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THE CONSISTENCY OF CEMENT PASTES AND THEIR RHEOLOGICAL PARAMETERS

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Abstract. This paper presents the results of the analysis of slump test parameters and rheological properties of cement pastes prepared with use of cement CEM II/B-V 32.5R. Slump parameters were determined with use of a special marked plate, while the rheological parameters were determined with use of the rotational viscometer Viscotester VT 550, manufactured by Haake, with coaxial cylinders, equipped with the MV2 system. Cement pastes consisted of a mixture of cement and water. The mixture concentration, described with use of the water to cement ratio W/C, fell into the range 0.65–0.33. In this study the dependence of the slump parameters on the water to cement ratio W/C was determined. The pseudo-curves of flow of cement pastes, obtained as a result of viscometric tests, were then adjusted to actual curves of flow and approximated with use of the tri-parametric, generalised Herschel-Bulkley model. The dependence of the rheological parameters of the model τ_o, k, n, as a function of the water to cement ratio W/C was calculated. The existing correlation between rheological parameters and slump parameters was determined for the analysed cement pastes.

Key words: cement pastes, slump, rheological model, rheological parameters

INTRODUCTION

The technological processes of preparing and managing concrete mixture are characterised by the presence of various loads that have a significant influence on the parameters of flow. Actions causing such loads include: mixing, transport, pumping, laying, compaction and finishing activities. „Workability" is a term that links the issues of optimal management of concrete mixture to its rheological properties in the context of the existing, complex system of loads [Szwabowski 2006].

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Rheology is a science that enables to analyse and forecast the behaviour of material subjected to various loads and allows controlling the behaviour of materials in specific technological processes. Rheological phenomena occurring in mixtures of cement concretes and mortars have a very complex nature. This results from the multi-ingredient and multi-phased structure of the mixture and the differentiation of load systems in various technological processes. In concrete technology, rheology aims to determine the correlations between these factors and to ensure their optimised co-operation. The main aim is to guarantee an efficient course of technological processes. Moreover, the development of technology also requires knowledge development, in order to enable solving and explaining the problems that occur with the new technology [Sokołowski and Pużak 2002, Szwabowski 2006].

The analysis of subject literature demonstrates that concrete mixture under load behaves, similarly to certain cement and cement-limestone mixtures, like a viscoplastic body, which may be described e.g. with use of the Bingham model [Kilian 2008, Świerzko 2011, Szaj 2012]. The main parameters of this model are: yield stress τ_o and plastic viscosity η_{pl}. The description of the rheological behaviour of a concrete mixture is a difficult and complex process. The existence of large-size grains makes it impossible to use standard rotational viscometers equipped with too narrow, 1–5 mm wide gaps. However, it is possible to apply hydraulic systems that meet the requirements for pipe viscometers. Such tests are difficult to conduct in laboratory conditions due to large amounts of the analysed material, time limitations resulting from the occurring hydration and binding processes and the problem of disposal of concrete mixture after the completion of pipeline tests. An opportunity to conduct such tests is to install measuring equipment in the so-called „concrete unit", where the main parameters of hydrotransport (total pressure loss, rate of flow) may be analysed, which, in turn, will allow for the determination of the pseudo-curve of flow of concrete mixture. In practice, for the determination of consistency and workability of fresh concrete and mortar mixtures slump tests are widely used. This has been reflected in European standards [EN 1350-2, EN 1350-5, EN 1015-3].

The cement paste plays the main role in the formation of the properties of concrete mixtures. It constitutes the liquid phase of the mixture, so knowledge of its rheological properties is an important stage of the determination of mixture parameters. From the technological point of view, analysing the rheological properties of cement paste is much easier, due to the small size of grains and the small volume of the required samples, and it may be performed with use of rotational viscometer. Practical evaluation of the consistency of the paste consists in conducting a slump test and measuring the slump diameter and height at the central point. The obtained results are closely linked by rheological properties. On the basis of the slump test results, in particular the slump height, most often the value of the yield stress is determined [Pashias et al. 1996, Saak et al. 2004, Roussel et al. 2005, Roussel and Coussot 2005]. In the literature can be found also relations between the parameters of Bingham model (yield stress, plastic viscosity) and the diameter and height of the slump [Wallevik 2006, Gao and Fourie 2015].

