. When 10% of sand content was replaced by PS waste, the structure of the cement stone remained dense. The cohesion between the waste and the cement stone is good, no cracks are observed. If 20% of sand content is replaced by PS, the structure of the cement stone becomes highly porous, it includes abundant interconnecting pores and capillaries; the cohesion between the components is low.

XRD analysis shows the predominating minerals in batches of specimens are the same. They include: ettringite, quartz, portlandite, dolomite, calcite; only the peaks of their intensity differ. Larger content of calcite was found in specimens formed upon using larger quantities of PS waste.

PS waste may be used for mortar mixtures in small quantities by replacing up to 5% of the content of cement or natural fine filler, thus reducing their content in the mixture and slightly altering the properties of the mortar. When this waste is used, it is possible to work at higher air temperatures, because it retards hydration, or, if needed, an accelerator of hardening may be used in addition.

Conflict of interest

The authors declare that they have no conflict of interest.

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