Industrial Crops and Products 77 (2015) 724–734

Contents lists available at ScienceDirect

Industrial Crops and Products

journal homepage: www.elsevier.com/locate/indcrop

Impact of hemp shives aggregate mineralization on physical–mechanical properties and structure of composite with cementitious binding material

Giedrius Balčiūnas[a,*], Ina Pundienė[b], Lina Lekūnaitė-Lukošiūnė[a], Sigitas Vėjelis[a], Aleksandrs Korjakins[c]

[a] Vilnius Gediminas Technical University, Scientific Institute of Thermal Insulation, Laboratory of Thermal Insulating Materials, Linkmenų st. 28, LT-08217 Vilnius, Lithuania
[b] Vilnius Gediminas Technical University, Scientific Institute of Thermal Insulation, Laboratory of Building Products Technology, Linkmenu st. 28, LT-08217 Vilnius, Lithuania
[c] Riga Technical University, Institute of Materials and Structures, Azenes st. 16/20, Riga LV−1048, Latvia

ARTICLE INFO

Article history:
Received 30 April 2015
Received in revised form 4 September 2015
Accepted 5 September 2015
Available online 8 October 2015

Keywords:
Fibre hemp shives
Cement
Compressive strength
Thermal conductivity
Plant based aggregates mineralization

ABSTRACT

This article analyses the possibility of fibre hemp shives (FHS) to be used as lightweight aggregate in composite with cement binding material. Cement and plant origin aggregates are hardly compatible, because water-soluble or alkaline environment soluble compounds inhibit cement hydration. To avoid harmful effect on cement hydration, it is necessary to mineralize FHS aggregates with complex mineralizer (which consists of $Al_2(SO_4)_3$ and $Ca(OH)_2$) in order to minimize the impact of hydration retarders on cement hydration. Rational amount of super plasticizer for forming mixture is selected in accordance with viscosity of cement paste. Amount and ratio of mineralizer was selected according to kinetics of hydration temperature of forming mixture, compressive strength, ultrasonic pulse velocity (UPV). It was determined that the increase in the amount of mineralizer from 27% to 54 % (estimated based on FHS mass) promotes the cement hydration, shortens the time needed to reach EXO maximum and increases its temperature, because the higher content of the $Ca(OH)_2$ participates in the process of dissolution and makes the EXO reaction faster. The highest acceleration of cement hydration in a composite sample is caused by optimum amount of component mineralizer which is 54%. The lower content of component mineralizer did not completely bind sugar released from FHS, therefore, causing inhibition of cement hydration. Optimal amount and ratio of mineralizer allow obtaining the compressive strength after 28 days of curing of 8.03 MPa as well as thermal conductivity of 0.179 W/(m K). Microstructure analyses show that cement matrix with optimal amount of complex mineralizer is dense enough and well bonded with new monolithic hydration products.

1. Introduction

Fibre hemp is one of the most rational plants because all of its parts are used in industry. Hemp seeds are used for the edible oil extraction (Elfordy et al., 2008) and biodiesel production (Casas et al., 2005; Li et al., 2010), bast fibres are used in special paper production, cars (for thermoplastics reinforced with natural fibres and used in the manufacture of door panels), construction (thermal insulating boards, reinforced concrete), other (agro and geotextiles, mattresses, shoes) industries. Fibre is obtained by soaking the hemp stalks in order to separate the fibres and non- fibrous components called shives. Hemp shives are ligneous woody tissues, which are considered as fibre products obtained by secondary manufacturing (Karus and Vogt, 2004). It is estimated that the global market for hemp consists of more than 25,000 products (Salentijn et al., 2015). Nowadays, the use of plant particles as building material aggregates is justified by two main reasons:

- Thepreservation of natural resources such as mineral aggregates whose extracting conditions become increasingly difficult.
- Theneed to design efficient building materials (thermal insulating, sound absorbing, sound insulating) with lower

* Corresponding author.
E-mail addresses: giedrius.balciunas@vgtu.lt (G. Balčiūnas), ina.pundiene@vgtu.lt (I. Pundienė), lina.lekunaite@vgtu.lt (L. Lekūnaitė-Lukošiūnė), sigitas.vejelis@vgtu.lt (S. Vėjelis), aleksandrs.korjakins@rtu.lv (A. Korjakins).

http://dx.doi.org/10.1016/j.indcrop.2015.09.011
0926-6690/

environmental impact especially in regard to the carbon footprint (Arnaud and Gourlay, 2012).

Fibre Hemp Shives (FHS) are used as aggregate for the manufacture of hemp concrete. Lime is the most commonly used binder for the manufacture of hemp concrete. The main advantages of hemp concrete with lime binder are good thermal insulating properties (Evrard and De Herde, 2010; Elfordy, 2008), low impact on the environment (Bevan and Woolley, 2008), buffer moisture properties (Collet and Pretot, 2012). FHS- lime composites are characteristic of good enough thermal insulation (Daly et al., 2010) and acoustic (Glé et al., 2011) properties, however, their mechanical properties are rather low (up to 1 MPa) (Arnaud and Gourlay, 2012). The compressive strength of hemp concrete with lime binder is too low for installation of bearing structures, therefore, it is usually used in combination with wooden load-bearing frame (Gross and Walker, 2014), or additional technological processes in manufacture, e.g. pressing of the formation mixture, are required in order to improve strength characteristics. The maximum compressive strength of hemp concrete with lime binder processed in this way might be 3 MPa (Nguyen et al., 2009). The mechanical properties of cement binder are higher in comparison to lime, therefore, it can be expected to develop composite material meeting the requirements applicable to structural material, if lime is replaced with cement. However, manufacture of cement—plant origin aggregates requires mineralization of the aggregates, because water-soluble compounds or compounds soluble in the basic environment contained in FHS inhibit cement hydration (Semple and Evans, 2000). The major compounds inhibiting cement hydration in composite materials consisting of cement and plant origin aggregates include sugars and a part of hemicellulose which can release sugar under certain conditions (Thomas and Birchall, 1983; Bilba et al., 2003). French scientists (Sedan et al., 2008) have used hemp fibres for the reinforcement of concrete. For better adhesion of matrix and fibres, sodium hydroxide and aluminium chloride solutions was used for fibre treatment (mineralization). It is found that composite containing 16 vol.% of fibres has the flexural strength by approximately 40% higher than that of the cement paste. Likewise it is observed a decrease in Young's modulus of composite compared to the cement paste. Indeed, the treatments applied on hemp fibres in this study have improved only the flexural strength. An alkaline treatment improves the mentioned strength by approximately 94% compared to the cement paste. Alkali treatment affects not only the fibre strength, but the fibre–matrix adhesion in a positive way as well. Brazilian scientists (Jarbo et al., 2013) have used corn stalk fibres and pine pulp for fibre-cement formation. It is found out that in order to obtain the same mechanical properties as those achieved when the product is produced with pine pulps, the combination of corn stalk fibres and pine or synthetic fibres is required. Although, the best results have been obtained using the NaOH–anthraquinone process, boiling the corn stalk in 10% solution of NaOH and the temperature of 140 °C for 30 min is the optimal pulping process. Mild boiling conditions in the NaOH–anthraquinone process is favourable for obtaining longer fibres which could be used for final product with better mechanical, physical and solid retention properties. It was also found that starch and tannins can also inhibit cement hydration in combination with the aforementioned sugars (Vaickelionis and Vaickelionienė, 2006). Other extract materials aggravating compatibility of cement and plant origin aggregates are resin, fatty acids, terpenes, and terpenoids, simple sugars or salts contained in FHS (Pehanich et al., 2004). Chemicals such as CaCl2 (Semple and Evans 2004; Courard et al., 2011), $Al_2(SO_4)_3$ (Beck et al., 2004), $Na_2(SiO_2)$ nO (liquid glass) (Ma et al., 2010), sometimes also referred to as the mineralization agents (mineralizers), improve compatibility of cement and plant origin aggregates. Complex mineralizers, such as $Al_2(SO_4)_3 + Ca(OH)_2$ (hydrated lime), are also used. When $Al_2(SO_4)_3$ is used as a mineralizer, it impedes release of sugar from organic aggregates and reduces higroscopicity and water absorption thereof.