The aim of this study is to find, in laboratory tests, the correlation between slump parameters and rheological parameters of the Herschel–Bulkley model for various concentrations of analysed cement pastes, described with use of water to cement ratio W/C.

MATERIALS AND METHODS

The analysed cement pastes are mixtures of ash Portland cement CEM II/B-V 32.5R with water.

Basing on laboratory tests, conducted pursuant to PN-B-19707:2003, PN-EN 196-1:2005, PN-EN 196-3, PN-EN 196-6, the basic physical and strength parameters of the analysed cement CEM II/B-V 32.5R were determined and compared with manufacturer's data. The obtained parameters fell within the ranges specified by the manufacturer.

The preparation of paste for analysis consisted of two basic stages. The first stage, common for slump tests and the analysis of rheological properties, consisted in manual and mechanical mixing of cement with an adequate amount of water, for approx. 9 minutes. The aim was to ensure correct hydration of cement and to destroy the thixotropic structure of the paste. This duration meets the requirements for optimal mixing time, which, according to Kurdowski [1991], should last for 5–10 minutes. The second stage are the actual tests, which, for viscometric measurements, consist of a preliminary shearing cycle that lasts for approx. 3 minutes and improves the results obtained in the first stage, and of the measurement itself that lasts for approx. 2 minutes. The preparation of samples and the determination of slump parameters and rheological properties were conducted pursuant to the methodology described in the study [Kempiński and Świerzko 2010, Kasińska 2014].

Slump tests were performed on a special plate marked with centrally and coaxially located circular grooves. The measurement cylinder, of a height of 100 mm and a diameter of 50 mm, placed in the centre of the plate, is filled with paste. Then, the surface of the cylinder should be levelled and raised vertically in a quick, single movement. Slump diameter is read from the circles and the thickness of the paste is measured with use of callipers.

Viscometric tests were performed with use of Haake VT 550 Couett-Searle Viscotester equipped with MV2 measurement system.

Laboratory tests were conducted as part of an MSc thesis [Kasińska 2014].

TEST RESULTS

Slump tests

The slump tests of cement pastes were conducted for various water to cement ratios W/C, within the range 0.33-0.65. The obtained results concerning slump diameter *d* and slump height *h* at the central point of slump are listed in Table 1.

Table 1. Results of slump tests of cement pastes

Water to cement ratio W/C	0.65	0.60	0.55	0.50	0.45	0.40	0.35	0.33
Slump diameter *d*, mm	180	170	150	140	120	90	70	50
Slump height *h*, mm	3	3	4	5	7	11	18	23

