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RHEOLOGICAL PROPERTIES OF CEMENT PASTES MODIFIED WITH PUMICE, TRASS AND CHALCEDONITE POWDER

WŁAŚCIWOŚĆI REOLOGICZNE ZACZYNÓW CEMENTOWYCH MODYFIKOWANYCH PUMEKSEM, TRASEM I MĄCZKĄ CHALCEDONITOWĄ

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Abstract

The article presents the influence of pumice, trass and chalcedonite powder on rheological properties of cement pastes. Cement was being replaced both of additions 10% or 20% by mass and combination of pumice or trass and chalcedonite powder in the amount 5% or 10% of each of them. The main purpose of the publication was to assess the effects of chalcedonite powder with selected mineral additions in terms of rheological properties and compare them with the results obtained for pastes modified with only one of the additions. In each case, the additive or combination of additives introduced into the paste reduced the flow, compared to the result of paste without additive(s). Rheological properties varied depending on type and amount of additive(s).

Keywords: cement paste, pumice, trass, chalcedonite powder, consistency, yield stress, consistency index

Streszczenie

W artykule przedstawiono badania wpływu pumeksu, trasu oraz mączki chalcedonitowej na właściwości reologiczne zaczynów cementowych. Cement zastępowano każdym z dodatków w ilości 10% lub 20% oraz kombinacją pumeksu lub trasu z mączką chalcedonitową w ilości po 5% i 10% każdym z nich. Głównym celem pracy była ocena współdziałania mączki chalcedonitowej z wybranymi dodatkami mineralnymi w zakresie właściwości reologicznych oraz porównanie rezultatów badań z wynikami uzyskanymi dla zaczynów modyfikowanych tylko jednym z dodatków. W każdym z przypadków dodatek lub kombinacja dodatków wprowadzone do zaczynu wpłynęły na zmniejszenie rozpływu, w porównaniu do wyników uzyskanych dla zaczynu bez dodatku(ów). Właściwości reologiczne były zróżnicowane w zależności od rodzaju i ilości dodatku(ów).

Słowa kluczowe: zaczyn cementowy, pumeks, tras, mączka chalcedonitowa, konsystencja, granica płynięcia, wskaźnik konsystencji

1. INTRODUCTION

Building mortars and concrete are commonly used in civil engineering. Main ingredient used in their production is cement. However, one should note that the production of this binder involves large energy inputs and emission of dust and gases such as nitrogen oxides, sulfur dioxide, carbon oxides, etc. to the atmosphere, what has direct impact on the

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increase in the greenhouse effect. Estimated annual production of cement in Poland is over 19 million tons, and the production of 1 ton of cement clinker causes carbon dioxide emissions about 800 kg [1-6]. Because of the ideas of sustainable development and rational waste management, the type of solutions that would allow meeting the requirements imposed on the industry cement are sought in various ways. The use of mineral additions, including waste materials, allows a significant reduction in the emission of harmful gases into the atmosphere and also has a positive effect on the idea of a closed economy [1, 6-9]. Commonly used in cement production are: fly ash, granulated blast furnace slag and limestone flour [1, 10]. The PN-EN 197-1 standard also allows the use of other natural or artificial pozzolans. This is particularly important due to the limited resources of fly ash or due to the decarbonization of steel production processes and, therefore, the availability of blast furnace slag [1]. In many countries, one can find the technology of producing pozzolanic cements, containing natural pozzolans of volcanic or organic origin [11]. The optimal solution would be to use raw materials with a low carbon footprint that do not require additional thermal or chemical treatment. There are publications in the literature on the use of two- or three-component binders in which the cement is partially replaced by, among others: metakaolin [12, 13], zeolite [14-16], spongiolite [17], stone powders [1, 7-9, 18-20], simultaneously: zeolite and silica fume, fly ash and granulated blast furnace slag, limestone flour and zeolite, granite powder and fly ash [10, 14, 21, 22]. This is a current topic, especially due to the widespread use of Portland cements with additions and the popularization of low-clinker cements. Solutions are being sought that will be profitable from an ecological and economic point of view, bearing in mind the assumption that the properties of newly created binders must comply with standard requirements, ensuring the durability of the structure. Analyzing the literature, it can be concluded that researchers focus mainly on determining the impact of additions on mechanical properties, binding and hardening processes, corrosion resistance, microstructure of materials based on cement binder [7, 8, 10, 11, 13-15, 20-21, 23-25]. Only in a few publications rheological issues were analyzed [9, 12, 17, 26, 27].