Aluminium sulphate in the form of hydrate is characteristic of acidic reaction in water (pH = 3–5). Hydrated lime ($Ca(OH)_2$—is characteristic of alkaline reaction in water pH = 11–12), enhances efficiency of aluminium sulphate, neutralizes the acidic environment caused by $Al_2(SO_4)_3$ and improves workability of the mixture (Boltryk and Pawluczuk, 2014). Aggregate mineralization also leads to improved adhesion between aggregate and cement stone (Małaszkiewicz and Bołtryk, 2008).

Concrete industry, especially Portland cement manufacture is known as a heavy contributor to the environmental damage and CO_2 emissions. Environmental pressing can potentially reduce the use of cement and natural aggregates (for example gravel) in concrete production. Lower content of cement and natural aggregates in concrete industry can reduce the impact on the environment (Limbachiya et al., 2012). Mineralization with $Al_2(SO_4)_3$ and $Ca(OH)_2$ additives is also beneficial in terms of environmental protection, because composites with cement binder and $Ca(OH)_2$ absorb CO_2 from the environment. This effect is determined by carbonation reaction of hydrated lime used for aggregate mineralization with $Ca(OH)_2$ and carbonization of cement hydration products, i.e. portlandite. When carbonization efficiency rate is 100 %, 1 ton of cement can absorb 0.5 ton of CO2 and produce 1.5 tons of calcium carbonates and silicon gel (Boltryk and Pawluczuk 2013).

Composite materials consisting of hemp shives aggregate and Portland cement binder have been little explored. The Polish scientists have carried out research with reed and straw (Bołtryk and Pawluczuk, 2014). Cement binder has been used for composites with flax aggregate (Khazma et al., 2010). Based on the experimental researches carried out by the aforementioned scientists, the measured values of compressive strength of a composite with flax aggregate and cement binder is rather low and ranges from 2.08 MPa to 2.74 MPa when densities ranged respectively from 700 kg/m3 to 1013 kg/m3. In the later study, scientists (Khazma et al., 2014) have investigated the impact of flax shives treatment with linseed oil on physical–mechanical properties of cementitious composites. It is found that linseed oil treatment reduces setting time and improves compressive strength as well as thermal conductivity. The composition and properties of agricultural waste fibres have a significant effect on the properties of cement, e.g. hydration and setting (Li et al., 2004). Recent research efforts have been aimed to reinforcing fibre-cement boards with agricultural residues and focused on preventing the adverse effects of the water soluble constituents of such residues on the hydration and the strength development of cement (Soroushian et al., 2004). The Czech scientists have carried out researches by using hemp shives and magnesium oxychloride cement for formation of composite material (Števulová et al., 2013). The scientists have found that in case of using magnesium oxychloride cement composites the achieved mean compressive strength is 2.73 MPa, when the mean density is 1040 kg/m3. So it may be concluded that composites of high enough density can be obtained even when using cement as binder. In addition to this, these composites are characteristic of low strength variations of which can have a negative impact on the potential of using such composites for industrial purposes. Composites characteristic of lower density and higher strength should be developed in order to successfully expand application of FHS. However, some problems still impede the development of plant origin aggregates and cement composites. To solve this problem the effective chemical admixture must be selected.

The aim of this paper is to choose a rational amount of plastifying and complex mineralizer, composite with FHS aggregate and cement binder, formation mixture and to evaluate the effect of the

Table 1
Chemical composition of FHS.

Raw material	Cellulose (%)	Lignin	Extractives	Ash	Hemicelluloses	Pectins
Hemp stalks	46.9	17.4	1.9	1.3	24.6	7.9

Table 2
Declared performance of cement.

Characteristics of cements	Performace	Harmonised technical specification
CEM I 52,5R		EN
Compressive strength, MPa:	≥30.0≥52.5	197-
Early Strength		1:2001
Standard strength		
Setting time, min	≥45	
Stability of volume, mm	≤10	
Calcination loss, (%)	≤5.0	
Residue, (%)	≤5.0	
Amount of sulphates (SO_3), (%)	≤4.0	
Amount of chlorides, (%)	≤0.1	
Fineness (Blaine), cm^2/kg	500	

additives on the structure and physical–mechanical properties of the composite.

2. Experimantal

2.1. Materials and methods

Shives received from fibre hemp (variety USO 31) grown in the Eastern Lithuania were used as an aggregate. The used shives were characteristic of 2.5–10 mm fraction. After sieving and primary treatment of the aggregate it was found that 2.5–5.0 mm fraction made 45% and 5.0–10 mm fraction made 55% of the mass. The measured bulk density of the used FHS was 105 kg/m3. FHS has porous microstructure, which is suitable as an aggregate for lightweight concretes. Porous structure ensures lower density and thermal conductivity of composites. As it is shown in Fig. 1a, FHS structure is porous perpendicularly to the plane of growing direction (pores are of two different sizes: 1 (so called vessel) and 2 (so called xylem ray (Amaducci et al., 2014) may be seen). The pores of first type have the form of ellipse; the length of longer ellipse axis is averagely of 85 μm and shorter ellipse axis is of 45 μm. The pores of second type are of various forms, i.e. ellipse, rectangle, circle; average diameter of the pore is of 25 μm. As it can be seen from Fig. 1b, there are two types of pores in the plane parallel to the growing direction as well, i.e. close to the form of ellipse and rectangle/rhombus. The pores of the first type have the form of ellipse; the length of longer ellipse axis is averagely of 345 μm and shorter ellipse axis is of 37.5 μm. The pores of second type are of various forms, i.e. ellipse, rectangle, circle; average diameter of the pores is of 45 μm. Chemical composition of FHS is presented in the Table 1.

CEM I 52.5R brand cement, in compliance with the requirements established in Standard EN 197-1 was used as the binder. The declared performance is provided in Table 2.

Complex mineralizer, consisting of aluminium sulphate ($Al_2(SO_4)_3{\cdot}16H_2O$) and hydrated lime (particle surface area estimated by means of Blaine method was 6176 cm^2/kg), was used for FHS mineralization.

Polycarboxylate ether-based superplasticizer (SP) Glenium 430 was used for enhancement of rheological properties of formation mixture and mechanical properties of the composite.

For cement paste with SP additive rheological properties evaluating SV-10 vibro-viscosimeter was employed. With SV-10 vibro-viscosimeter it is possible to define the dynamic viscosity of pastes up to 12,000 mPa·s with 0.01 mPa·s accuracy in a very small amount of paste (35 ml). The instrument measures paste viscosity resistance to constant vibration of gauge plates at 30 Hz frequency. The proportional to viscosity resistance force is converted into electrical signal and recorded. The W/C ratio in cement pastes was 0.32 and in all cases constant, thus also enabling measurement of maximal amounts of SP. Cement pastes with SP contents dosed as follows 0%, 0.3%; 0.6%; 0.9%; 1.2% (depending on cement amount) were prepared for the tests. The dynamic viscosity of the prepared pastes was measured instantly and then after 5, 10, 15, and 20 min. The time chosen for measurements corresponds with actual concrete placing terms.

2.2. Composition of formation mixtures

Upon identification of the optimum SP content (0.9%), further it was focussed on search of the optimum content of complex mineralizer for FHS aggregate mineralization. The aggregates were being mineralized based on the technology developed by Bielystok scientists (Bołtryk 1994; Bołtryk and Rutkowska 2005) designed for wood aggregate mineralization. The aggregates were mineralized by Bielystok scientists by using $Al_2(SO_4)_3$ and the acidic $Al_2(SO_4)_3$ reaction was neutralized with hydrated lime ($Ca(OH)_2$). The rational content of mineralizer identified based on the technology of the Polish scientists is: 9% of $Al_2(SO_4)_3$ and 18% of $Ca(OH)_2$ of the shives mass. Although the chemical composition is similar, but the percentage of component content in FHS and wood is different, moreover, the density and structure of FHS and wood differ as well. Identification of the optimum contents of mineralizer is necessary due to the aforementioned differences. Therefore, different ratios and quantities of $Al_2(SO_4)_3$ and $Ca(OH)_2$ were tested for FHS mineralization. 8 compositions of formation mixtures (the compositions are provided in Table 3) were formed in order to determine the effect of ratio and content of complex mineralizer on the course of hydration of cement paste as well as physical-mechanical properties of the composite. The content of complex mineralizer was estimated based on FHS quantity. The ratio between $Al_2(SO_4)_3$ and $Ca(OH)_2$ increased from 1:1 to 1:2 (Table 3). Furthermore, it can be emphasized that the total content of mineralizer was being increased from 30% to 81% (based on FHS mass).