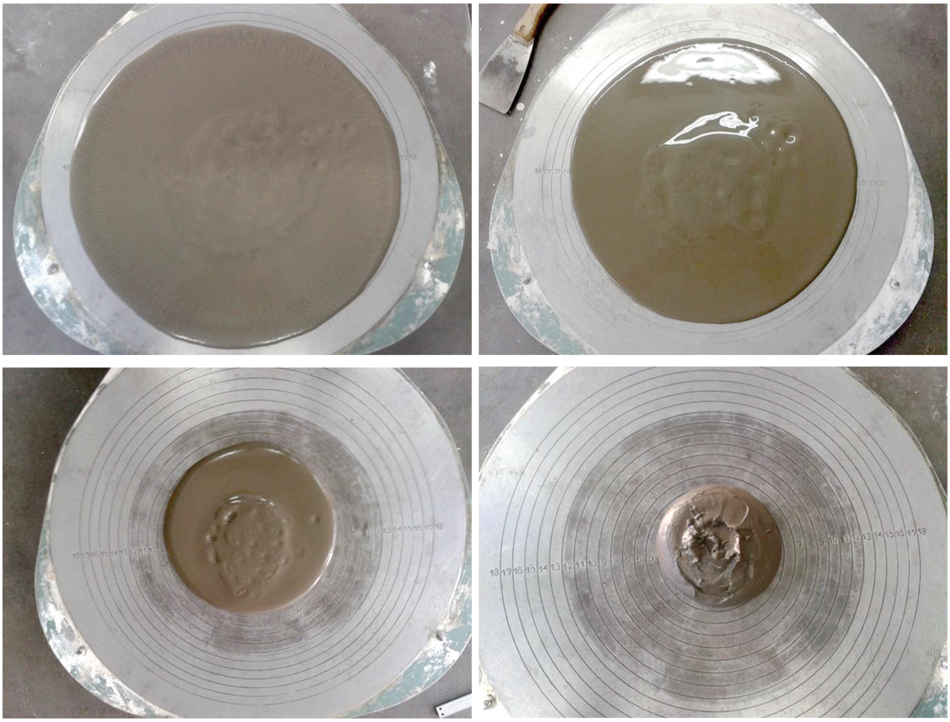

Fig. 1. Slump tests of cement pastes, W/C = 0.65, 0.60, 0.45, 0.35

Figure 1 shows the course of cement paste slump diameter measurement for W/C equal to 0.65, 0.60, 0.45, and 0.35, respectively. It is clearly visible that the slump parameters vary depending on the concentration of the paste. The measurement of slump diameter d does not cause any problems, while further interpretation of the *h* parameter is difficult, due to the fact that the mixture subsides noticeably at the central point of the slumped sample. This is particularly noticeable for low water to cement ratios, W/C = 0.45 and lower.

The value of the slump diameter d increases along with the increase in the water to cement ratio W/C. A strong correlation between the analysed parameters was observed. The determination coefficient reaches the value $R^2 = 0.9948$. Cement pastes with a higher W/C have a more liquid consistency and they are characterised by larger slump diameters. Pastes of water to cement ratios 0.65 and 0.60 were too diluted. Sedimentation of cement particles was observed. The particles fell to the bottom, while water accumulated on the surface of the paste. As the water to cement ratio decreased, the pastes were characterised by lower liquidity and the paste structure became more homogeneous, not subject to stratification. The visualisation of the variability of the measured slump parameters is shown in Fig. 2.

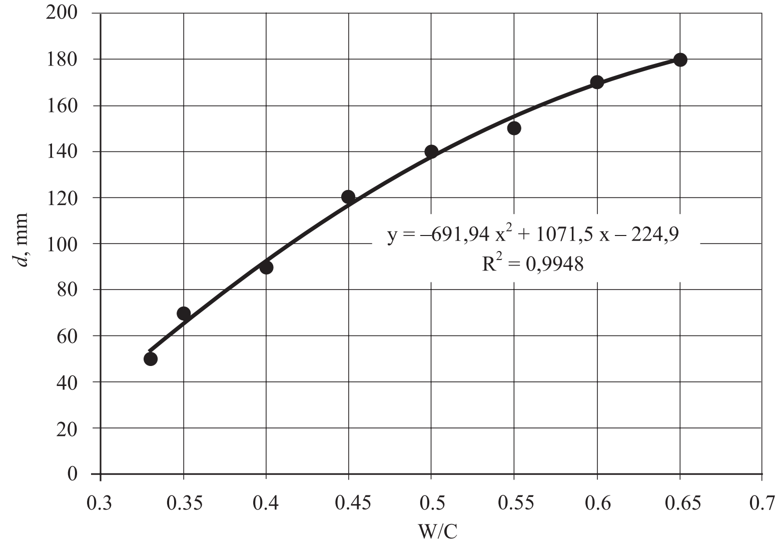

Fig. 2. Correlation d (W/C)

Viscometric tests

Along with slump tests, a rheological analysis of cement pastes was conducted. The obtained measurement results enabled us to obtain pseudo-curves of flow, presented in Fig. 3.

