

Evaluation of the contact angle and wettability of hydrophobised lightweight concrete with sawdust

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Abstract: The aim of the research presented in the paper was to evaluate the feasibility of using hydrophobic preparations based on organosilicon compounds for protection treatment on the lightweight concrete modified with sawdust. The experimental part of the work concerns the physical and mechanical properties of lightweight concrete and the influence of two hydrophobic agents on the contact angle of the material. Lightweight concrete contact angle (θ_w) was determined as a time function using one measuring liquid. Water repellent coatings in lightweight concrete structure with the coarse aggregate sawdust (CASD) using electron microscopy were presented. The effectiveness of hydrophobisation of porous lightweight concretes was determined on the basis of the research. For the hydrophobic surface, the contact angle decreased and it depended on the used agents. The lowest contact angle of 40.2° ($t=0$) was obtained for reference concrete before hydrophobisation and 112.2° after hydrophobisation with a methyl-silicone resin based on organic solvent. The results of scientific research confirm the possibility to produce lightweight concretes modified with CASD with adequate surface protection against external moisture.

Keywords: sawdust, lightweight aggregate-concrete, organosilicon compounds, hydrophobisation, contact angle

1. Introduction

A key global concern for societies, governments and industry, especially in the wood processing and production sector, is the sustainability of production systems. This sector is well placed to provide products that enhance long-term environmental sustainability through

products based on renewable natural resources from forests such as sawdust. Following the eco-trend in the nineteenth-century sawdust concrete was created. Due to its cost-effectiveness and lightweight, it has been well recognized [1–6]. To reduce the environmental impact, developed countries have now created opportunities for using wood waste in concrete structures. Besides sawdust in the production of building materials, the following waste materials from the wood industry is also used – wood shavings [7], wood fibre [8], wood ash [9–11], chips [12–14], cork [15], sawdust ash [16, 17].

Energy-saving blocks made from lightweight concrete are characterized by higher absorbability and they have much more pores compared to its trading equivalent, resulting from the porous structure of lightweight aggregate such as waste from the wood industry [18]. This is an essential problem when lightweight concrete mixes are designed. This causes the water transport, which is pulled up capillary (in the final product) [19], which significantly affects the heat flow process, increasing the thermal conductivity of materials several times [20, 21].

Damage caused by moisture is a major factor in the degradation of building materials [22–24]. Water repellent treatment is one of the methods of protecting the surface of lightweight aggregate-concrete [25, 26]. As a result of hydrophobisation, the absorption of capillary water decreases, which allows for proper water vapour permeability. In the case of impregnation, the so-called polymer cement concretes (PCC), obtained by adding polymer, oligomer or monomer to the concrete mix, gained popularity. This results in concrete with better workability of the mix and increased tensile strength compared to normal concrete [27]. In the case of existing facilities, polymer impregnated concrete (PIC) obtained by impregnating hardened concrete with a monomer or prepolymer is popular. For the hydrophobisation of concrete, organosilicon compounds such as methyl-silicone resins or siloxanes are mainly applied [28, 29].

The applications of silanes for surface modification of materials [30–32] or in the mass of concretes [33] and mortars [34–36] are known from the literature. These compounds make the concrete surface non-wetting by water and corrosive compounds, such as water-soluble salts [37]. The ability of building materials to be wetted by liquids is of particular importance, e.g. during their hydrophobisation, in the effectiveness of anti graffiti agents, during surface cleaning.

Materials' contact angle is an indicator of their wettability [38]. Higher wettability occurs at low contact angle (CA) $< 90^\circ$ and water repellency at high contact angle $> 90^\circ$. For determining the surface tension [39], the surface free energy (SFE) [40] and the adhesive action [41, 42] the contact angle can be used. CA is influenced by many factors. For example, CA is affected by the type of measuring liquid or droplet sizes of measuring liquids. The surface homogeneity, contamination and surface roughness, longitudinal elasticity coefficient of material which is being tested, ambient temperature or humidity also have an influence on CA [43, 44].

The most commonly used CA measurement methods include bubble method, geometric method, direct measurement and capillary liquid growth on a sample of the tested material [38]. A very popular method of contact angle determination is direct measurement. For example, a goniometer is used to test CA in this method but also a contact angle analyzer can be used [45, 46].

Based on the properties of waterproofing agents, there is a possibility to increase or reduce the CA and thus the material surface tension, resulting in their non-wetting properties, which is connected, inter alia, to frost resistance and chemical corrosion resistance.