FHS content was estimated based on bulk density so that the entire size of the mould would be filled, the cement content was selected based on aggregate and binder ratio (A/C = 0.25, here: A/C—aggregate binder ratio), water was added to the formation mixture in two stages, i.e. I—for aggregate mineralization (1.5 · mFHS, here mFHS—hemp shives mass, kg), II – for preparation of cement paste (w/c = 0.32, here: w/c—water cement ratio).

2.3. Aggregate mineralization, preparation of formation mixture and samples

Preparation of formation mixture consists of several stages. FHS was mineralized during the first stage: the weighed FHS was poured into the rotating mixer pouring $Al_2(SO_4)_3$ solution of the required concentration on top of it. This tends to impede the release of sugar from organic aggregates and reduce hygroscopicity and water absorption thereof.

Water required for preparation of formation mixture was divided into two portions: the first portion was for FHS mineralization, the second one was for preparation of cement paste. After pouring the $Al_2(SO)_4$ solution into the mixer with FHS, mixing took 3 min and then the mass was left for 15 min so that FHS would mineralize. After the treatment, lime was poured into the mixer and mixed for 1 min for the acidic $Al_2(SO_4)_3$ reaction neutralization with hydrated lime ($Ca(OH)_2$).

The water intended for the cement paste was mixed with SP additive and poured into a separate container with a weighed

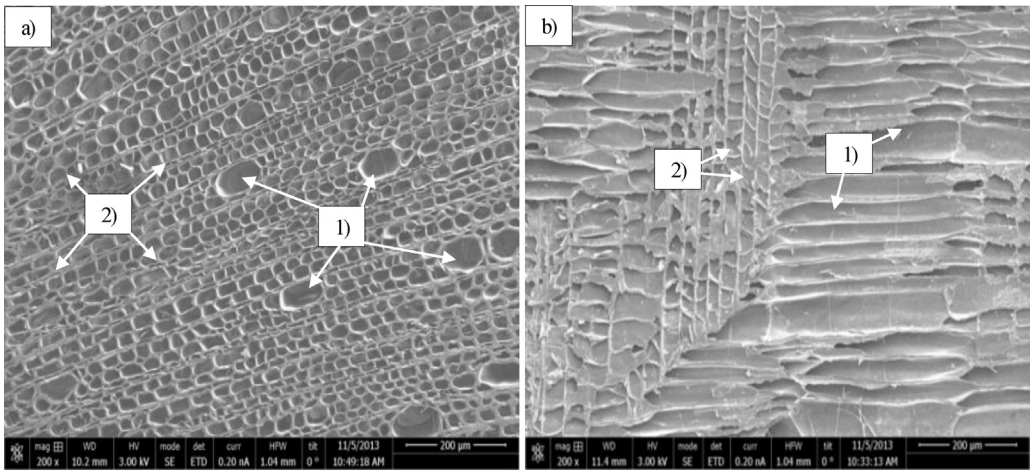

Fig. 1. Microstrucure of FHS.

Table 3
Forming mixtures of the composite with different amount of complex mineralizer.

Composition Number	Amount of complex mineralizer, %	Ratio $Al_2(SO_4)_3$ to $Ca(OH)_2$	Amount of SP,	Water		A/C[a] ratio
				w/c[a] ratio	Water for mineralisation	
Control	0	–	0.9	0.32	1.5	0.25
1	30	1:2			.	
2	36	1:1			m_{FHS*}	
3	45	1:2				
4	48	1,67:1				
5	54	1:2				
6[b]	63	1:2				
7	81	1:2				

[a] A/C – Aggregate (FHS-Fibre hemp shives)/Cement ratio; w/c – water/cement ratio, m_{FHS} – mass of FHS (mass of FHS for 100 × 100 × 100 mm samples was 175 g and for the 300 × 300 × 50 mm samples was 787 g).
[b] Due to the large amount of lime, which have a very high surface area, the forming mixture was not mixed up, when w/c = 0.32 were used, therefore, 30% of water was added additionally.

required quantity of cement. It was being mixed for 2 min using an electric mixer, the prepared paste was poured into the mixer containing mineralized FHS and mixed until homogeneous mass was obtained (approximately for 2–3 min). The prepared formation mixture was placed in metal (100 × 100 × 100) mm moulds in three stages and compacted using a metal bar. The samples were left to get harder for 24 h, later on they were removed from the moulds and hardened at (23 ± 2)°C temperature and under conditions of (50±5)% relative humidity.

2.4. Experimental methods

2.4.1. Compressive strength

Compressive strength of composite samples was being estimated after 7 and 28 days of hardening. Tests were carried out in accordance with the requirements of Standard EN 12,390 using a laboratory press Tinius Olsen H200 KU, the maximum load was 200 kN, load accuracy was ±0.5%, positioning accuracy was ±0.01%.

2.4.2. Thermal conductivity

Thermal conductivity tests were performed using heat flow meter apparatus FOX 304 (LaserComp, USA). Measurement range of the apparatus is from 0.5 W/(m K) to 0.004 W/(m K) with a centrally located heat flux transducers having dimensions of (100 × 100) mm. Thermal insulating properties of materials were measured in accordance with EN 12,664 and ISO 8301.

2.4.3. Exo temperature

The concrete mix hardening temperature kinetic was followed by the exothermic (EXO) profile, according to the Alcoa methodology (Alcoa, 1999). The raw materials being used were air-conditioned at (20 ± 1)°C temperature before the test. The fresh composite mix heat development, which results from the exothermic reaction of the cement hydration, was determined at (20 ± 1)°C with fresh 1.0 kg composite mix samples, placed in an insulated (10 × 10 × 10) cm textolite chamber. A thermocouple (type T), imbedded in the sample, is linked to a data capture system and the temperature is recorded as a function of time. The formation mixture placed to the moulds was immediately placed to a metal box (without taking it out of the mould) which was insulated with a 50-mm-thick expanded polystyrene foam insulation. Four compositions with different mineralizer contents were formed at the same time. Cement hydration kinetic of the egzothermic reaction temperature was measured during the EXO temperature test. Compositions with cooled and non-cooled FHS were tested in order to estimate whether the released heat during mineralization process has an impact on the cement hydration.

In the first case, the mineralized FHS was cooled down to room temperature (~20 °C), the cement paste was poured to mineralized and cooled down FHS, mixed for 2 min by forced mixing in a rotary mixer and poured into moulds in order to avoid the effect of excessive heat resulting from complex effect of mineralizer reaction on cement hydration temperature kinetic. In the second case the EXO temperature was measured applying the same principle as it was applied to formation of the samples, i.e. the cement paste was poured to non- cooled, mineralized FHS. The temperature of

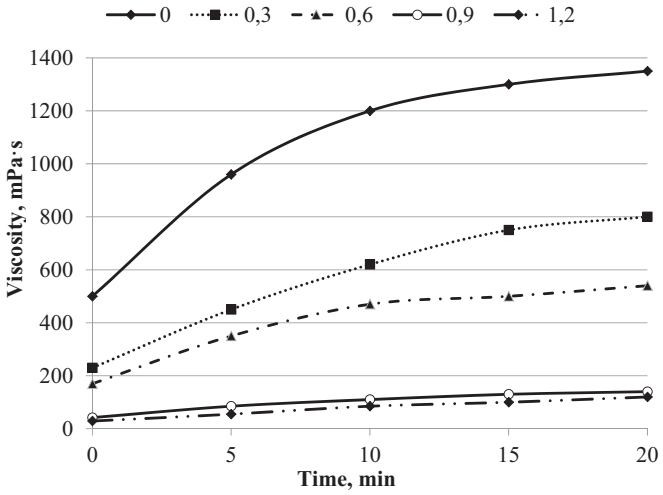

Fig. 2. The change in dynamic viscosity of cement pastes depending on amount of SP.

