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Kinetics of the hydration process and rheology of cement pastes containing mechanically activated chalcedonite powder

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Abstract. Reducing the amount of Portland clinker in the cement composition is one of the key aspects of the strategy aiming to reduce carbon dioxide emissions. One solution is to use waste materials for this purpose, for example, stone fine powder, especially limestone, basalt, granite, and melaphyre powder. There is insufficient information about the use of chalcedonite powder as an addition in cement or cement composites. Also, there is no precise information about the impact of this material on the particle size and the resulting changes in the properties of cement pastes. Therefore, this study investigates how the particle size and amount of chalcedony powder affect the hydration processes and rheological properties of cement pastes. The addition was used at 10%, 20%, and 30% by weight of the cement, with grain sizes up to 10 µm, 20 µm, and 36 µm, respectively. The hydration kinetics of the pastes were assessed based on calorimetric measurements, supplemented by DTA-TG analysis and mechanical properties tests. Additionally, the rheological properties were determined. The study demonstrated that chalcedony powder affects the tested parameters to varying degrees. In rheological research, the cement paste containing the largest grain size (CHP36) chalcedonite powder showed the most similar properties to the CEM cement paste (without the addition). In contrast, the pastes modified with chalcedonite powder, in which the cement was replaced by 10% of the additive, exhibited the highest amount of cumulative heat. In terms of 28-day compressive strength, the materials containing CHP20 powder demonstrated the most similar properties to the reference paste, without the addition.

Keywords: carbon dioxide emissions; cement paste; chalcedonite powder; waste; particle size; hydration processes; rheology.

1. INTRODUCTION

Currently, material modifications in the production of cement, mortar, and concrete are intricately linked to global industrialization, climate change, natural resource management, and government regulations [1–3]. The main challenge in the cement industry is the need to reduce greenhouse gas emissions associated with cement clinker production. Proposed solutions involve introducing changes to production processes in cement plants, for example, using alternative fuels, technologies of CO_2 capturing, high-performance classifiers, more efficient grinding technologies, as well as modifications to the composition of the binders themselves [1, 3–5]. The use of cement with additions and low-clinker cement is a solution that allows for reducing the clinker content in cement compositions [4, 5]. Additionally, the process of mechanical activation of cement components may have a beneficial effect on accelerating the hydration process, increasing the pozzolanic activity of non-clinker cement components, or improving mechanical properties [6, 7]. In addition to commonly used mineral additives, researchers are seeking other alternative materials that demand low energy consumption in production processes and enable the efficient use of waste. Stone powders are natural mineral materials, usually subjected only to mechanical processing, enabling the production of materials with varying grain sizes depending on the needs [8–11]. The literature provides information on the effects of limestone, basalt, granite, marble, and melaphyre powders [8–16]. Chalcedonite has so far been used mainly as a partial substitute for addition and fine aggregate in ordinary concretes [17], a component of reactive powder concrete [18], and a partial substitute for binders in pastes and mortars [19–21]. It is also used in other industries as an ingredient in biodegradable polymer composites [22, 23]. However, there is insufficient information on its effect on the properties of pastes, mortars, and concretes, in which it acts as a partial cement substitute. Literature analysis indicates that it affects the consistency, rheological properties, cement hydration process, and mechanical properties, among others [17–21]. Studies conducted to date focus solely on assessing the effect of chalcedonite powder or chalcedonite sand on selected properties of cement composites. The effect of its grain size on the standard or functional properties of the materials has not been analyzed yet. The authors of the article [18] applied the chalcedonite fillers of fraction 0/250 µm and 250/500 µm and proved that chalcedonite-based concrete with a smaller amount of cement had decreased compressive strength and frost resistance. On the other hand, the strength values were sufficiently high to consider this substance to be high-performance concrete.

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Chalcedonite dust with a maximum grain size of 72 μm in the research [17] was used to check the impact on concrete parameters resulting from the designed concrete classes C30/37, C35/45, and C40/50. Chalcedonite powder influenced the average compressive strength reduction of 10%, but all series of concrete modified with chalcedonite powder achieved the assumed strength classes. Analysis of the rheological properties of cement pastes with 10% and 20% chalcedonite powder with a grain size of up to 10 μm shows that this addition increased the yield stress and consistency coefficient and decreased the mini-slump flow in comparison to the paste without it [20]. Based on literature studies, it can be concluded that chalcedonite powder affected the tested physico-mechanical properties to varying degrees. Due to its grain size, this addition reduced consistency (reduced flow, cone penetration, and mini slump cone), which is also noticeable by increasing the consistency coefficient. Calorimetric measurements showed lower heat release dynamics for pastes modified with chalcedonite powder, with varying effects on the compressive strength of the tested materials.