Pumice, trass and chalcedonite powder were used as a partial replacement for cement. In this study, the authors focused on determining the rheological properties of cement pastes modified with varying amounts of one of the additions or their combination. The main aim of the research was to assess the impact of chalcedonite powder in the context of the properties of three-component binders. Additionally, the effect of each of the additions used was determined separately as a partial substitute for cement. Consistency testing was performed and rheological properties were determined, such as consistency coefficient, yield stress, and thixotropy.

2. MATERIALS AND METHODS

The industrial ordinary Portland cement CEM I 42.5R was used in the research. Three materials were selected as additions: pumice, trass and chalcedonite powder. Pumice and trass are volcanic rocks. They were used in the work in the form of finely ground powder. They belong to the group of natural pozzolanas. Chalcedonite powder is a waste material generated during aggregate production through appropriate (contamination-free) grinding and air classification. The chemical composition of cement, pumice, trass and chalcedonite powder was presented in Table 1. It was measured by XRF method by Axios X-ray Fluorescence XRF spectrometer (Malvern Panalytical Ltd.).

The grain size distribution was measured with laser granulometer HELOS KR (Sympatec GmbH). Basing on the Table 2 it was establish that the smallest grain size had chalcedonite powder. This material contained much more fine particles than other additions – about 79% of the particles in range 0-5 mm and about 98% of the particle in range 0-10 mm. Cement and trass contained about 33%, but pumice about 23% of the particles in range 0-5 mm. On the other hand content of particles in range 0-10 mm for cement and trass was about 48%, for pumice – about 39%. Cement, trass and pumice contained about 83% of the particles in range 0-35 mm.

Eleven type of pastes were prepared, in which Portland cement was substituted with pumice, trass and chalcedonite powder in the amounts of 10% (T10, P10, CH10) or 20% (T20, P20, CH20) and combination pumice with chalcedonite powder or trass with chalcedonite powder in the amount of 5% (T5CH5, P5CH5) or 10% (T10CH10, P10CH10) of each of them and the last kind of samples was the reference material – paste without mineral additions (C). Water/binder ratio was constant for all pastes and equal 0.5. The pastes mix proportion were detailed in Table 3.

Table 1. Chemical composition of cement and mineral additions [%]

Material	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	TiO_2	MnO	P_2O_5	L.O.I.
Cement	18.33	4.71	4.25	64.13	1.65	0.05	0.59	2.68	0.26	0.19	0.16	2.99
Pumice	54.27	20.50	2.07	0.65	0.11	9.20	5.62	0.07	0.21	0.42	0.07	6.38
Trass	50.08	17.61	5.46	4.16	1.70	3.61	4.67	0.05	0.81	0.21	0.34	10.05
Chalcedonite powder	99.01	0.84	0.04	0.05	0.03	0.05	0.04	–	0.02	0.01	0.03	0.07

Table 2. Grain size distribution of cement and additions

Material	x_{10} mm	x_{50} mm	x_{90} mm	Content of particles, %			
				≤5 mm	≤10 mm	≤20 mm	≤35 mm
Cement	0.78	11.07	45.31	32.67	47.92	68.27	83.66
Pumice	1.36	13.88	47.26	23.55	39.69	65.13	82.56
Trass	0.94	10.55	47.48	33.05	49.14	69.13	83.21
Chalcedonite powder	0.43	1.80	7.31	78.76	97.75	100.00	100.00

Table 3. Composition of tested pastes [g]

Symbol of paste	Cement	Pumice	Trass	Chalcedonite powder	Water
C	70.0	0.0	0.0	0.0	35.0
P10	63.0	7.0	0.0	0.0	35.0
P20	56.0	14.0	0.0	0.0	35.0
T10	63.0	0.0	7.0	0.0	35.0
T20	56.0	0.0	14.0	0.0	35.0
CH10	63.0	0.0	0.0	7.0	35.0
CH20	56.0	0.0	0.0	14.0	35.0
P5CH5	63.0	3.5	0.0	3.5	35.0
P10CH10	56.0	7.0	0.0	7.0	35.0
T5CH5	63.0	0.0	3.5	3.5	35.0
T10CH10	56.0	0.0	7.0	7.0	35.0

The research of consistency was done by mini-slump cone test [28]. Every time, paste was prepared and filled into the cone with few layers to avoid the air bubbles. The cone was lifted with care and the sample was allowed to spread on the glass table flow. The flow diameter (mini-slump flow) and height sample (*H*) after flow were measured. The arithmetic mean of four measurements was given the final mini-slump cone.