Examining the structure of the topcoat of waterproofed lightweight concrete with CASD regarding wettability, among other things, it will allow evaluating the material's behaviour under the presence of corrosive compounds and water. In situations where significant resist-

ance of the surface layer of lightweight concretes to the corrosive environment is required, it is advisable to use preparations causing the highest contact angle.

2. Materials and methods

2.1. Concrete mixtures

The concrete mix consisted of CASD – lightweight with grain sizes from 8 mm to 16 mm, Portland cement CEM II 32.5 R, sand with grain sizes from 0 mm to 2 mm and water from the city's water distribution system. On the basis of standards EN 206-1:2003 [47] and PN-B-06265:2004 [48], an experimental formula was developed, which was used to produce specimens.

The following components must be used to make 1 m³ of lightweight concrete: CASD – 157 kg, sand – 1350 kg, cement – 350 kg, water 280 kg, superplasticizer 5.25 kg.

Once the components were thoroughly mixed and the concrete was placed in the moulds, the concrete was compacted on a vibrating table. Immediately upon mixing of concrete components specimens with the following dimensions 150×150×150 mm were made. Concrete in the moulds was laid in two layers, each of which was vibrated until the appearance of the cement grout. For 24 hours, all specimens were prevented from losing moisture and kept at a temperature of approximately 23 °C till they were removed from the moulds. The samples were then inserted into the water tank for 14 days. By the time of the test (28 days after the samples are made), the specimens had ripened under constant laboratory conditions.

In accordance with standard EN 197-1 [49], Portland cement CEM II 32.5 R has been tested. The parameters of the cement CEM II 32.5 R are shown in Tab. 1.

Table 1. Technical data of CEM II 32.5 R Portland cement. *Source: own study*

Parameters	Unit	
Specific surface	(cm ² /g)	3985
Initial setting time	(min)	248
Compressive strength after		
2 days	(MPa)	17.9
28 days		43.0
Density	(g/cm ³)	3.02
Loss on ignition by mass cement	(%)	5.0
Volume stability	(mm)	< 10

Polycarboxylate ethers were used as a superplasticizer in the amount of 1.5% by weight of cement. Its density at 20° C was 1.065 g/cm³.

The sawdust was collected from a local sawmill. They have been dried by drying in a laboratory dryer to remove any moisture that might affect the final cement-water ratio (w/c) of the concrete. They were then sieved through sieves to obtain the 8-16 mm fraction for the production of coarse aggregate sawdust (CASD) with a specific gravity of 0.36 (g/cm³), a water absorption capacity of 89% and a grinding module of 3.79.

2.2. Methods

The physico-mechanical properties of concrete with CASD have been established in accordance with the following standards: EN 1936:2010 [50], PN-EN 1389:2005 [51], EN 12390-7:2019 [52], EN 12390-3:2019 [53], PN-B-06250:1988 [54].

2.3. Applied water repellents

For laboratory testing, two commonly used hydrophobic agents on the market have been chosen, differing in solvent type, concentration and viscosity:

HA1 – water-based methyl-silicone resin in the potassium hydroxide,

HA2 – methyl-silicone resin based on organic solvent.

In Tab. 2, the primary characteristics of preparations that were used in the study are presented.

Table 2. Properties of hydrophobic agents. *Source: own study*

Type of formulation	Surface tension σ (N/m · 10 ⁻³)	Viscosity η (Pa · s · 10 ⁻³)	Density at 20°C (g/cm ³)	The quotient of the surface tension and viscosity σ/η
HA1	67.92	1.10	1.20	61.73
HA2	24.30	2.85	0.85	8.54

The lowest viscosity coefficient η has a formulation with the lowest concentration of the active substance and it is an aqueous solution of HA1 formulation. Methyl-silicone resin in petrol HA2 is characterized by higher viscosity by almost 250% compared to that of preparation HA1. The highest value of the ratio of surface tension to the viscosity of solutions is shown by the water-based preparation HA1.

The study did not analyse the concentration of the preparation and the number of layers applied. It should be assumed that these factors influence the penetration capacity of the preparations and at the same time the contact angle and effectiveness of hydrophobisation.

The agents were applied twice by brush, one layer after another, so as not to interrupt the capillary action ('wet on wet' method).

2.4. Evaluation of the contact angle

The contact angle of the measuring liquid droplet was determined on a test stand which consists of a goniometer. The goniometer was integrated with the camera in order to take pictures of the droplets placed on the samples surface. Fig. 1 shows the measurement of the CA of reference samples and samples protected by hydrophobisation at the beginning of the test ($t_1=0$ s).