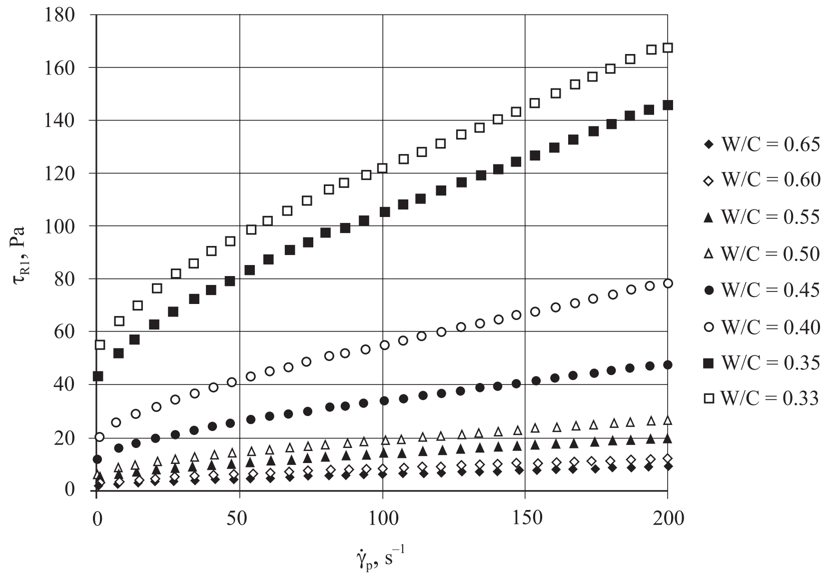

Fig. 3. Pseudo-curves of flow of cement pastes.

The analysed cement pastes behave like viscoplastic bodies, with a clear yield stress τ_0 and changeable plastic viscosity η_{pl}. Pseudo-curves of flow were adjusted to actual curves of flow pursuant to Krieger, Elrod and Maron, in compliance with the methodology presented in the study [Czaban 1987]. Curves of flow were approximated with use of the generalised 3-parametric Herschel-Bulkley model (1).

$$\begin{aligned} \tau &= \tau_0 + k\dot{\gamma}^n \quad \text{for} \quad \tau > \tau_0 \\ \dot{\gamma} &= 0 \quad \text{for} \quad \tau \le \tau_0 \end{aligned} \tag{1}$$

Actual curves of flow are presented in Fig. 4, and the obtained rheological parameters together with statistical evaluation (determination coefficient R^2, residual square sum RSS and residual standard error RSE) are listed in Table 2.

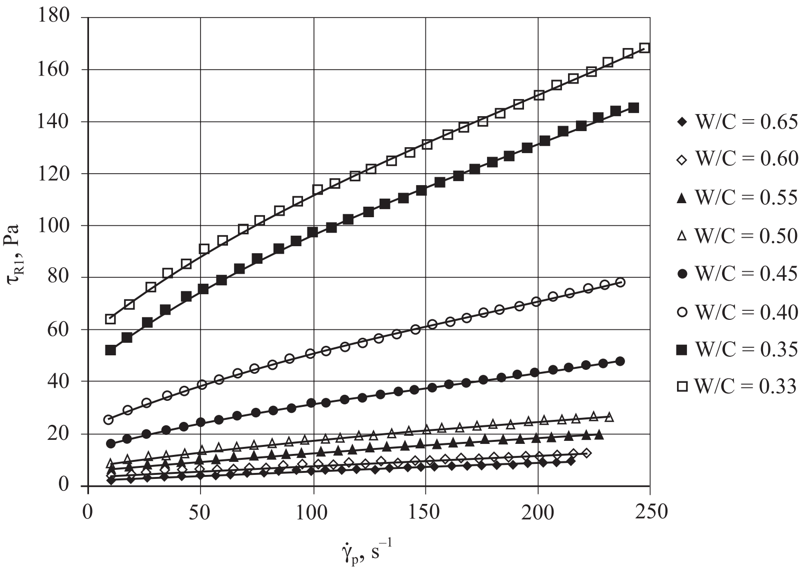

Fig. 4. Actual curves of flow of cement pastes.