This article focuses on determining the effect of both the quantity and grain size of chalcedonite powder on cement hydration processes and the rheological properties of pastes, partially replacing cement with chalcedonite powder. For this purpose, calorimetric and thermogravimetric studies were performed, supplemented by an assessment of the mechanical properties of the pastes. The yield stress, consistency coefficient, and thixotropy were assessed using rheological tests, which were supplemented by an examination of the paste consistency. The impact of the additive on cement hydration processes was also determined. Based on this, the interaction between cement and chalcedonite powder and its effect on the kinetics of reactions occurring in the cement paste was determined. This information is important for both a scientific and practical perspective, as well as for a comprehensive analysis and determination of the potential use of chalcedonite powder in cementitious composites.

2. MATERIALS AND METHODOLOGY OF RESEARCH

2.1. Materials

2.1.1. Materials characterization

The ingredients used in this research included ordinary Portland cement CEM I 42.5R NA, chalcedonite powder, and water. Chalcedonite waste was produced from chalcedonite rock. Deposits of this rock are located in Poland, in Inowłódz, near Tomaszów Mazowiecki. The material used for this research was only prepared in mechanical industry processing, characterized by different grain sizes with maximum 10 μm, 20 μm, and 36 μm (name of additions: CHP10, CHP20, CHP36).

The chemical composition of cement and chalcedonite dust is introduced in Table 1. It was measured with an Axios X-ray fluorescence (XRF) spectrometer, Malvern Panalytical Ltd. The percentage quantity of all components of cement is typical for this binder. As one can note (Table 1), all 3 types of chalcedonite powder have similar compositions, characterized by a quantity of over 98% of SiO_2. The mechanical properties of cement were assessed. The research was performed according to PN-EN 196-1.2-days, and 28-days flexural strength of cement was 5.13 MPa and 8.60 MPa, but 2-days and 28-days compressive strength was 22.43 MPa and 48.54 MPa.

2.1.2. Methods for determining physical properties of materials

The specific density of materials was determined using the pycnometric method according to PN-EN 1097-7 and the specific surface area using the Blaine method according to PN-EN 196-6. Granulometry of ingredients was measured using a laser particle sizer (HELOS KR, company Sympatec GmbH). Granulation of all additions was smaller than that of the cement (Table 2, Fig. 1). The specific density of cement was 3.11 g/cm^3, of CHP10 was 2.66 g/cm^3, and of CHP20 and CHP36 was 2.67 g/cm^3. The specific surface of cement was 2990 cm^2/g. CHP36 had the smallest specific surface, equal to 7910 cm^2/g (comparing only the addition with different grain sizes). The remaining waste powders (CHP10 and CHP20) were characterized by a specific surface area equal to 16 590 cm^2/g and 12 950 cm^2/g.

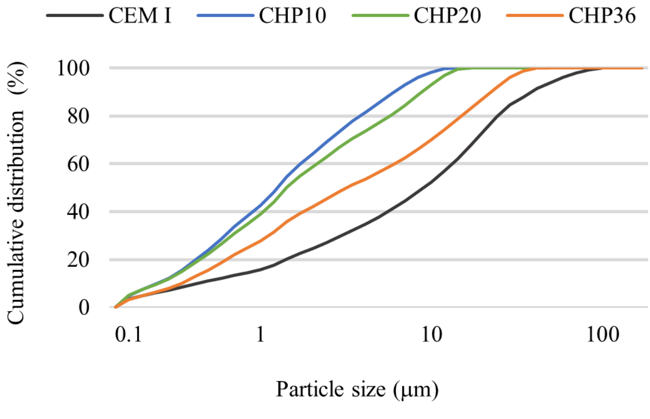

Fig. 1. Particle size distribution of ingredients

Table 1
Chemical composition of materials, [%]

Material	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	TiO_2	MnO	P_2O_5	L.O.I.
CEM I	20.49	3.98	4.19	65.54	0.73	2.90	0.56	0.37	0.31	0.09	0.28	0.56
CHP10	98.90	0.90	0.04	0.05	0.02	–	0.03	0.04	0.02	0.00	0.00	0.00
CHP20	99.00	0.80	0.04	0.04	0.03	–	0.03	0.04	0.02	0.00	0.00	0.00
CHP36	98.89	0.88	0.05	0.06	0.02	–	0.03	0.04	0.02	0.00	0.00	0.01

Table 2
Information on the median diameter of selected particles of cement and chalcedonite powder

	CEM I	CHP10	CHP20	CHP36
d_{10} (µm)	0.45	0.27	0.27	0.36
d_{50} (µm)	9.25	1.29	1.49	3.34
d_{90} (µm)	39.92	6.24	9.04	22.83

2.1.3. Method for determining pozzolanic activity of chalcedonite powder

Additionally, the pozzolanic activity of additions was determined by the chemical method called the Chapelle test [24, 25]. It should be noted that according to the Chapelle method, material is considered to be pozzolanic active if its reactivity after 1 day of measurement is at least 650 mg $Ca(OH)_2$/g. According to this research, each chalcedonite powder exhibited pozzolanic activity after just 1 day (Table 3). The smaller the granulometry (Fig. 1), the higher the activity of the addition.