The rheological measurements were carried out using Discovery HR-1 hybrid rheometer (TA Instruments) in a Peltier Concentric Cylinder system with a DIN rotor according to [29]. The standard gap for the DIN cylinder system (5.917 mm) was employed. The same procedure was maintained for the preparation and testing of all samples. After manual mixing of the dry ingredients with water (1 min), the sample was immediately located into the cylinder for testing. The measurement was started by a 60 s preshear at 50 s^{-1} followed by 60 s of resting time in order to re-homogenize the sample and to eliminate its shear history. The preshear started 3 minutes after the start of mixing. Then the shear rate increased and decreased in a range from 0 to 100 s^{-1} through 30 steps with 15 s of measuring time at each shear rate. Flow curves were measured after 5 and 30 minutes from the start of mixing the materials. Yield stress (τ_0), consistency coefficient (*K*) and fluidity index (*n*) were calculated from the decreasing branch of the flow curve using the Herschel-Bulkley model [30]:

$$\tau = \tau_0 + K \cdot \dot{\gamma}^n \quad (1)$$

where: τ – shear stress [Pa]; τ_0 – yield stress [Pa]; K – consistency coefficient (comparable to plastic viscosity) [Pa·s]; $\dot{\gamma}$ – share rate [s^{-1}]; n – fluidity index.

Thixotropy of the pastes was determined by the TRIOS software as a hysteresis area between the flow curves. During preparation and during consistency and rheological tests, the temperature in the laboratory was kept at 20°C ±2°C. The water/binder ratio was constant for all pastes in both research.

3. TEST RESULTS AND DISCUSSION

The results of consistency measurements for all pastes are shown in Table 4 and in Figure 1.

Table 4. Consistency results of the tested pastes

Symbol of paste	Consistency		
	Mini-slump cone [mm]	Standard deviation [mm]	*H* [mm]
C	61.3	1.3	14.5
P10	41.7	1.0	22.4
P20	40.5	0.5	26.8
T10	43.5	0.4	20.1
T20	43.1	0.2	21.4
CH10	46.1	0.5	18.2
CH20	42.2	0.5	22.1
P5CH5	41.4	0.3	22.2
P10CH10	42.0	0.4	21.6
T5CH5	40.1	0.3	27.7
T10CH10	41.2	0.5	22.1