Table 2. Rheological and statistical parameters of cement pastes

W/C	$\tau_{0,HB}$ Pa	k Pa · s^n	n –	R^2 –	RSS Pa^2	RSE Pa
0.65	1.830	0.085	0.832	0.9979	0.236	0.093
0.60	2.453	0.179	0.738	0.9987	0.224	0.091
0.55	4.382	0.265	0.756	0.9997	0.145	0.073
0.50	6.261	0.383	0.738	0.9998	0.202	0.087
0.45	12.560	0.512	0.785	0.9995	1.264	0.216
0.40	21.036	0.651	0.835	0.9996	2.633	0.312
0.35	42.057	1.436	0.794	0.9993	14.410	0.731
0.33	54.184	1.240	0.838	0.9991	24.279	0.948

ANALYSIS OF THE TEST RESULTS

The generalised, tri-parametric Herschel-Bulkley model adopted for the approximation of curves of flow properly describes the viscoplastic properties of cement pastes (Table 2). This is proven by the statistical parameters determined for the applied approximation. The residual standard error RSE informs us about the average deviation of theoretical values from empirical values. The lower the values of the error, the better adjusted the model. The RSE value for analysed cement pastes did not exceed 1 Pa. Basing on the obtained data, one may conclude that the suitability of the model decreases with the decrease in the water to cement ratio W/C.

The obtained rheological parameters allowed for an analytical and graphic presentation of the correlation together with paste consistency defined by the W/C parameter (Fig. 5, 6, 7).

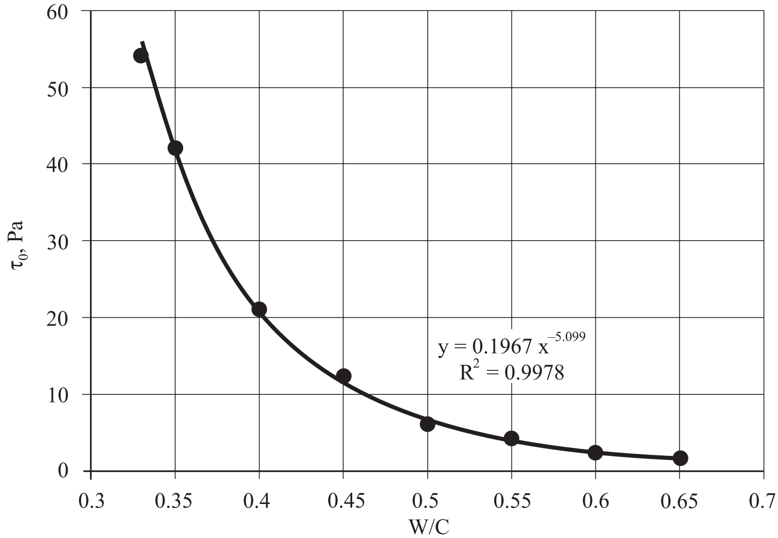

Fig. 5. Correlation τ_0 (W/C)

The yield stress τ_0 decreases noticeable with the increase in the water to cement ratio W/C, which demonstrates that the internal structure of the paste simplifies [Ferguson and Kembłowski 1995]. The correlation between the analysed parameters, expressed by the determination coefficient R^2 is strong.

Similar behaviour is observed for the k (W/C) correlation. The consistency index *k* decreases with the increase in W/C. The correlation between the analysed parameters should be considered as sufficient ($R^2 = 0.9567$).

The flow behaviour index *n* adopts changing values within the narrow range $n = 0.738$-0.838, with an unsatisfactory correlation with the water to cement ratio W/C. The change trend is slightly decreasing. For practical purposes, this parameter may be adopted as a constant, on the level of $n = 0.8$.

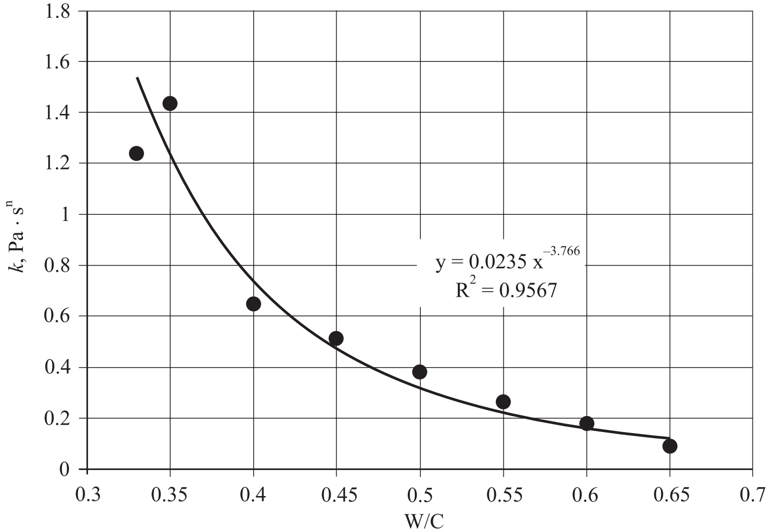

Fig. 6. Correlation *k* (W/C)