Table 3
Pozzolanic activity of chalcedonite powder, (mg $Ca(OH)_2$ g^{-1})

Type of CHP	After 1 day	After 3 days
CHP10	832	1414
CHP20	1166	1419
CHP36	727	1371

2.1.4. Preparation of pastes

Ten types of pastes were prepared for testing. Base paste CEM, consisting only of cement and water, and nine pastes in which cement was replaced in amounts 10%, 20% and 30% by weight with the chalcedonite powder of various grain sizes: CHP10, CHP20, CHP36, respectively. The water-to-binder ratio was constant for all pastes and was 0.5. The composition of all binders used for research is shown in Table 4. The pastes under rheological and consistency tests contained 90 g of binder and 45 g of water. The samples for calorimetric measurements consisted of 4 g of binder and 2 g of water. The pastes under strength tests and DTA/TG analysis contained 160 g of binder and 80 g of water. Before each test, the dry ingredients were pre-mixed, and then the specified amount of water was added.

Table 4
Percentage composition of binders

Type of paste	CEM [%]	CHP10 [%]	CHP20 [%]	CHP36 [%]
CEM	100	–	–	–
CH10_10	90	10	–	–
CH10_20	80	20	–	–
CH10_30	70	30	–	–
CH20_10	90	–	10	
CH20_20	80	–	20	
CH20_30	70	–	30	
CH36_10	90	–	–	10
CH36_20	80	–	–	20
CH36_30	70	–	–	30

2.2. Methodology of research

Figure 2 shows all types of tests introduced. As can be seen, the research consisted of two main parts: the assessment of the properties of the pastes in the plastic state and the evaluation of the setting and hardening processes at different stages of material curing. The rheological properties of the pastes were compared. Each time ingredients were mixed, the sample was placed into the cylinder of testing (rheometer Discovery HR-1, company TA Instruments), and the remaining part was used for the test of consistency. The hybrid rheometer was equipped with a Peltier Concentric Cylinder system with a DIN rotor (standard DIN cylinder system of 5.917 mm). The temperature during the measurements was 20°C. Yield stress (τ_0), consistency coefficient (K), and fluidity index (n), among others, were determined in accordance with the Herschel-Bulkley model [26]. Thixotropy of the samples was determined using the TRIOS software, considering the hysteresis area between ascending and descending flow curves. For each type of paste, 3 results were compiled, introducing the measurements taken after 5, 30, and 60 min.

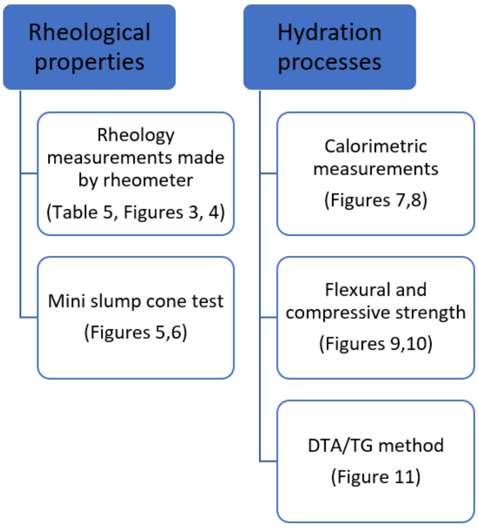

Fig. 2. Diagram of research methods

The shear rate changed in range firstly from 1 s^{-1} to 100 s^{-1} and secondly from 100 s^{-1} to 1 s^{-1}. The mixing method of pastes was as follows: 1 minute of mixing manually → 2-minute break → 1-minute of mixing manually → 1-minute break, and measurement. In the consistency test – mini slump cone test [27], the flow diameter (average of 4 measurements) and the height of the sample (average of 3 measurements) were determined. An electronic caliper was used for measurements.

The heat of hydration was measured in a TAM Air eight-channel isothermal microcalorimeter (TA Instruments company). Each time, dry ingredients and water were stored in ampoules in a calorimeter for 24 h in order to achieve thermal equilibrium, then they were mixed, and the measurement was started. The hydration processes of pastes were monitored for 72 hours.