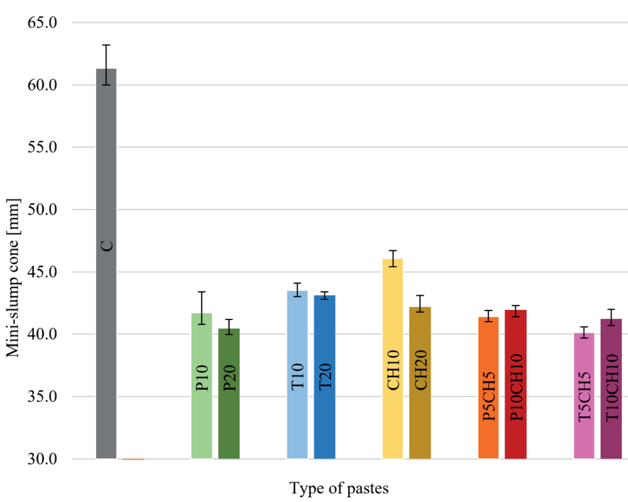


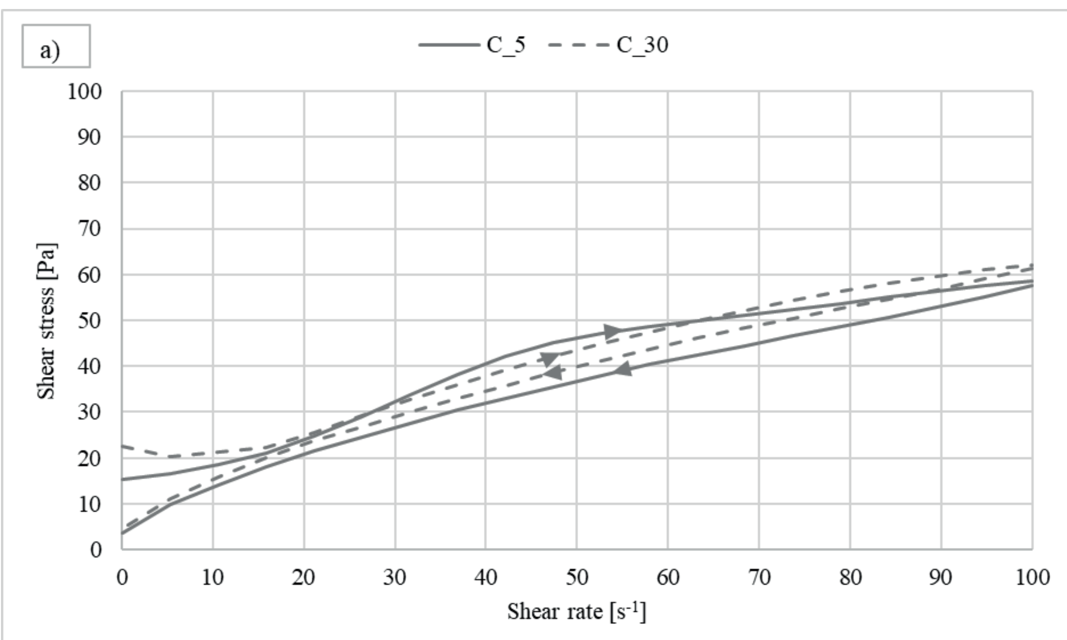
Fig. 1. Mini-slump flow of pastes

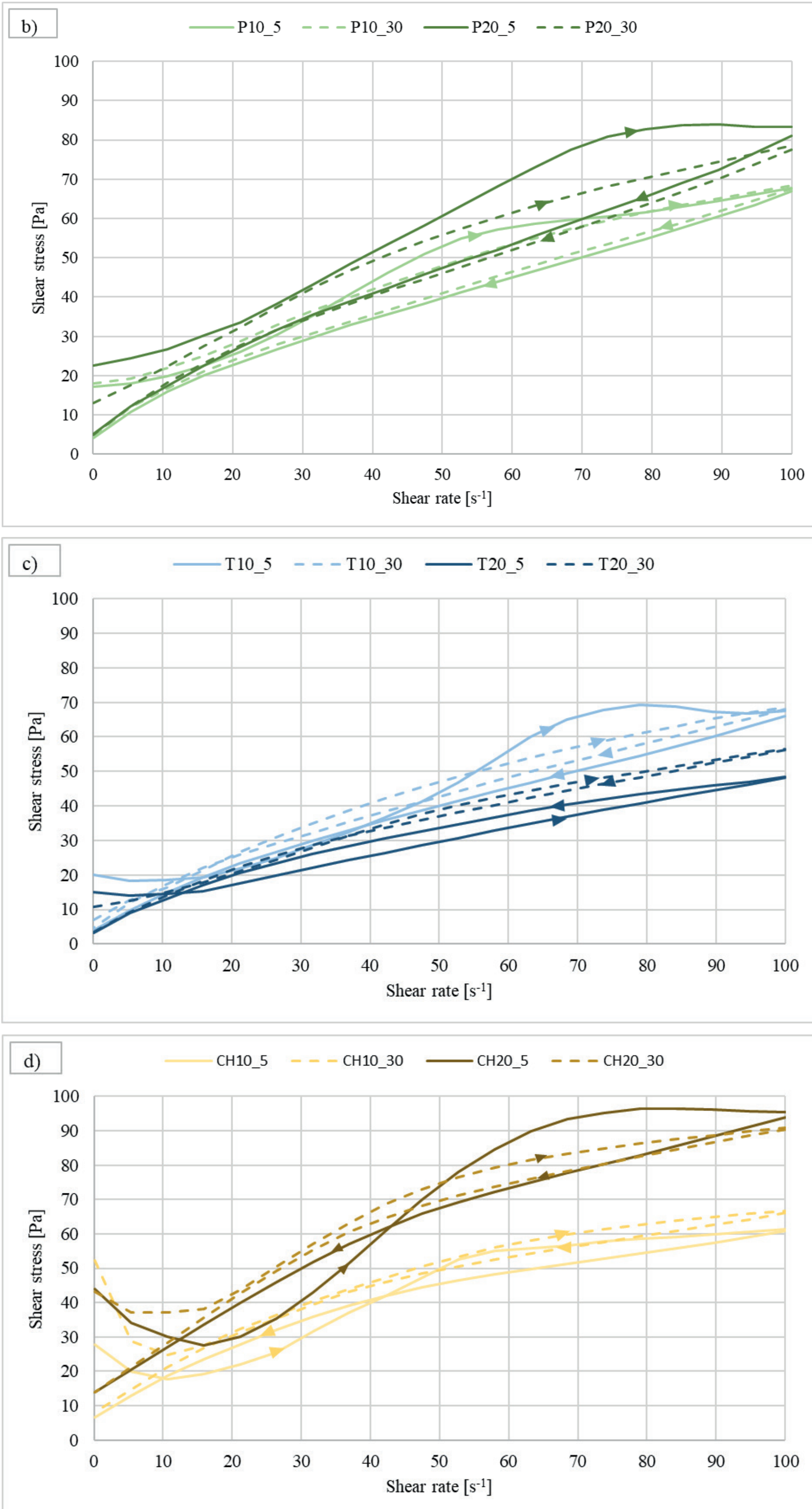
The biggest mini-slump flow was achieved for paste without additions (C) and it was equal 61.3 mm. All modified pastes had smallest consistency compared to reference sample, about 25-35% smaller flow than C paste. Paste contained combination of trass and chalcedonite powder in the amount of 5% of each of them (T5CH5) had the smallest flow (40.1 mm). In the case of replacing cement with one of the additions, the greater its amount, the smaller the flow was recorded. The greatest change in consistency was observed in pastes containing chalcedonite powder, which was related to the smallest grain size of this additive (especially the content of particles ≤5 mm). Increasing its share from 10% to 20% resulted in a reduction in flow by 39 mm (comparing the flow of CH10 and CH20 pastes). The influence of the amount of trass on paste flow was negligible. A different effect was noted when a combination of two additions was used. Increasing the total amount of additions from 10% to 20% resulted in even a slight plasticization of the pastes (wider spread). The height of samples (*H*) in consistency research was bigger for all pastes compared to C sample. The increase in the amount of additive resulted in an increase in the *H* parameter. The increase in the amount of combine additives resulted in a decrease in the *H* parameter (comparing pastes P5CHP with P10CH10 and T5CH5 with T10CH10). It can be concluded that the effect of one additive was thickening, but replacing part of the pumice or trass with chalcedonite powder gave a liquefying effect.