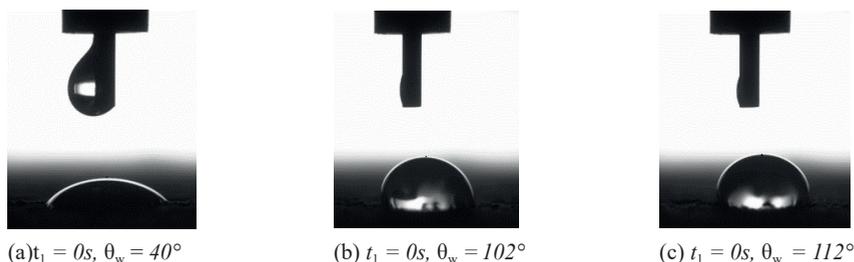


Fig. 1. Lightweight concrete surfaces during CA test: (a) reference sample S, (b) sample protected with agent HA1, (c) sample protected with agent HA2. *Source: own study.*

For testing the CA, distilled water has been selected as the measuring liquid. The drops of liquid with a volume of 1.8 mm³ were applied with a micropipette [38, 55]. Due to the heterogeneity of the material, 6 drops were applied to each sample. Measurements were taken 3 times: at the time of application of the drops and after 5 and 45 minutes.

2.5. Scanning electron microscopy (SEM)

The morphology and microtopography of water-repellent concrete with CASD were analysed using FEI Quanta 250 FEG scanning electron microscopy. The device was equipped with a chemical composition analysis system which was based on energy dispersion spectroscopy (EDS). The specimens have been formed in thin-layer tiles. They were subjected to X-ray microanalysis under field conditions and the composition of the elements for concrete were assessed.

3. Results and analysis

3.1. Characteristics of lightweight concrete with CASD

Mechanical and physical characteristics of the studied concrete are shown in Tab. 3.

Table 3. Properties of lightweight concrete with CASD. *Source: own study*

Density ρ_d (kg/m ³)	Apparent density ρ_a (kg/m ³)	Tightness (%)	Porosity (%)	Compressive strength (MPa)	Flexural strength (MPa)
2550	1504	59.0	41.0	26.6	5.9

The absorbability values after 1, 3, 7 and 14 days of concrete are shown in Fig. 2.

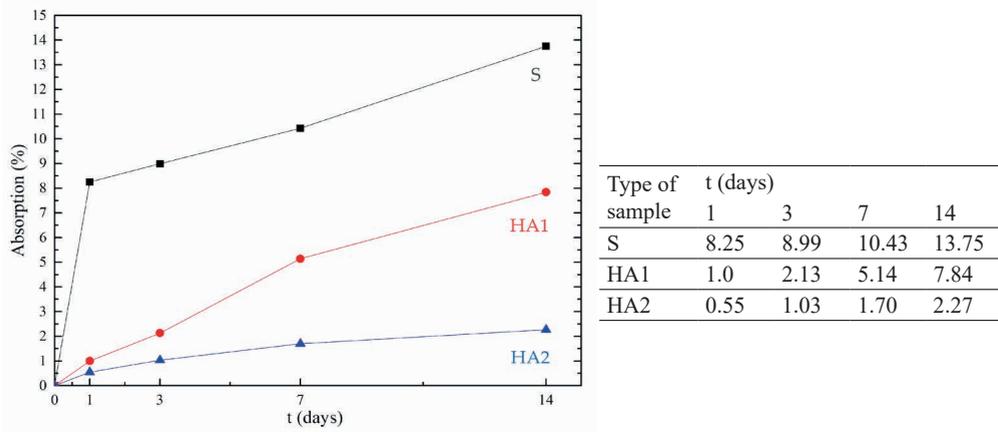


Fig. 2. Absorption of concrete after 1, 3, 7 and 14 days. *Source: own study*

Analysing the above data, it can be seen that after 14 days of the water absorption test the reference concrete increased its mass by 67%. This is due to the porosity of the concrete with CASD, which was as much as 41%. This shows the need for concrete modifications to reduce

water absorption. Thus the surface water repellent treatment was applied. The best results were obtained for concrete protected with HA2. After the first day, it reduced the water absorption of concrete by 93% and after 14 days by 83%. On the last day of the test, absorbed water by concrete samples with CASD was 3.5 times lower for HA2 than for HA1.