Mechanical properties of pastes were determined after 3 and 28 days of curing. All ingredients were mixed manually for 1 minute. For this purpose, samples of moulds with dimensions of 20 mm × 20 mm × 100 mm were formed. After forming, the samples were stored for 24 h in air-dry conditions (protected with foil against excessive water evaporation) and then seasoned for two or 27 days in containers, in water. Flexural strength research was determined using a 3-point flexural test. The cross-section of the bending surface had dimensions of 20 × 20 mm, but the spacing between the supports was equal to 70 mm. The compressive strength test was performed on halves of the beams, where the compressed surface was a square with a cross-section of 20 × 20 mm. The flexural strength was determined as the average of 3 measurements for each type of paste, and the compressive strength was calculated as the average of 6 results. All tests were performed at a temperature of 20 ± 2°C. After testing the mechanical properties, samples were taken for DTA/TG measurements (STA 2500 Regulus thermogravimeter, company NETZSCH GmbH & Co.).

Each hardened paste was manually ground in a mortar, and the hydration process was stopped using acetone. Between eight and ten drops of acetone were added, and each sample was dried for 15 minutes in a stream of cool air. During thermal analysis, the pastes were placed in a nitrogen stream and heated to a temperature of 1000°C at a rate of 10°C/min. The analyzed sample mass was 300 mg. The resulting data were evaluated using Proteus software, version 6.1.0.

3. RESULTS AND DISCUSSION

3.1. Rheological properties

The results for the rheological parameters are introduced in Table 5 and Figs. 3 and 4. The results of rheological measurements indicated that the addition of chalcedonite waste powder resulted in a much higher increase in rheological parameters, especially yield stress and consistency coefficient, within 1 hour compared to cement paste without addition (Fig. 3). The change in these parameters was visible, considering both the amount and the grain sizes of the addition (Fig. 4). The higher amount of addition and the reduction in its grain size increased τ_0 and K parameters. These results are related to the grain size and, therefore, the specific surface area of the waste material. The exceptions are two pastes modified with CHP10 powder in the amounts of 10% and 30%, in the case of which the consistency index decreased over time. The addition of the finest CHP10 to the cement paste can enhance the time growth of the inter-slip structure (flocculation + nucleation/ionic effects) $\Rightarrow \tau_0$ increases, but at the same time it can improve lubrication during flow (better particle packing + surface charge at high pH) $\Rightarrow$ viscosity decreases [28]; this is also clearly visible in the flow curves in Fig. 4. For CH10, the curve rises steeply at low shear rates (high viscosity) and after exceeding 30 s^{-1}, the steepness of the flow curve decreases significantly (viscosity decreased at higher speeds = easier flow, greater lubrication). With repeated shear forces, particle packing can be further improved (reduction of viscosity, increase of the yield point over time). In the coarser chalcedonite powder case,

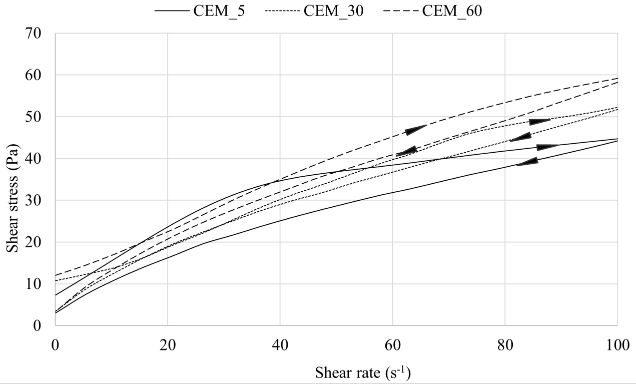

Fig. 3. Results of shear stress for cement pastes after 5, 30, and 60 minutes of measurements

Table 5

Rheological parameters of the tested pastes

Type of paste	t [min]	τ_0 [Pa]	K [Pa·s]	n [–]	R^2 [–]	Thixotropy [Pa·s^{-1}]
CEM	5 30 60	2.18 2.68 2.61	1.90 2.06 2.25	0.67 0.69 0.69	0.99941 0.99952 0.99929	611 208 350
CH10_10	5 30 60	1.75 2.59 3.17	5.01 4.88 4.58	0.50 0.52 0.55	0.99470 0.99704 0.99811	245 132 –22
CH10_20	5 30 60	4.93 5.46 4.26	9.22 9.60 10.55	0.40 0.38 0.36	0.96922 0.97221 0.97431	947 169 224
CH10_30	5 30 60	6.93 9.68 11.36	18.13 17.88 15.93	0.33 0.32 0.35	0.94347 0.94813 0.96423	1347 372 360
CH20_10	5 30 60	4.97 3.41 3.00	2.71 4.01 4.56	0.61 0.52 0.48	0.99792 0.99820 0.99714	821 186 125
CH20_20	5 30 60	6.47 4.18 4.05	5.08 6.92 6.71	0.51 0.44 0.44	0.98847 0.98785 0.98887	845 194 220
CH20_30	5 30 60	10.21 8.59 7.88	6.89 8.77 9.06	0.49 0.44 0.43	0.97768 0.97464 0.97775	1100 326 377
CH36_10	5 30 60	3.29 2.68 2.74	2.20 2.82 3.21	0.63 0.60 0.56	0.99873 0.99913 0.99880	707 224 103
CH36_20	5 30 60	2.28 1.88 1.92	3.35 3.98 4.04	0.53 0.52 0.53	0.99758 0.99803 0.99792	487 131 115
CH36_30	5 30 60	4.05 3.12 2.98	4.34 5.95 6.43	0.53 0.47 0.46	0.99550 0.99346 0.99433	662 160 127