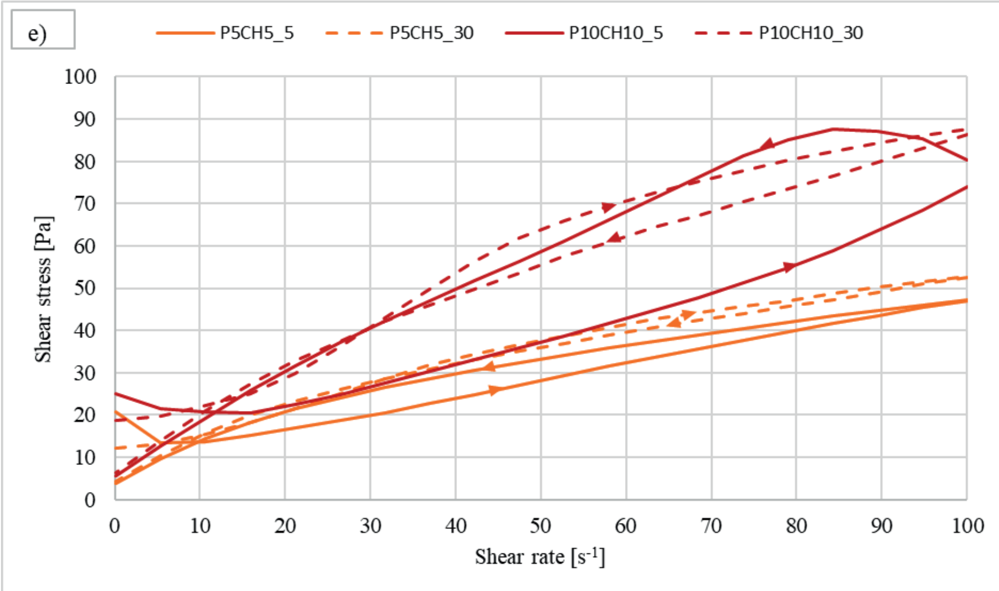
The results of determining rheological parameters of pastes are presented in Table 5. The flow curves of cement pastes without additions and the flow curves of cement pastes with additions are shown in Figure 2 (measurements done after 5 and 30 minutes). As can be seen from the values of the determination of R^2 (Table 5), the results of rheological parameters described by the Herschel-Bulkley model characterized by a high fit (R^2 close to 1 value). In this model, the highest values of yield stress (τ_0) was obtained for the pastes modified with 20% of chalcedonite powder. The consistency coefficient (K), which is a measure of the viscosity of pastes had the highest values for the samples with chalcedonite powder, which means that these pastes had relatively the highest viscosity. Pastes containing both pumice and trass were characterized by a lower K parameter than pastes without the additive. As the date presented in Table 5 show that replacement cement by chalcedonite powder caused increase in pseudoplasticity (decrease of n parameter), opposite

Table 5. Rheological measurements of pastes

Series of paste	Time of measure-ments after mixing [min]	τ_0 [Pa]	*K* [Pa·s]	*n* [-]	R^2 [-]	Thixotropy [Pa·s^{-1}]
C	5	2.94	2.26	0.69	0.9997	592
	30	3.71	2.51	0.68	0.9998	390
P10	5	4.50	1.74	0.77	0.9989	739
	30	4.90	1.91	0.75	0.9993	564
P20	5	5.38	1.85	0.80	0.9987	1199
	30	5.22	2.07	0.77	0.9984	679
T10	5	3.66	1.93	0.75	0.9994	606
	30	4.33	2.41	0.71	0.9998	244
T20	5	1.36	3.20	0.59	0.9979	-198
	30	1.85	2.91	0.64	0.9989	112
CH10	5	3.04	5.11	0.53	0.9922	200
	30	4.18	5.58	0.53	0.9929	33
CH20	5	9.48	4.71	0.63	0.9936	515
	30	7.82	7.03	0.54	0.9863	495
P5CH5	5	2.11	3.46	0.56	0.9988	-259
	30	2.77	3.42	0.58	0.9990	102
P10CH10	5	4.78	2.06	0.84	0.9995	-1638
	30	4.49	3.45	0.69	0.9994	463
T5CH5	5	4.05	1.77	0.80	0.9995	-1025
	30	3.14	3.22	0.64	0.9991	310
T10CH10	5	4.44	3.20	0.67	0.9990	-1021
	30	2.54	4.61	0.59	0.9958	182




b)
P10_5
P10_30
P20_5
P20_30
Shear stress [Pa]
Shear rate [s-1]
c)
T10_5
T10_30
T20_5
T20_30
Shear stress [Pa]
Shear rate [s-1]
d)
CH10_5
CH10_30
CH20_5
CH20_30
Shear stress [Pa]
Shear rate [s-1]