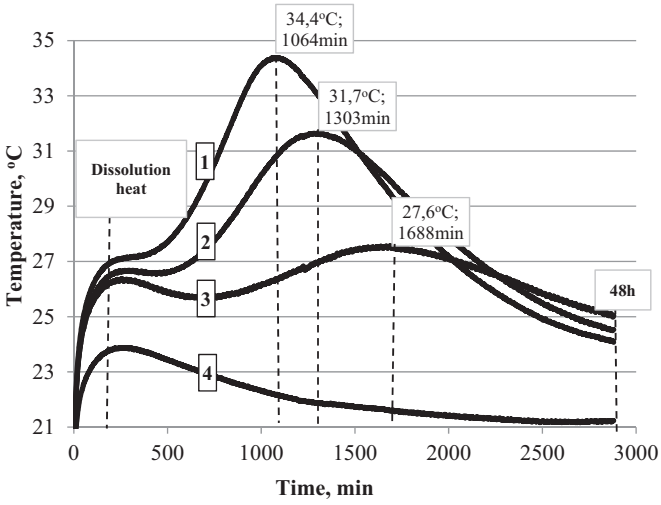

Fig. 3. The change curves in hydration temperature of forming mixtures with different amount of complex mineralizer, when cement paste was added to cooled FHS. 1—amount of complex mineralizer – 54%; 2—amount of complex mineralizer – 45%; 3—amount of complex mineralizer – 27%; 4—non-mineralized FHS.

formation mixture was being recorded every 1 min. The temperature was being measured and its values were being recorded continuously as long as the heat release processes prevailed in the sample.

2.4.4. Microstructure

The microstructure of composite was investigated using SEM "FEI Helios NanoLab 650"(Japan), having resolution of 0.8 nm.

2.4.5. Ultrasonic pulse velocity

"Pundit 7" device was used for determination of ultrasonic pulse velocity (UPV). Time in which the ultrasonic waves pass through the sample was measured perpendicularly to the sample formation direction.

UPV was determined based on the methodical instructions specified in EN 12,504-4 and the provided formula:

$$V_{\mathrm{UPS}} = \frac{l}{\tau \times 10^{-6}}, \mathrm{m/s} \quad (1)$$

here l—composite sample length, m, — signal propagation time, s, 10^{-6}—conversion rate.

3. Results and discussion

3.1. Cement paste viscosity measurements

SP impact on dynamic viscosity kinetic of cement paste was evaluated using vibroviscosimeter enabling to check viscosity changes within the preferred period of time easily. Increase of dynamic viscosity indicates changes in rheological properties of cement pastes within the course of hydration process and development of new formations. Tests were carried out in presence of identical W/C ratio in the paste, changing only SP content. Tests of control cement paste without SP additive is provided for comparison (Fig. 2). Viscosity of cement paste without SP additive increased from 500 mPa·s to 1350 mPa·s within the test period. The gradual decrease in cement paste viscosity indices both in the initial phase and the ones determined at the end of the measurement period within increase of SP content in comparison to the control paste. Upon increase of SP content up to 0.9% and 1.2%, cement paste viscosity decreases up to 38 mPa·s and 30 mPa·s, and become even in both pastes making 120 mPa s at the end of the measurement. It can be seen that increase of SP content up to 1.2% changes cement paste viscosity to a minor extent, therefore, SP content amounting to 0.9% is the optimum one. The aforementioned SP content has been selected for further testing.

3.2. EXO temperature measurements

EXO temperature test of composite material formation mixture was carried out in order to assess the effectiveness of complex mineralizer additive. Compositions of formation mixtures characteristic of steady ratio of complex mineralizer components $Al_2(SO_4)_3$ and $Ca(OH)_2$, i.e. 1:2, but the total complex mineralizer content increases to 27%, 45%, and 54%, have been selected for the EXO temperature test. The EXO temperature test was also carried out with control formation mixture—without a complex mineralizer additive. As mentioned above, EXO temperature of the forming mixture was being measured in two cases. In the first case, when the cement paste was poured to mineralized and cooled down FHS, the temperature change curves of the forming mixtures have been provided in Fig. 3.

It can be seen from Fig. 3, cement hydration temperature rises along with increase of complex mineralizer content for FHS mineralization. It can be assumed that the dissolution heat rises within increase of complex mineralizer content and the temperature during the first stage (~3 h) rises. The smallest increase in temperature, only by 2.5°, is recorded in case of composite composition 4 (without a complex mineralizer additive)(from 21 °C to 23.5 °C), and in case of compositions 3, 2, and 1 the temperature rises from 21 °C to 26 °C and 27 °C.

It can be presumed that temperature changes in the 4th composition are determined by the cement dissolution heat which releases when the reaction of pectin and cement minerals occur. During the cement minerals dissolution process, Ca ions interact with pectin (Sedan et al., 2008). In case of cement and water interaction, small quantity of ettringite is formed. As it is mentioned in the study, OH− trapping should be due to the free carboxylate and alcohol functions found in the chemical structure of the fibre compounds, in particular in the pectin. These functions are ionised in alkaline media and then, fibre surface carries an electrical negative charge that can interact with calcium ions to form $Ca(OH)_2$ nodules. It seems that pectin contained in the FHS can form complex molecules with calcium ions and could be responsible for the observed delay of hydration. Because of this the further cement hydration does not occur and CHS is not formed (Sedan et al., 2008),

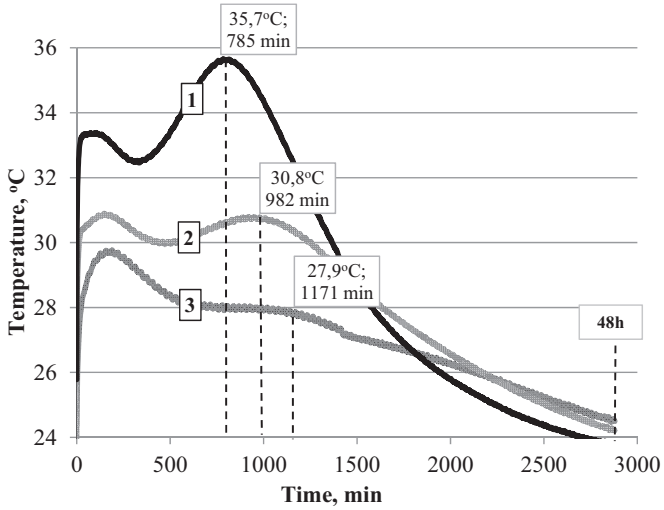

Fig. 4. The change curves in hydration temperature of forming mixtures with different amount of complex mineralizer, when cement paste was added to non-cooled FHS. 1—amount of complex mineralizer – 54 %; 2—amount of complex mineralizer – 45%; 3—amount of complex mineralizer – 27%

possibly only ettringite is formed. When the mineralizer is used, the cement hydration occurs because $Al_2(SO_4)_3$ impedes the release of sugar from FHS and Ca ions that come from complex mineralizer component ($Ca(OH)_2$) and react with pectin stimulating formation of $Ca(OH)_2$ nodules resulting that there are enough of Ca ions for cement hydration. It can be observed that temperature increases when higher amount of the mineralizer is used. Ca ions that come from the mineralizer and stay unbonded on FHS surface increase the dissolution temperature of cement minerals and take part in cement hydration. When the amount of mineralizer is lower, the peak of cement minerals dissolution is visible. The intensity of the peak depends on the amount of mineralizer (peak intensity is higher when the higher amount of mineralizer is used). After dissolution, it can be noticed the induction period, which duration also depends on amount of mineralizer (higher amount of mineralizer significantly shortens duration of induction period).

During the exothermal reaction the highest hydration temperature (peak of EXO temperature) is reached when the maximum complex mineralizer content, i.e. 54%, is used for aggregate mineralization. In presence of the same composition of formation mixture increasing of complex mineralizer content even more is irrational, because in this case the total content of fine particles in the composite was increased and increase of complex mineralizer $Ca(OH)_2$ component content resulted in unworkable formation mixture and poor mixing qualities. The peak EXO temperature, i.e. 34.4 °C, of formation mixture with 54% of complex mineralizer (Fig. 3, curve 1) is reached after 1064 min. The peak EXO temperature, i.e. 31.7 °C, of formation mixture with 45% of complex mineralizer (Fig. 3, curve 2) is reached after 1303 min. The peak EXO temperature, i.e. 27.6 °C, of formation mixture with 27 = % of complex mineralizer (Fig. 3, curve 3) is reached after 1688 min. The peak temperature, i.e. 23.9 °C, of control sample (Fig. 3, curve 4) during hydration is associated with emission dissolution heat and it is reached after 252 min.