Thalmaier G. et al. [56] made five different types of clay brick samples. The content of sawdust in the specimens was 15% for all, but they differed in the size of the sawdust fraction used to make them. They found a significant increase in water absorption from 23% to 25% depending on the sawdust fraction used. While the water absorption of the reference samples was 18%. It shows a significant influence of porosity, which was 25% and up to 40% for reference samples and those with sawdust, respectively. In their research, Md Noor et al. [57] examined water absorption of concrete with palm oil kernel shells (PKS). After the research, they found that water absorption values range from 4.1% to 4.7% for the reference concrete, but increased from 4.5% to 12.1% when 25% to 75% of the coarse aggregate was replaced by PKS. This is due to the porous structure of PKS. Oba and Amadi [16] in their work created a mathematical model for water absorption of Sawdust Ash (SDA) – Sand Concrete. They replaced the fine aggregate with 5% SDA in concrete and thus achieved a concrete water absorption rate of 3.734% to 9.568%. In the paper, Castro et al. [58] showed that water absorption by concrete may also depend on the way of sample conditioning and of the volume of paste in the samples. They have shown that specimens conditioned at 50% relative humidity can exhibit almost six times higher total water absorption than similar specimens at 80% relative humidity.

The porosity of the material is the main factor on which the effectiveness of hydrophobisation depends. Water repellency is more effective as the porosity of the material increases. This is because it reaches deeper parts of the material. Szafraniec et al. in [59] showed the effectiveness of surface hydrophilisation of porous mortars with perlite. Samples surface-treated with aqueous emulsion based on organofunctional silanes diluted in demineralised water in the ratio of 1:4 proved to be the most resistant to water absorption. Absorption of water was 7.5 times lower after 1 day and 2.9 times lower after 14 days in comparison with the reference mortars. Research conducted by Suchorab et al. [60] shows that lightweight aggregate concrete with sewage sludge, thanks to its porosity and absorbency, has absorbed more water repellent. This reduced the water absorption of this concrete by about 57%. Zhu et al. [31] concluded that surface hydrophilisation of concrete with recycled aggregate reduces capillary water absorption by up to 96% and improves resistance to chloride penetration and carbonation.

3.2. Contact angles of water

Average wetting angles CA of water from six measurements are shown graphically in Fig. 3. Analysing the test results presented in Fig. 3, it can be seen that the values of contact angles depend on the type of hydrophobic agents.

The results of contact angle measurements showed that in all cases the contact angle (θ_w) decreases with time. The smallest contact angle $\theta_w=40.2^\circ$ was obtained for standard samples at $t_1=0$ and the largest $\theta_w=112.2$ at $t_1=0$ for HA2, which indicates very good surface water repellency obtained by this preparation. The highest decrease by 57.2% of wetting angle value equal to $\theta_w=23^\circ$ was observed after 45 minutes from applying water droplets on the reference specimens. Regarding hydrophobic concrete treated with HA2, the contact angle was reduced slightly by 7.75%. The CA for HA1 decreased after 45 minutes of testing by 19.65%.