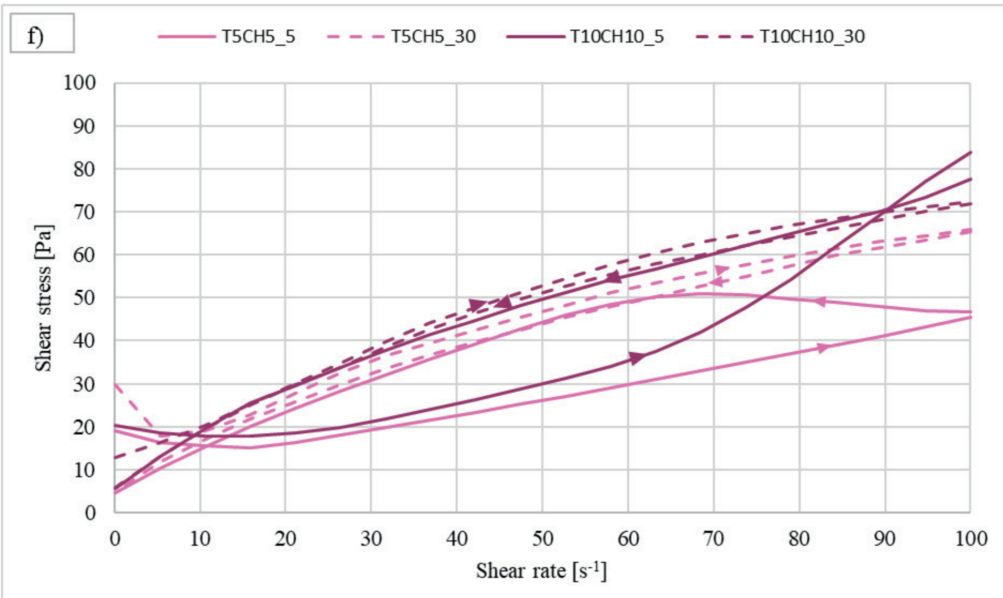


Fig. 2. Flow curves for all tested pastes

to an action of pumice (decrease in pseudoplasticity). This phenomenon was visible regardless of the amount of additive used. In other cases, the effect was varied. For cement pastes with one type of addition, a deterioration of thixotropic behavior over time (decrease in thixotropy values) was observed, which is due to ongoing hydration and pozzolanic reactions of the binder forming denser paste structures. The opposite trend was evident for pastes with combined additives when there was a change in behavior from rheopectic to thixotropic over time (an increase in thixotropy values from negative to positive). Apparently, in the initial phase of hydration of pastes with a combination of the addition of chalcedonite with pumice or trass, cohesive forces between these particles are formed, which are broken over time by hydration and pozzolanic reactions. This idea can also be supported by the opposite trend of yield stress development over time for pastes with a combined addition (a decrease in yield stress due to the interruption of cohesive forces) in contrast to an increase in yield stress for pastes with one type of addition due to the increasing density of ongoing reactions.

The changing of yield stress and consistency coefficient (comparable to plastic viscosity) over time are shown in Table 5 and Figures 3 and 4. Analysis of rheological research results of cement pastes with pumice, trass and chalcedonite powder showed varied impact of additions on yield stress and consistency coefficient compared to values of these parameters for cement pastes without additions. The addition of pumice to cement resulted in an increase in yield stress and a decrease in paste viscosity (regardless of the amount of addition and time of measurement). It is probably due to the internal structure of this material. On the other hand, cement pastes with chalcedonite powder had higher yield stress and consistency coefficient than reference cement paste due to the very fine particles (with a larger specific surface area) and pseudocrystalline composition of chalcedonite. Suspensions of crystalline substances flow more poorly (have a higher viscosity) compared to suspensions of amorphous substances. Trass in the addition of 10% had a similar effect to pumice; in the addition of 20% there was a decrease in yield stress and an increase in viscosity of pastes. The interaction of the addition of chalcedonite powder with pumice and chalcedonite powder with trass were varied. Although individual additions of pumice and chalcedonite powder in the amount of 10% increased the yield stress, the combination of 5% pumice and 5% chalcedonite decreased the yield stress of the cement paste. For the other combinations, there was usually an increase in the yield stress of the pastes. The viscosities of pastes with combined additives also varied greatly. The time trend of the viscosity development was, with minor exceptions, increasing. On the basis of the presented curves (Fig. 2) and the results presented on Figures 3 and 4, it was found that the type and the amount of additive(s) influenced not only yield stress and viscosity of pastes prepared from them, but also their rheological stability. Pastes containing 10% pumice and combination of 5% pumice and 5% chalcedonite powder achieved the most stable viscosity over time.