In the second case, when the cement paste is poured to non-cooled, mineralized FHS, the temperature change curves of reached cement hydration temperatures have been provided in Fig. 4. It can be seen from Fig. 4 that higher complex mineralizer content not only elevates hydration temperature, but it also accelerates the hydration process.

It can be seen in Fig. 4, after pouring cement paste to non-cooled, mineralized FHS, the dissolution heat rose within increase of complex mineralizer content during the first stage. The temperature rises from 23 °C to 33.5 °C in case of formation mixture with 54% of complex mineralizer.

It shows that by increasing the amount of mineralizer, the higher part of $Ca(OH)_2$ participates in the process of dissolution, increases the dissolution temperature and stimulates cement hydration. It is well known that at higher temperatures cement minerals dissolve faster (Lothenbach et al., 2008). When the amount of mineralizer is lower, dissolution heat decreases, as can be seen in the case with 45% and 27% of complex mineralizer. The temperature rises from 23 °C to 30.5 °C in case of formation mixture with 45% of complex mineralizer (Fig. 4, curve 2), meanwhile in case of formation mixture with 27% of complex mineralizer (Fig. 4, curve 3) the temperature rises from 23 °C to 29.7 °C. It should be noted that peak of dissolution temperature is more evident in the compositions where lower amount of mineralizer is used. It can be presumed, that the lower amount of $Ca(OH)_2$ is used, the higher amount of ettringite is formed.

The higher complex mineralizer content was contained in the mixture, the shorter the induction period in samples was. The peak of EXO temperature, i.e. 35.7 °C, of formation mixture with 54% of complex mineralizer (Fig. 4, curve 1) was reached after 785 min. Comparing to samples where cement paste was poured to cooled mineralized FHS, the temperature was higher by 3.7% and the peak of EXO was achieved earlier by 26.2%. This data also confirms the presumption that some part of calcium from the mineralizer participates in the hydration process.

The nature of peak EXO temperature change curve in case of formation mixture with 45% of complex mineralizer (Fig. 4, curve 2) is different from analogous curve showing hydration reaction temperature of formation mixture with cooled FHS. Apparently the dissolution heat is higher in case formation mixture with non-cooled FHS and, therefore, temperature of 30.9 °C was reached after the first stage after 146 min. The peak EXO temperature, i.e. 30.8 °C, is reached after 982 min., i.e. earlier by 25.6% in comparison to analogous composition with cooled FHS. It should be noted that the temperature reached during the first stage was higher than the peak of the main EXO temperature. The nature of hydration temperature kinetic in case of mixture with 27% complex mineralizer (Fig. 4, curve 3) is similar to the sample with 45% complex mineralizer (Fig. 4, curve 2), i.e. the temperature reached during the first stage is higher than during the peak of the main EXO. Within the first stage, on the 190th minute of the test the temperature in the sample rises up to 29.7 °C, the peak of hydration reaction temperature (28.0 °C), and the curvature of the curve in the rage of ~600–1400 min is insignificant. Probably these differences are affected by different ratio of hydration products in the samples. That is confirmed by the results from research (Sedan et al., 2008), where it is mentioned that calcium silicate hydrates (CSH) are responsible for setting and, the higher amount of CSH, the faster setting occurs.

When composition contains FHS, the silicium concentration in cement paste increases, because it cannot precipitate with calcium to form calcium silicate hydrates (CSH) responsible for setting. Pectin which can trap calcium acts as a growth inhibitor for CSH hydrates which is the major hydration product of Portland cement. Pectin and consequently FHS by their calcium adsorption will prevent the precipitation of CSH and allow silicium to be retained in solution (Sedan et al., 2008). Therefore, the use of mineralizer can affect hydration process. The neutralization reaction is terminated when using cooled FHS, almost all Ca ions from complex mineralizer bond with FHS. It can be presumed that the rest Ca ions participate in the hydration reaction, more of portlandite forms and the temperature of EXO reaches the highest peak. When non cooled FHS are used, reaction of neutralisation is not terminated, temperature is high enough, more Ca ions which were added with complex mineralizer remain and, therefore, cement hydration occurs in different way, even faster.

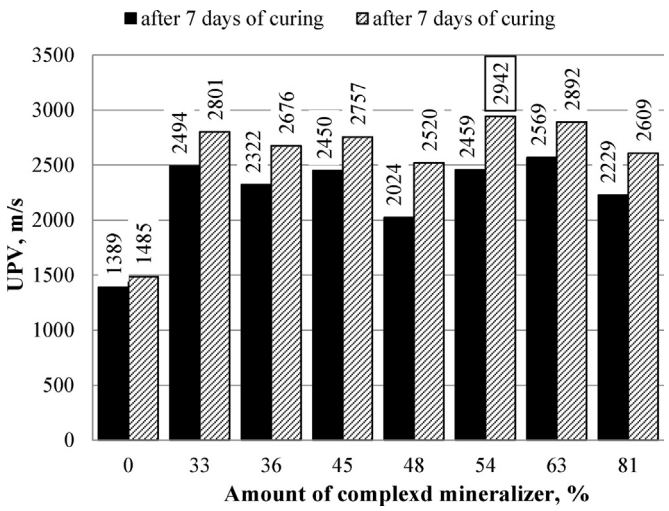

Fig. 5. UPV of the samples with diferent amount of complex mineralizer.

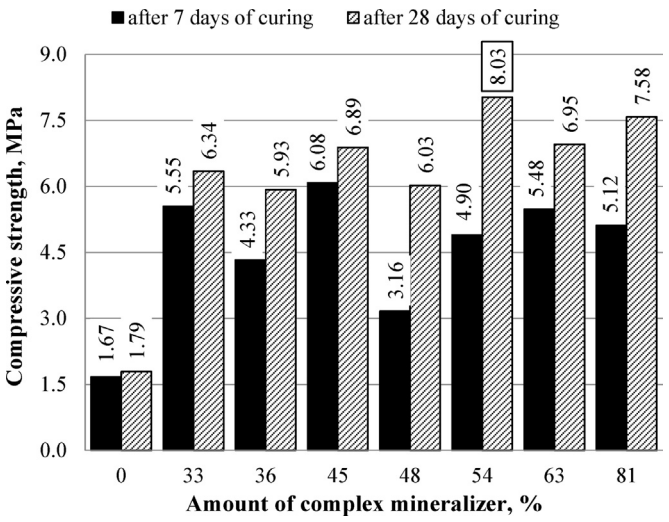

Fig. 6. Compressive strength of the samples with different amount of complex mineralizer.

Ca ions that did not bond with pectin during cement hydration process form a mixture of hydrated cement minerals in which, excluding portlandite, higher amount of different basicity CSH can be found. Because of this, the main EXO peak shows lower temperature and its beginning is earlier. Research which has been carried out by French scientists (Khazma et al., 2014) also confirms the importance of aggregate treatment. In summary, it can be proposed that hydration is stimulated by the Ca ions which comes as a part of the complex mineralizer. Higher amount of Ca ions determine faster cement hydration. According to the mentioned effect, the optimal forming mixture can be selected.

3.3. *Tests of UPV in samples with different contents of complex mineralizer*

For investigation of hydration and hardening structure development process, the UPV methods are often used recently. It is known that UPV can be used very effectively to monitor the hydration and formation of microstructure of cementitious pastes. Therefore, tests with 7 composite sample compositions containing different complex mineralizer content were carried out after 7 and 28 days of hardening. Based on the data in Fig. 5, it can be seen that complex mineralizer has a positive impact on the composite structure both after 7 and 28 days of hardening. Complex mineralizer allows increasing UPV values in samples up to two times comparing to samples containing no complex mineralizer, because complex mineralizer enhanced suitable conditions for cement hydration.