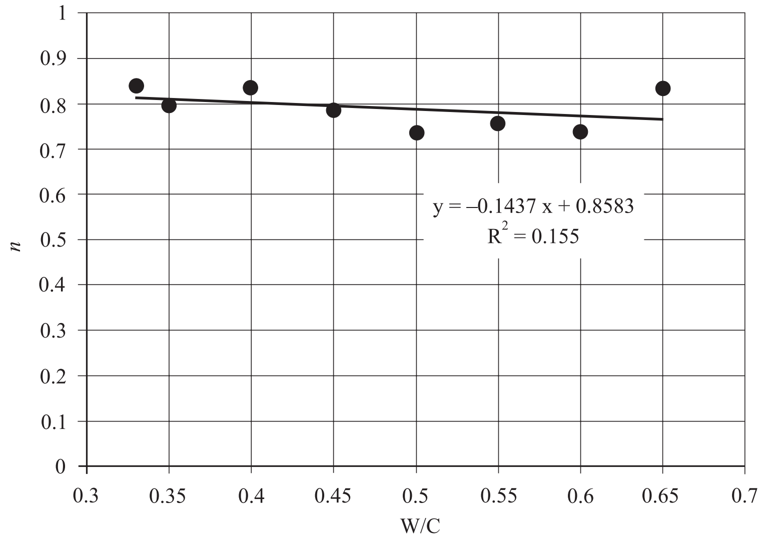

Fig. 7. Correlation *n* (W/C)

Knowing the slump parameters and rheological properties of the analysed cement pastes for various water to cement ratios W/C, the authors attempted to find correlations between them.

Figure 8 illustrates the correlation τ_0 (*d*), described by an exponential function that is characterised by strong suitability, expressed by the determination coefficient $R^2 = 0.9897$. The course of the function is decreasing. The yield stress τ_0 decreases with the increase in slump diameter.

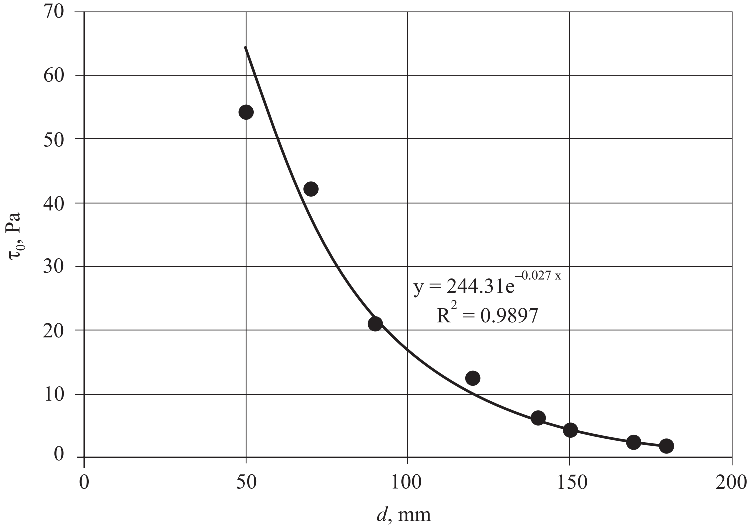

Fig. 8. Correlation τ_0 (*d*)

The correlation *k* (*d*), shown in Fig. 9, is characterised by a similar course, where correlation coefficient R^2 = 0.9213. In the opinion of the authors, such correlation is satisfactory for practical purposes. The consistency index *k* that describes the „viscosity" of the cement paste decreases with the increase in slump diameter *d*.