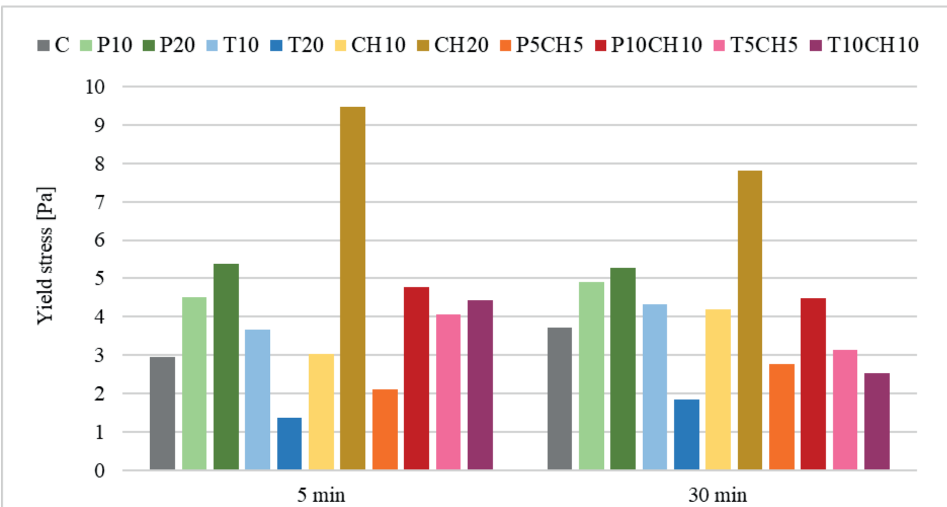


Fig. 3. Yield stress of pastes

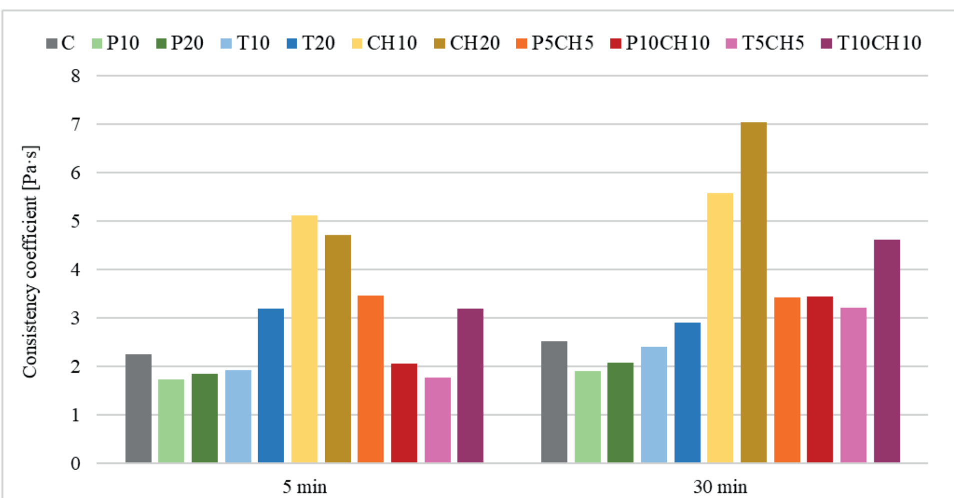


Fig. 4. Consistency coefficient of pastes

4. CONCLUSIONS

All modified pastes were characterized by lower flow than cement paste without the addition(s). The pumice, trass and chalcedonite (added one by one) acted as thickeners.

The influence of the type(s) of additions used and their amounts on the rheological properties of cement pastes is varied.

The rheological properties of two- or three-component pastes depend, among others, on the grain size of the addition(s) used.

Partial replacement of pumice or trass with chalcedonite powder had a differential impact on the consistency and rheological properties of the pastes (depending on the amount of additive). In most cases, chalcedonite powder reduced consistency of pastes (compared pastes with only pumice or trass), except paste with combination 5% pumice and 5% chalcedonite powder. On the other hand, when comparing the properties of pastes with 10% and 20% of two additives – the increase in the amount of chalcedonite powder has a plasticizing effect, compared to the properties of pastes containing only pumice or trass. Chalcedonite powder reduced yield stress, but increased consistency of pastes with pumice, what was not so noticeable in pastes modified with chalcedonite powder and trass. The use of chalcedonite powder in combination with pumice or trass changes the thixotropic behavior of the cement slurry in the initial phase of hydration to rheopectic. However, over time, these cement slurries become thixotropic.

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