Based on UPV test data it can be concluded that optimum complex mineralizer content is 54% or 63%. UPV results deteriorated upon use of higher content of complex mineralizer or upon making changes in the ratio, therefore, as in case of composition with 48% of complex mineralizer. Apparently low complex mineralizer content does not ensure sufficient conditions for cement hydration, meanwhile higher complex mineralizer content does not ensure proper water access to the surface of cement particles because the dry matter of complex mineralizer uses a part of the water intended for cement hydration.

The compressive strength of composites was measured after 7 and 28 days of hardening. It can be seen in Fig. 6, the maximum compressive strength of the samples is achieved when 54% of complex mineralizer (estimated based on the FHS mass) is used for aggregate mineralization. The maximum strength of the samples in presence of the same content of binder and plasticizers is 8.03 MPa. It can be assumed that in cases when complex mineralizer content exceeds 54%, the water content is no longer sufficient for complete

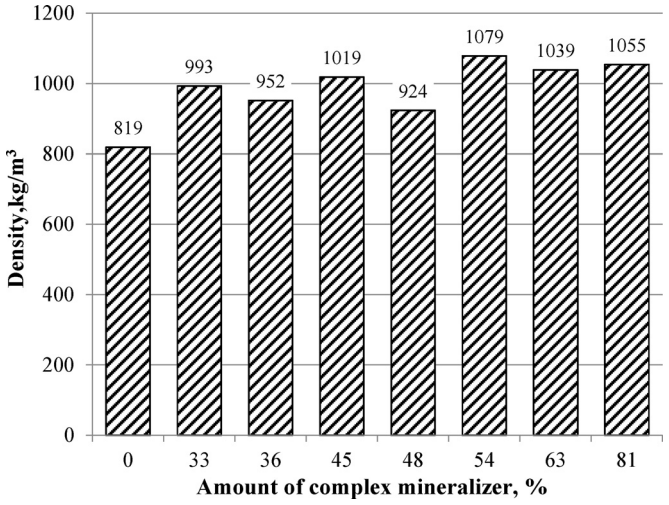

Fig. 7. Density of the samples with different amount of complex mineralizer.

cement hydration due to increase in dry matter characteristic in large quantity of fine particles. Upon reduction of complex mineralizer content in formation mixture up to 33% (estimated based on the FHS mass) the compressive strength achieved after 28 days is lower by 24% than in case of 54% of complex mineralizer content. It can be assumed that, if a lower lime content is used in the complex mineralizer, it is not sufficient to neutralize sugar released from FHS and this has a negative impact on cement hydration (inhibits it). This is confirmed by EXO analysis which shows that compositions with lower amount of mineralizer have reached lower values of EXO maximum temperature and it needs more time to reach maximum temperature. It can be assumed that 54% of complex mineralizer is the optimal for neutralisation of sugars, ensuring process of hydration and reaching high compressive strength. These assumptions are confirmed by the research of scientists (Sedan et al., 2014). Chemical treatment of the fibres surface by an alkaline and calcium rich solution degrades hemicellulose contained in the fibres and seems to roughen the surface. This surface modification seems to play a major role in the strengthening of the cement/fibres interface. These results show that hemp fibres introduced in cement pastes exhibit a typical composite behaviour compared to cement sample, which lead to an improvement of the mechanical properties. This means that the interfacial adhesion strength gets stronger after a chemical treatment. Spanish scientists (Jarabo et al., 2013)

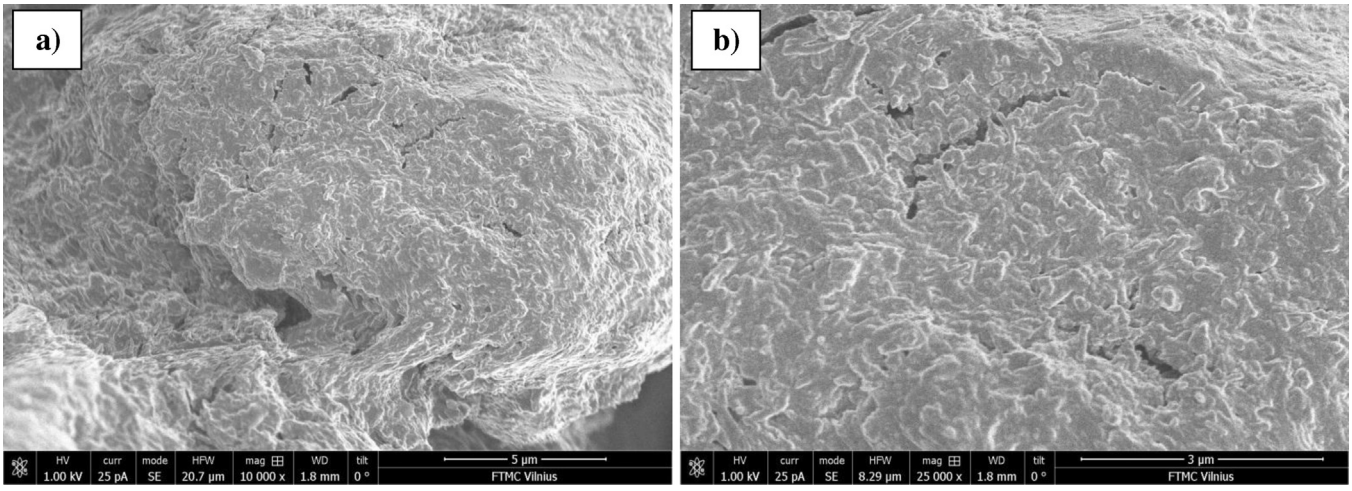

Fig. 8. Microstructure of the composite with non-mineralized FHS aggregate (a) magnification x10000, (b) magnification x50000).

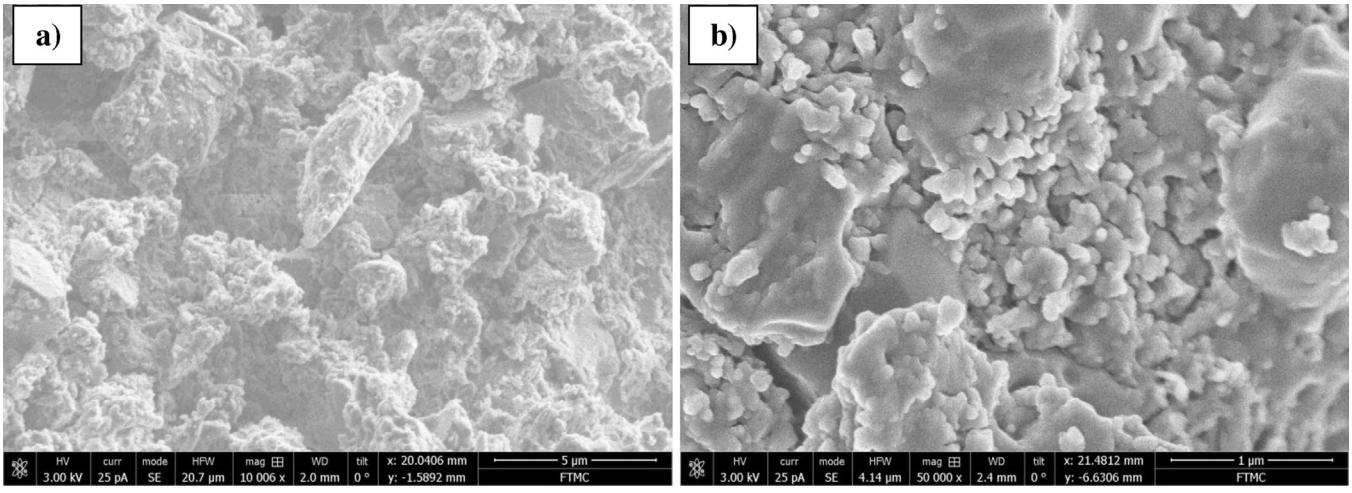

Fig. 9. Microstructure of the composite with mineralized FHS (amount of complex mineralizer is 27%) (a) magnification x10000, (b) magnification x50000).