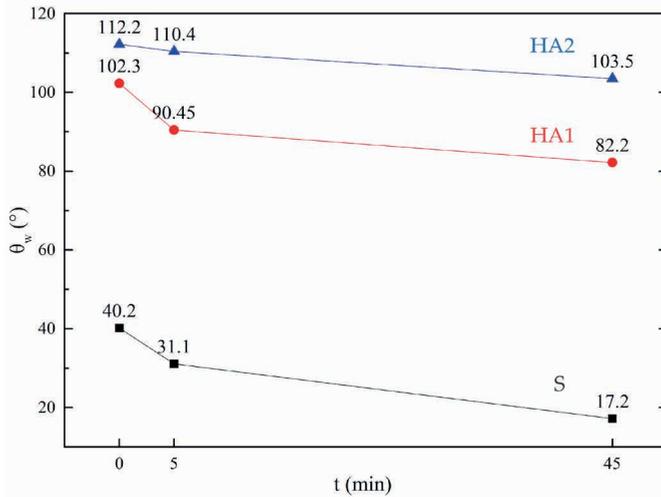


Fig. 3. CAs for lightweight concrete containing CASD. *Source: own study*

The use by Barnat-Hunek et al. [61] of alkyl-alkoxy-siloxanes for surface hydrophobisation of porous mortar with expanded cork resulted in 2-3 times higher contact angle compared to unprotected samples. Qu and Yu [62] added ground granulated blast furnace slag as a waterproof admixture to lightweight concrete. This resulted in a contact angle of 92°. Barnat-Hunek et al. [63] in their work showed that surface waterproofing of lightweight concrete with sewage sludge with organic solvent-based methyl silicone resin increased 60-75-fold the contact angle values (compared to reference samples). A superhydrophobic concrete (S-concrete) was created in the work of Lei et al. [64]. By using copper mesh and stearic acid they achieved a contact angle of 159°. Contact angle at this level has been achieved because the micro-nano-structure of S-concrete has created a stable air bubble which is trapped between the droplet and the micro-nano-composite structure. It stops the droplet and prevents the specimen surface from wetting. Fic and Szewczak [65] have tested the effectiveness of hydrophobisation of building ceramics protected by polymers which have been disintegrated by ultrasound. Moreover, to the polymers, a filler in the form of microsilica was added. In their research, they determined the contact angles for two liquids (w - water and d - diiodomethane). For samples hydrophobised in pure (non-disintegrated) silicone solution, the average θ_d value was 92° and the θ_w value was about 125°. In the case of samples hydrophobised in the aqueous polymer solution, which has been disintegrated by ultrasound for 15 minutes, the θ_d value increased to 95°, with a θ_w of about 129°. For this reason, the developed coating is more effective as it prevents the absorption of both polar liquids (water) and organic solvents (apolar). For samples immersed in a disintegrated aqueous silicone solution with the addition of microsilica, there was a significant decrease in the θ_d value, while the θ_w value remained above 100°. This indicates the preservation of the hydrophobic character of the obtained coating.

3.3. SEM microstructural analysis

In order to determine the effectiveness and distribution of waterproof surfaces HA1, HA2, microscopic SEM analyses were performed. Fig. 4 shows the microstructure of the concrete.

The hardened mortar made of Portland cement consists mainly of about 67% of hydrated calcium silicates, the so-called C-S-H phases, about 22% of calcium hydroxide and products of hydration of aluminium and calcium aluminate ferroaluminium. Microscopic examination at the interface between cement mortar and CASD demonstrated good adhesion. No cracks or cavities in the joint between the two materials. Minor amounts of needle-shaped ettringite crystals are found (Fig. 4). CASD influenced the increased porosity of concrete compared with ordinary concrete.

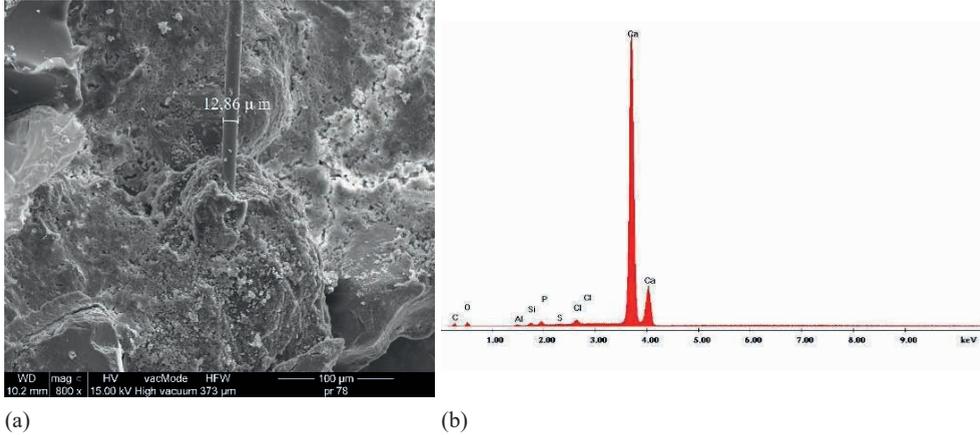


Fig. 4. The scanning microscope structure of lightweight concrete with CASD (a) and results of elemental analysis in EDS micro-area (b) – compact clusters of C-S-H phase particles can be shown. *Source: own study.*

The distribution of polysiloxane gel in the structure of lightweight concrete with CASD is shown in Fig. 5.