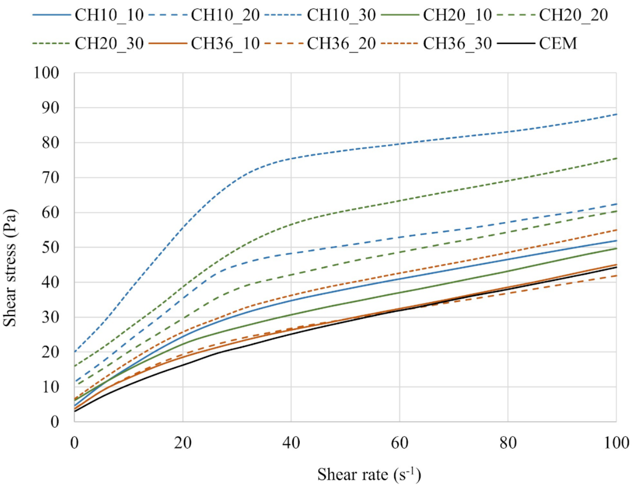

Fig. 4. Selected results of shear stress for pastes after 5 minutes

contrary, frictional/hydrodynamic dissipation and poorer lubrication may prevail ⇒ viscosity increases, while the colloidal network is weaker/diluted ⇒ τ_0 decreases [29].

All modified pastes had a smaller flow diameter and a smaller height of cone than the base cement paste (Figs. 5 and 6). The average mini slump cone of cement paste was 65.78 mm, average cone height was 13.72 mm. Average mini slump cone of paste with CHP10 was in the range 45.44÷55.40 mm, for paste with CHP20 in the range 46.12÷55.29 mm, for paste with CHP36, 52.43÷54.43 mm. Average height of cone for paste with CHP10 was in the range 18.20÷21.63 mm, for paste with CHP20 in the range 14.80÷20.47 mm, for paste with CHP36 in the range 13.91÷15.87 mm. The consistency of modified pastes decreased by a maximum of 31% compared to the sample without the addition. In each case, increasing the amount of addition resulted in a decrease in flow and an increase in the cone height. This is due to the smaller grain size and the higher specific surface of additives compared with the parameters of cement. The biggest changes were noted for pastes with CHP10, the smallest for samples with CHP36. When comparing pastes containing chalcedonite powder with one type of granulometry but different amounts of additive, it is evident that a larger amount translates into a lower consistency and a higher H parameter. Pastes containing 10% of additive, regardless of grain composition, were characterized by a similar flow. Paste CH36_10 containing 10% of CHP36 chalcedonite powder had the closest properties to cement paste without addition. The effect of chalcedonite powder on the consistency of the pastes is consistent with the results of the consistency coefficient from rheological measurements. Both the increase in the addition and the degree of fineness were noticeable in the increase in the consistency coefficient and in the reduction in the mini slump cone. The results of the consistency test (the impact of chalcedonite powder on paste flow) presented in the article are consistent with the results of pastes containing CHP10 chalcedonite powder presented in [20].