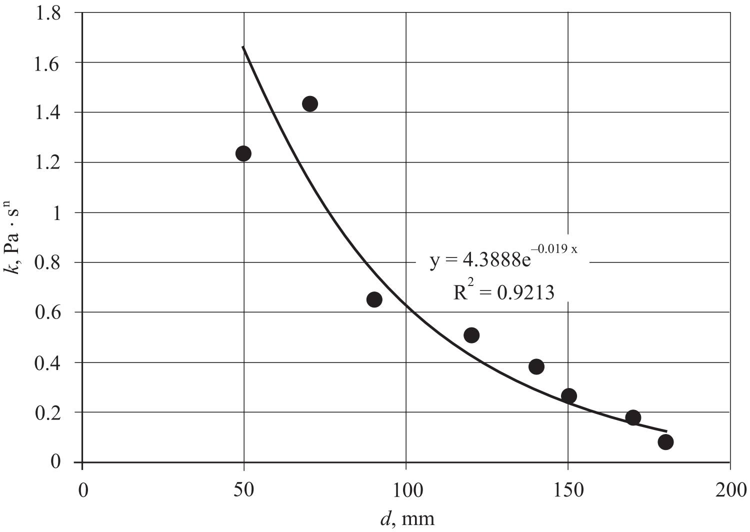

Fig. 9. Correlation *k* (*d*)

SUMMARY AND CONCLUSIONS

The conducted tests, concerning both slump parameters and rheological properties, allowed for the determination of their mutual correlation. For the analysed concentrations, described by the water to cement ratio W/C = 0.33–0.65, behave like viscoplastic mixtures, with a noticeable yield stress τ_0 and a variable plastic viscosity η_{pl}. The adequate Herschel-Bulkley model was applied for the approximation of curves of flow. The correlations of rheological parameters τ_0, k, and n as a function of water to cement ratio were determined by providing their mathematical formulae.

A similar functional relation was determined for the slump parameter *d* (W/C).

This enables to determine the rheological parameters of the model τ_0, k, n and the slump parameter *d* for any W/C ratio within the range of the analysed concentration levels.

The rheological parameters τ_0 and k are strongly correlated with the slump diameter *d*. Mathematically described formulae allow for the preliminary determination of the rheological parameters of the analysed cement paste basing on a simple slump test. This is important for practical applications. There is a need to evaluate e.g. the pressure loss in the pipe for paste transport, for example during injection works, which requires the knowledge of rheological parameters. Their approximate values may be calculated pursuant to the correlations provided in this study. The flow behaviour index n is not characterised by high variability for the analysed pastes, so, for practical purposes, it may be adopted at a constant value of $n = 0.8$.

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KONSYSTENCJA ZACZYNÓW CEMENTOWYCH A ICH PARAMETRY REOLOGICZNE

Streszczenie. W pracy przedstawiono wyniki badań parametrów rozpływu oraz parametrów reologicznych zaczynów cementowych, wykonanych na bazie cementu portlandzkiego CMII 32,5R. Parametry rozpływu ustalono z wykorzystaniem specjalnej płyty trasowanej, natomiast cechy reologiczne określono za pomocą wiskozymetru rotacyjnego Viscotester VT 550, firmy Haake, o cylindrach współosiowych, wyposażonego w system MV2. Zaczyny cementowe stanowiły mieszaninę cementu z wodą. Koncentracja mieszaniny, określana za pomocą wskaźnika wodno-cementowego W/C, zmieniała się w zakresie 0,65–0,33. Przeprowadzono normowe oznaczenie wytrzymałości użytego cementu, ustalając zgodność z danymi producenta oraz określono zależność parametrów rozpływu od wskaźnika wodno-cementowego W/C. Otrzymane, z badań wiskozymetrycznych, pseudokrzywe płynięcia zaczynów cementowych skorygowano do rzeczywistych krzywych płynięcia i aproksymowano przy pomocy 3-parametrowego, uogólnionego modelu Herschela-Bulkley'a. Określono zależności parametrów reologicznych modeli τ_0, k, n, w funkcji wskaźnika wodno-cementowego W/C. Ustalono występującą korelację pomiędzy parametrami reologicznymi i rozpływu dla badanych zaczynów cementowych.

Słowa kluczowe: zaczyny cementowe, rozpływ, model reologiczny, parametry reologiczne

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