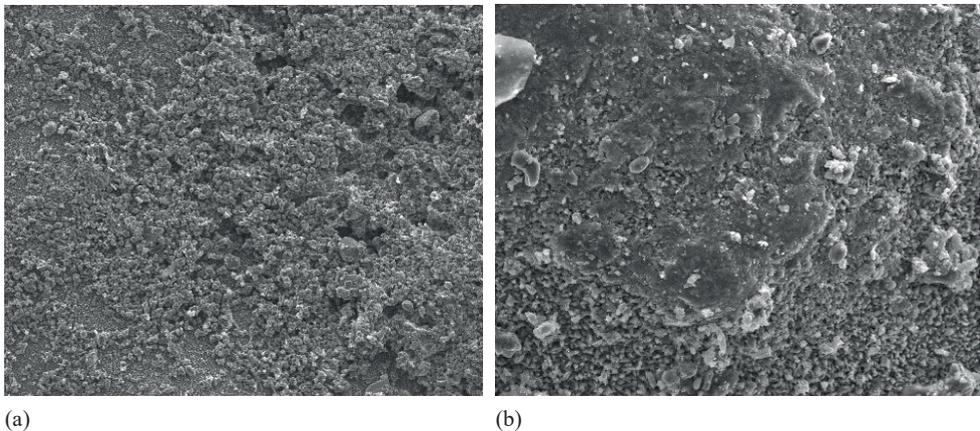


Fig. 5. The structure of hydrophobised concrete with CASD: (a) water-soluble preparation HA1, (b) methyl-silicone resin HA2. *Source: own study*

On the structure of the reference samples, the products used produced an evenly distributed coating without visible cracks or scratches. Agent HA2 created a silicone coating which

covers the concrete structure most accurately (Fig. 5b). It has sealed the surface pores in this way, which may cause the porosity of the mortar to decrease slightly. In the case of CASD, their porosity remains unchanged. The preparation HA1 with an aqueous solvent has created a continuous and very thin film (Fig. 5a). HA1 has lessened the structure of lightweight concrete, which was also shown by CA measurements of a drop of water, which fell by 20° after 45 minutes, while in the case of HA2 only by 8.7°. The decomposition of both preparations does not cause too much sealing of the pores, so they should not interfere with steam permeability and diffusion.

In the paper by Qu and Yu [62], SEM images showed that samples containing 5% and 10% of GGBS have a denser structure. As the amount of GGBS increases, more pores appear in the concrete structure. With 20% addition of GGBS pores larger than 20 µm occurred in lightweight concrete. The SEM images shown in the work by Yoon et al. [66] pointed out the presence of calcium hydroxide and calcium silicate gels in conventional foam concrete. However, in the specimens of foam-concrete embedded in the aerogel, a large distribution of spherical nanoparticles was observed on the surface of pores and products of hydration. The structure of S-concrete shown in the study Lei et al. [64] presents a hierarchical micro-nano-structure and irregular fibres at the nanoscale. This structure contributes to the roughness of the sample surface. Furthermore, it was proved that stearic acid contributed to reducing the surface energy of the concrete and preventing the penetration of water into the concrete. Meso and micromorphology of concrete surface treated with control and superhydrophobic (SC) emulsion are presented in the paper by She et al. [67]. In the SEM images, specimens had a rough surface and consisted of multi-plane phases. The modified surface is composed of mechanically reinforced micro-pillars with a nano-roughness structure on them. The surface tomography was similar in meso- and micro-meter scale, but on the surface of concrete treated with SC there were more phases in the nanoscale. The researchers proved that the addition of silica can decrease the Ca/Si ratio of cement hydrate in the surface layer, thus improving mechanical properties. Franczak-Balmas in her study [68] showed how roughness is an important factor. She analysed the influence of roughness of the contact surface as a parameter shaping the load-bearing capacity of the joint of the composite concrete elements. In her paper, she proved that mechanical adhesion is a key component of the contact load-bearing capacity because as the surface roughness increases (development of the contact area), load-bearing capacity increases by 60%.

4. Conclusions

The presented studies showed that sawdust in the form of CASD can be used as additive in lightweight aggregate production. CASD can be effectively used in lightweight concrete production, which is proven with the results of physical and mechanical properties tests. The microscopic examination showed good adhesion of the contact points of cement paste and lightweight aggregates. Because of the higher absorbability and porosity caused by the CASD content in concrete, it is necessary to apply anti-moisture treatment on the surface of concrete in the form of hydrophobisation.

Measurement of the contact angle is one of ways of controlling wettability development of hydrophobised building materials. Application of different preparations leads to different wetting and adhesion properties of concrete. Lightweight concrete surface hydrophobisation has been observed to contribute to the formation of different CA. The value of CA grows noticeably on the water-repellent surface, especially with the organic solvent methyl silicone resin (HA2). For non-hydrophobic concrete, the CA values are 3-6 times lower than for the impregnated surface.

Introduction of organo-silicon compounds into the surface zone of lightweight concrete results in a reduction of absorbability which depends on the chemical structure of the preparations. This has the effect of limiting the penetration of corrosive substances into the structure of concrete, i.e. soluble salts, toxic gases, and thus influences its durability.

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