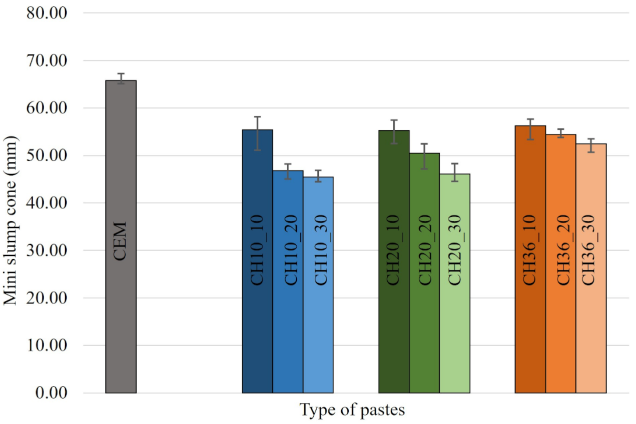

Fig. 5. Results of flow for all tested pastes

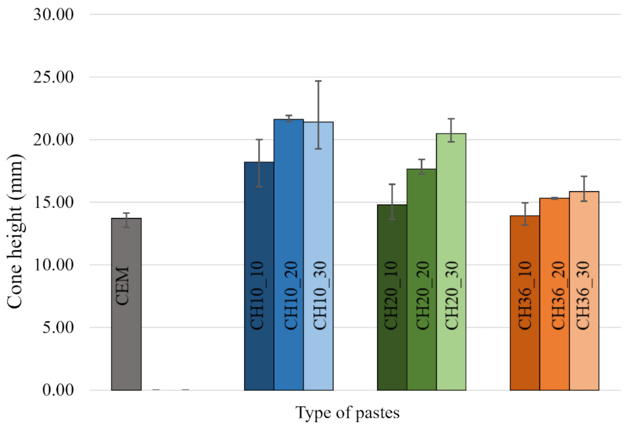

Fig. 6. Results of cone height for all tested pastes

3.2. Calorimetric measurements

Figure 7 presents the hydration heat evolution of the base cement paste CEM and modified pastes CH10, CH20, and CH36, while Fig. 8 shows the cumulative heat for all pastes. As shown by research, the addition of chalcedonite powder resulted in a noticeable decrease in the amount of heat evolution and contributed to a decrease in the amount of heat accumulated. The larger the amount of additives and the smaller their grain size (larger specific surface), the more prominent the changes were. The most similar heat release pattern when comparing the ref-

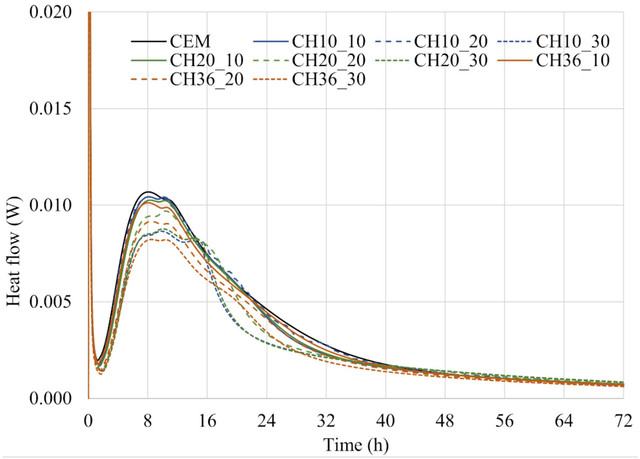

Fig. 7. Calorimetric curves of all tested pastes

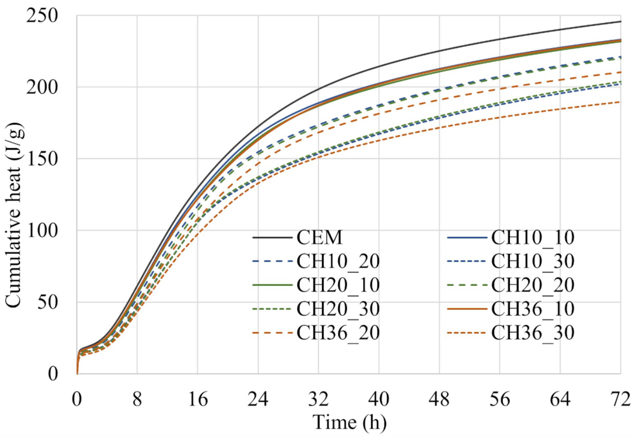

Fig. 8. Curves of cumulative heat for all tested pastes

erence CEM and modified pastes was attained by pastes with 10% powder in CHP10 and CHP20. The use of chalcedonite powder in each case (regardless of its grain size and quantity) resulted in a reduction in the occurrence of the main peak maximum on the hydration heat release rate curve. For all pastes, the second maximum was recorded after approximately 6–8 hours of hydration, which is related to the hydration of C_3S. This is associated with the nucleation and growth of C-S-H and portlandite [32]. The third peak is assigned to C_3A hydration after sulfate depletion (sulfate depletion point) and hydration of C_4AF. The fourth maximum is associated with monosulfate formation, and sometimes C_2S hydration may also contribute to this peak [32–34]. In some mixtures, CH20_20 and CH20_30 are prominent, whereas in CH36, it is shifted to later times (around 20 hours) and less prominent. The analysis of the calorimetric curve of the CH36_30 paste shows that the thermal effect is much more extended in time compared to other pastes. The induction period of the pastes modified with chalcedonite powder was slightly extended. The cement pastes without any addition had the highest amount of accumulated heat, equaling 246 J/g. The highest cumulative amount of heat evolution of pastes with addition was recorded in the case of the pastes modified with 10% chalcedonite powder, equal to about 233 J/g, and the lowest for the paste is equal to 189 J/g. The cumulative amount of heat evolution decreased by a maximum of approximately 23%, comparing all tested pastes. The biggest differences compared to the base paste CEM in the amount of accumulated heat were recorded for pastes with chalcedonite powder in the amount of 30% (regardless of the grain size of the addition).