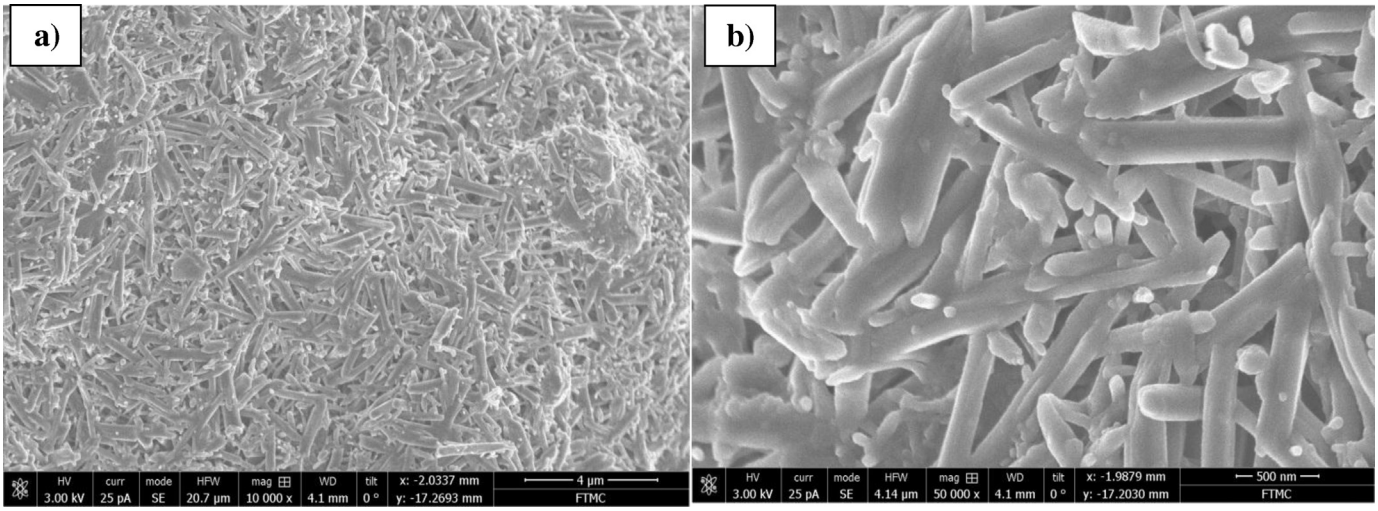

Fig. 10. Microstructure of the composite with mineralized FHS (amount of complex mineralizer is 48%) (a) magnification x10000, (b) magnification x50000).

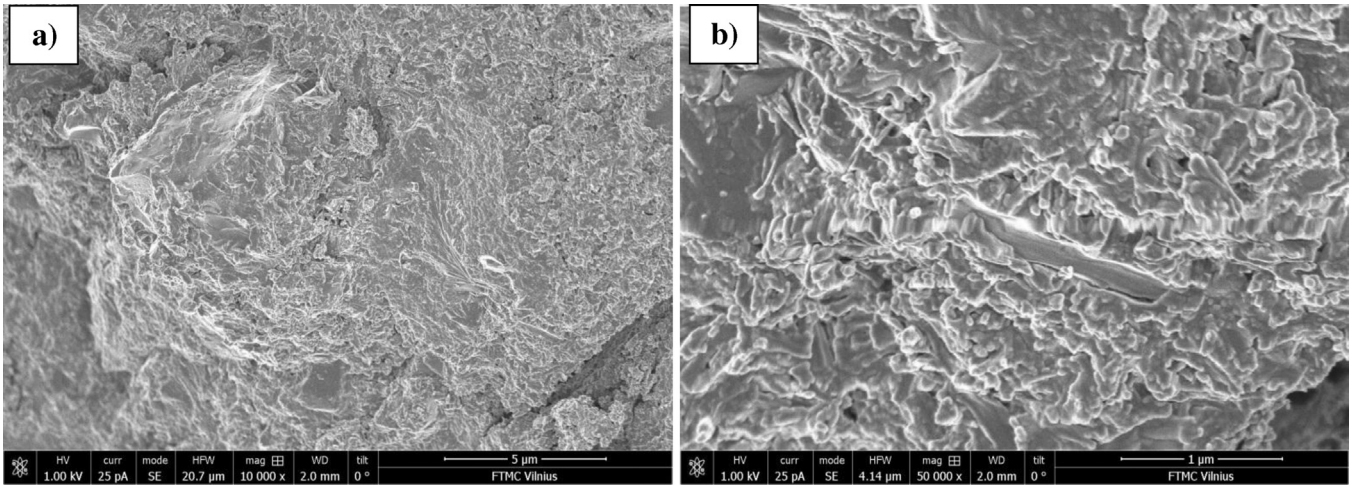

Fig. 11. Microstructure of the composite with mineralized FHS (amount of complex mineralizer is 54%) (a) magnification x10000, (b) magnification x50000).

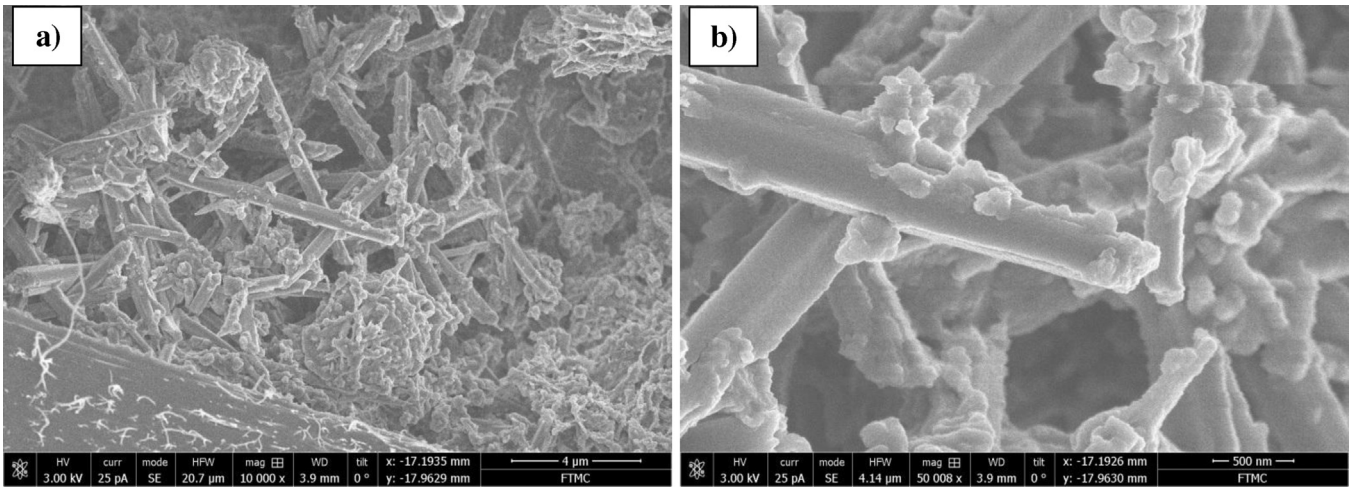

Fig. 12. Microstructure of the composite with mineralized FHS (amount of complex mineralizer is 63 %) (a) magnification x10000, (b) magnification x50000).

have confirmed the importance of treatment of the organic aggregate (corn stalk fibre), it is thought it degrades fibre surface which enhances the interaction among fibres and matrix (Sedan et al., 2008). Other scientists (Khazma et al., 2014) have stated that treatment of organic aggregates enhances its mechanical properties. Probably compressive strength is affected not only by previously mentioned factor but by forming of CSH as well.

It is found that the density of tested compositions samples ranged from 819 kg/m3 to 1079 kg/m3 (Fig. 7). Density of the control composition samples with non-mineralized FHS is the lowest. The highest density is characteristic to composition samples when complex mineralizer content is 54%. Having compared Figs. 5–7 it can be observed that the trends in changes of both compressive strength and UPV values are analogous to changes in density in case of composites with different complex mineralizer contents.