3.3. Flexural and compressive strength

Figures 9 and 10 present an increase in flexural and compressive strength after 3 and 28 days of curing. Each figure shows bars illustrating the average strength after 3 and 28 days. The highest flexural strength after 3 days was recorded for the cement paste without the addition (4.91 MPa). Slightly lower results were achieved by pastes with 10% chalcedonite powder, and the smallest pastes with CHP10, CHP20, and CHP36 in the amount 30%. The biggest flexural strength after 28 days was found in the cement paste without the addition of CEM (8.01 MPa). Pastes CH20_10 and CH36_10 with chalcedonite powder in the amount of 10% had the highest 28-day flexural strength modified pastes, among others.

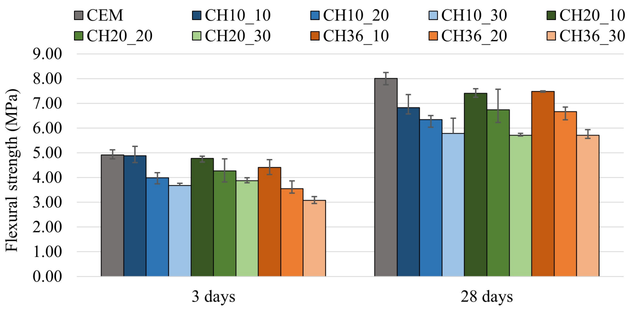

Fig. 9. Results of flexural strength for all tested pastes

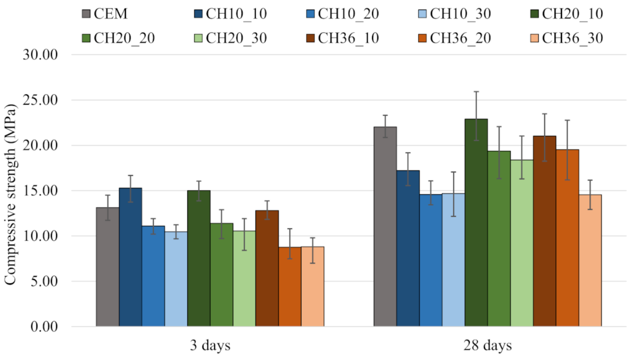

Fig. 10. Results of compressive strength for all tested pastes

It was noticed that increasing the amount of addition caused a decrease in flexural strength. On the other hand, the influence of grain size is not so evident. After 28 days, the flexural strength of the pastes containing chalcedonite powder was lower by 7÷29% than paste CEM. The biggest increase in flexural strength over time was achieved by the pastes with chalcedonite powder CHP36. Pastes with CHP20 and CHP36 powder were characterized by comparable results, comparing the amount of these materials. The CH20 and CH36 pastes with 10% and 20% chalcedonite waste had higher flexural strength than CH10 samples. The difference was only visible in the case of the paste CHP10_30. Paste with chalcedonite CHP10 in the amount of 30% (CH10_30) had close 28-day flexural strength to paste CH20_30 and CH36_30.

The compressive strength of the cement pastes without addition after 3 and 28 days of curing was equal to 13.14 MPa and 22.02 MPa. The samples CH10_10 and CH20_10 were characterized by the biggest compressive strength after 3 days, equal to 15.28 MPa and 15.00 MPa. On the other hand, the use of CHP20 chalcedonite powder in the amount of 10% (CH20_10) resulted in an increase in compressive strength compared to the base cement paste (CEM), both after 3 and 28 days. Similarly to the flexural strength research, the bigger the amount of addition within one type of grain size, the smaller the compressive strength. After 28 days, the compressive strength of the paste containing chalcedonite powder was lower by a maximum of 34% than the base cement paste without addition. The greatest increase in compressive strength over time was recorded for pastes containing CHP20 and CHP36. The most optimal solution, from the 28-day compressive strength point of view, seemed to be the use of CHP20 and CHP36 chalcedonite powder in the amount of 10% (22.90 MPa, 21.02 MPa).

Given calorimetric measurements and the 3-day compressive strength results, it can be seen that both increasing the amount of addition and increasing the specific surface area resulted in a decrease in the accumulated heat and a decrease in compressive strength compared to the reference sample. Only in the case of pastes containing 10% addition was there an increase in compressive strength observed, which may be due to the sealing of the material structure resulting from the addition of chalcedonite powder.