3.4. Microstructure

Microstructure of a substance determines physical and mechanical properties, in addition to this, thermal conductivity depends on it. Control sample of composite material (without complex mineralizer) and samples with 27%, 48%, 54%, and 63% of complex mineralizer have been selected for tests on microstructure. Cement matrix of microstructure of control sample can be seen from (Fig. 8) (without complex mineralizer), no hydration products or crystal compounds are observed. It is known from exothermic temperature tests (Fig. 3), there is no practical hydration in them because the peak of EXO reaction is not observed. Upon using minimum content of complex mineralizer, i.e. 27%, in the composite composition (Fig. 9), it can be seen that agglomerates and compounds of hydration products (new formations of non-defined form) are observed in the cement matrix. These compounds, according to the data of scientists (Khazma et al., 2014), could be hydrated calcium silicates and aluminates as well as portlandite and ettringite after 28 days of curing. The degradation of lignocellulosic aggregates in the forming mixtures with FHS could release carbon dioxide. This CO_2 leads to a carbonation of the portlandite. As it can be seen from compositions with FHS, the large amount of carbonates can produce. The portlandite can be observed in Fig. 9a and conglomerates of the cement minerals which are covered by germs of hydrates in Fig. 9b. This could be cement hydration minerals because a minor peak of EXO reaction was not observed based on Fig. 4. A considerable amount of disorderly set hydration products mix in the cement matrix (it can be assumed that this is a mixture of unshaped hydration products and probably ettringite needles) upon using 48% of complex mineralizer content in the composite composition

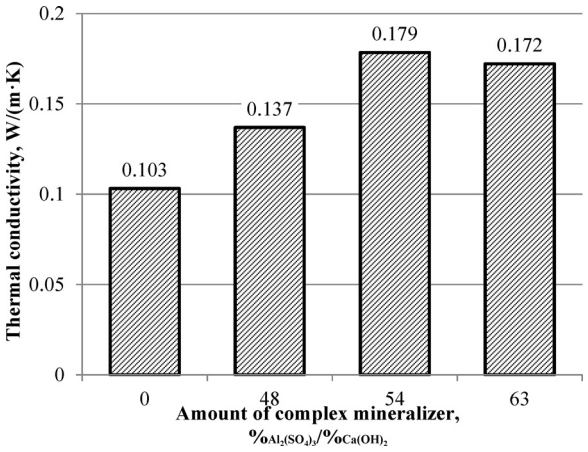

Fig. 13. Thermal conductivity of the composite with non-mineralized FHS and mineralized with 48%, 54% and 63% of complex mineralizer.

(Fig. 10) but upon changing the component ratio (1.7:1), i.e. upon reduction of $Ca(OH)_2$ content. This is also confirmed by UPV tests showing that UPV values after 7 days of hardening are the lowest in case of samples of this composition. The cement matrix is dense enough, well bonded monolithic hydration products of new formations can be seen, probably several ettringite needles can be observed upon using 54 % of complex mineralizer content in the composite composition (Fig. 11) without changing the component ratio (1:2). Hydration velocity in case of these samples is the highest (Fig. 4), therefore, the quantity of hydration products is high enough, and they formed solid adherent arrays of new formations. It can be observed that additional amount of Ca ions which comes from mineralizer stimulate the formation of higher quantities of compounds and probably the highest part of compounds are CSH.

It can be observed disorderly set ettringite needles abundantly covered in hydration products in the cement matrix upon increase of complex mineralizer content up to 63% (component $Al_2(SO_4)_3$ and $Ca(OH)_2$ ratio (1:2)) (Fig. 12) in the composition of the composite. It can be seen, the sample microstructure is obtained with more pronounced minerals in the shape of ettringite needles upon increase of the content of complex mineralizer component $Al_2(SO_4)_3$ up to 21% or 30% in the composition (6th and 4th and compositions). Such microstructure has a positive impact on test results in respect of thermal insulation properties, but it may have a negative impact on strength properties of the composite, as it can be seen from the data provided in Fig. 6.

3.5. Thermal conductivity

Thermal conductivity was measured in case of compositions of composite materials with different microstructure, i.e. control sample with non-mineralized aggregates, sample containing 54% of complex mineralizer characteristic of maximum compressive strength, and samples with needle-like microstructure with complex mineralizer content amounting to 48% and 63%. Thermal conductivity values have been provided in Fig. 13. As it can be seen in Fig. 13, the lowest thermal conductivity is characteristic to the sample where FHS aggregate has not been mineralized. There is almost no cement hydration in case of composite non-mineralized aggregate (marked as 0 on the chart) (Fig. 3, curve 4), therefore, no dense, homogeneous composite matrix formed, no new formations developed causing lower heat conduction through the sample. However, samples of the latter composition are characteristic of up to 4-fold lower strength comparing to mineralized aggregate (Fig. 6). This is confirmed by research of scientists (Khazma et al., 2014), who state that aggregate treatments increase thermal conductivity/apparent density ratio of the composites. It is as well stated that the incorporation of treated shives has involved an increase in apparent density and a less porous structure of the composites. These two effects should contribute to thermal conductivity increase (Ledhem et al., 2000; Khazma et al., 2011; 2012; Monreal et al., 2011 Monreal et al., 2011). In case of composites containing 54% and 63% of complex mineralizer, thermal conductivity values are similar and amounted to 0.179 W/(m K) and 0.172 W/(m K). In case of composite samples with 48% of complex mineralizer where the prevailing component is $Al_2(SO_4)_3$, the achieved thermal conductivity is much lower in comparison to samples where the prevailing component is $Ca(OH)_2$, i.e. samples with 54% and 63%. It can be assumed that microstructure in the shape of needles, similar to fibrous, causes a lower thermal conductivity (0.137 W/(m K)). Comparing to other light concrete, such as expanded clay concrete, the achieved thermal conductivity is significantly lower. For example, thermal conductivity in case of 800–900 kg/m^3 is 0.30 W/(m K) (Hens 2011), i.e. it is twice higher.

4. Conclusions

It is found that 0.9% (based on cement mass) polycarboxylate superplasticizer Glenium 430 is sufficient for ensuring the lowest viscosity values of cement CEM I 52.5R paste (W/C = 0.32).

Investigations of EXO reaction temperature for cement compositions with cooled and non-cooled mineralized FHS help to evaluate the role of complex mineralizer for cement hydration process. It is found that irrespective of whether a sample has been cooled or not, increasing of complex mineralizer in composition from 27% to 54% stimulates the cement hydration, shortens the time to reach EXO maximum and increases its temperature. However, in non-cooled samples time to reach EXO maximum is nearly a one third shorter than in cooled samples at the same EXO maximum temperature ranges. It was found that in non-cooled samples when increasing the amount of complex mineralizer from 27% to 54%, the bigger part of the $Ca(OH)_2$ participates in the process of dissolution, increases the dissolution temperature from 29 °C to 33 °C and stimulates faster EXO reaction from 1171 to 785 min in the samples. Lower content of complex mineralizer do not completely bind sugar released from FHS causing inhibition of cement hydration.

It is found that the maximum compressive strength which is 8.03 MPa of composite samples is achieved in case of 54% of complex mineralizer content (estimated based on FHS mass). Higher content of complex mineralizer has a negative impact on the mechanical properties of a composite because the total content of fine particles in the composite increase resulting in reduction of the amount of water able to react with cement. Content of complex mineralizer below 54% does not ensure sufficient cement hydration and reduces the compressive strength of samples after 28 days of hardening up to 30%.

UPV tests have shown that a more dense structure of a composite formed when the composition contained 54% or 63% complex mineralizer additive. UPV values decreased, a thinner structure is formed by using a higher content of it or by changing the ratio of the mineralizer components (in case of composition with 48% of complex mineralizer).

Upon studies made with composite microstructure it is found that there is no practical cement hydration in composite with non-mineralized FHS because no hydration products develop. In case of using mineralized FHS, cement hydration products (such as ettringite as well as agglomerates of new formations) are observed, possibly the development thereof is caused by cement mineral reaction with complex mineralizer. It is also observed that composites of different structure could be obtained by changing the ratio in the complex mineralizer: fibre-form structure of compos-

ite matrix could be achieved by increasing the content of $Al_2(SO_4)_3$ in the complex mineralizer, meanwhile a more dense structure composite structure could be achieved by increasing the content of $Ca(OH)_2$.

The developed composite is suitable for manufacture of thermal insulation-structural elements because it is characteristic of high compressive strength (up to 8 MPa) and almost twice as efficient thermal conductivity coefficient (up to 0.137 W/(m K)) as compared to expanded clay concrete.

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