3.4. DTA-TG results

The thermogravimetric results were conducted for the series of CEM paste, and all pastes: CH10, CH20, and CH36. Data showing the amount of chemically bound water H_20, portlandite $Ca(OH)_2$, and calcium carbonate $CaCO_3$ is plotted in Fig. 11. When analyzing the changes in the pastes over 28 days, an increase in chemically bound water and portlandite was noticeable for each of the pastes. On the other hand, the calcium carbonate content decreased over time. It can be seen that with the passage of time, the amount of chemically bound water was increasing, which corresponded to a larger amount of C-S-H phase. These results were consistent with the results obtained from the mechanical properties research, especially with compressive strength. The portlandite content in the reference sample CEM I was increasing as the hydration time progressed (from 13.90% to 17.48%). The differences in the amount of $Ca(OH)_2$ after 3 days of curing between the reference sample CEM and the other pastes might be due to the reduced amount of Portland clinker. The amount of portlandite with the addition of chalcedonite powder increased between 3 and 28 days, but it was not as large as in the case of cement paste without the waste powder. Analyzing the obtained results, it can be seen that the increase in the amount of the addition had a bigger impact on the change in the amount of portlandite than on the change in the amount of chemically bound water. Replacing some of the cement with an addition resulted in a reduction in portlandite. The greater the amount of chalcedonite powder, the lower the amount of $Ca(OH)_2$. The effect of grain size is not as noticeable as the effect of the amount of addition in terms of changes in the amount of chemically bound water and portlandite.

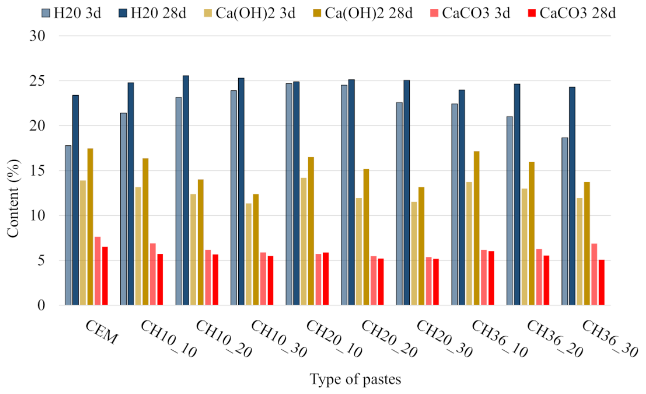

Fig. 11. Results of DTA/TG analysis

4. CONCLUSIONS

This article shows original research on the assessment of selected properties of cement pastes modified with dust of chalcedonite powder. The variable factors in the studies were the quantity and grain size of the addition. The focus was on rheological studies and the processes of setting and hardening of pastes. These properties are important in selecting cement for a specific application.

As shown, the amount of the chalcedonite powder and its grain size influence both the rheological properties and the hydration kinetics, as well as hardening processes after a longer curing time.

1. The amount and grain size of chalcedonite powder influenced the consistency of the tested pastes. The larger the amount of addition and the smaller its grain size, the smaller the flow of the paste. 10% replacement of cement with an addition regardless of grain size had the least impact on the change in consistency. Further increase in the amount of chalcedonite powder caused a decrease in the flow. In practical applications, obtaining the consistency of the grout modified with chalcedonite powder similar to the consistency of the cement grout without the additive would be possible if a plasticizing admixture were used.
2. The yield stress and consistency coefficient of the cement paste without addition are similar throughout the entire measurement period. Adding chalcedonite powder to cement and its grain size significantly affects the rheology of the tested pastes. An increase in the amount of chalcedonite powder causes an increase in the consistency coefficient. This parameter increases within an hour of the measurement. An increase in the amount of chalcedonite powder added, and an increase in grain size, causes an initial increase in the yield stress, which decreases after measurements taken after 30 and 60 minutes, which is most noticeable for pastes with chalcedonite powder CHP20 and CHP36.
3. In the case of the tested pastes, the amount of heat released decreases with increasing amounts of mineral addition. This effect is also visible with decreasing grain size of the chalcedonite powder. The total heat of hydration in the case of pastes containing chalcedonite powder is lower than in the base paste (without the addition).
4. Considering the compressive strength results after 3 and 28 days of curing, it seems most advantageous to replace cement in the amount of 10% with chalcedonite powder with a grain size of 20 μm or 36 μm, which was consistent with research results of DTA/TG. It is planned to perform long-term tests (determining mechanical properties after at least 90 days) to check the puzzolan activity of the addition after a longer maturation period.

Chalcedonite powder can be an alternative to the commonly used addition in cement production, reducing the carbon footprint of binders. The choice of an addition should be preceded by a thorough analysis of the properties of the materials obtained. It would be justified to adjust the appropriate cement-chalcedonite powder proportions to the appropriate applications of this binder in construction, considering both rheological properties and the evaluation of the setting and hardening processes.

Further research is planned in which chalcedonite powder will be used as a binder component in mortars (masonry or plastering). It will be important to select, among other things, the appropriate amount and grain size of chalcedonite powder to obtain optimal standard properties (consistency, compressive strength, adhesion) and application properties.

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