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BRITTLENESS DETERMINATION FOR CEMENT BLENDS WITH SILICEOUS ADDITIVES

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Abstract. The paper presents research of brittleness cement blends with siliceous additives using impact strength. The advantages of the developed method are simplicity of definition, low cost of equipment and operation. In the presented research were studied additives on the basis of finely ground glass obtained from waste glass containers and pozzolanic additives as fly ash, silica fume and synthetic silica. Increase in additives amount in respect to the cement mass reduces brittleness by ~10 % after 28 days of curing. The addition of 35 % of ground glass decreases the brittleness of the cement paste, whereas 10 % of pozzolanic additive increase it during the period from 1 to 28 days. Determination of brittleness using impact strength can be used to evaluate the activity of siliceous additives in cement blends.

Keywords: cement, compressive and flexural strength, additives, brittleness.

1. Introduction

The main criterion for assessing the cement quality is compressive and flexural strength. However, in European Standard EN 197-1,[1] only compressive strength is taken into account when classifying the strength classes, although the standard EN 196-1[2] foresees test for both compression and flexural strength. From our point of view, when evaluating the strength, one should take into account both types of strength. It is possible only by taking brittleness as the strength criterion, *i.e.,* the ratio of tensile strength to compression strength.[3,4] Since the hardened cement is considered to be a brittle material with residual signs of plastic deformation after the setting beginning, its destruction can be connected with a high concentration of stresses at the apex of the cracks.[5]

Brittleness cannot be used as a strength assessment criterion *via* classical methods,[6] because in the case of determining the compressive strength (R_c) the destruction mechanism operates. This mechanism is associated with the achievement of the boundary tensile under the action of compression forces,[7] which leads to the sample destruction throughout the entire volume (Fig. 1a). When the flexural strength (R_f) is determined, there is a bending force that leads to destruction in one plane (Fig.1b). In other words, in both cases there are two different mechanisms of destruction.

a) b)

Fig. 1. Appearance of test beams after compressive test (a) and after flexural test (b)

Four ellipsoid fracture surfaces are formed when compressing, and only two square surfaces in the case of flexing. In order for the brittleness to actually reflect strength it is necessary that two parts with the same shape of the fracture surface are formed when determining the compressive and flexural strength. In this work, using the high rate of load change which takes place when determining the impact strength, we attempt to apply a brittle fracture to inhibit the phenomenon of plastic deformation.

2. Experimental

The chemical composition of the materials used in the studies is shown in Table 1. For the experiments we used CEM I 32.5 R in accordance with European standard.[1]

The cement mortars were produced according to the standard method EN 196-1.[2] Compressive strength

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and flexural strength were measured for prisms of mortar 40×40×160 mm. Compressive strength of mortar was determined by two methods. The first one – according to EN 196-1[2] and the second one – using wedging. When determining the compression strength with the use of wedging, a special cell was developed (Fig. 2). This method allows obtaining two parts with the same shape of the fracture surface.

Table 1. Chemical composition of materials

Oxides	Content, wt %					
	Colorless glass	Brown glass	Borosilicate glass	Silica fume	Synthetic silica	Cement
SiO_2	72.20	72.15	81.00	94.6	93.5	21.20
Al_2O_3	1.80	1.75	2.00	2.10	–	5.80
CaO	10.10	10.00	0.50	0.44	–	64.40
MgO	1.65	1.55	–	0.62	–	1.90
Na_2O_{eq}*	13.59	13.9	4.50	1.14	0.31	0.46
Fe_2O_3	0.04	0.25	–	1.10	–	3.60
Cr_2O_3	0.01	0.03	–	–	–	–
B_2O_3	–	–	12.00	–	–	–
SO_3	0.40	0.32	–	–	0.39	2.50

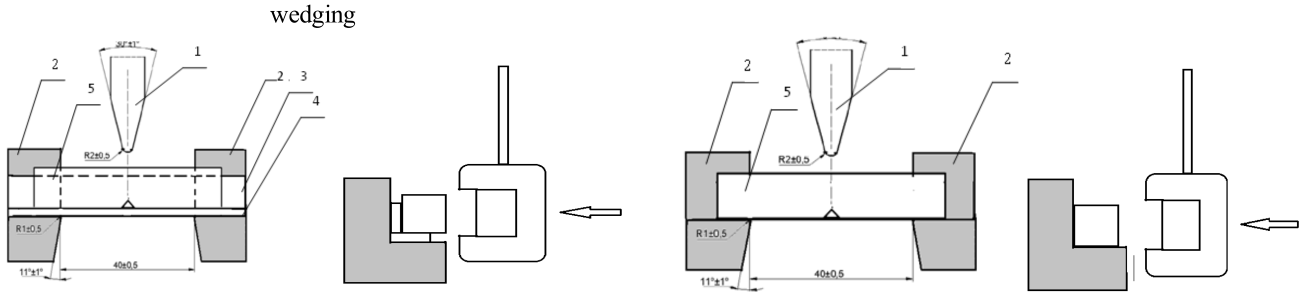
Note: * $Na_2O_{eq} = Na_2O + 0.658K_2O$

The impact strength was measured for prisms of paste 1×1×8 cm. To research impact strength hammer Charpy were employed.[8,9] To increase the accuracy of the impact strength determination for the samples prepared from cement paste to the standard impact tester Charpy an additional scale was developed, which allows to fit the hammer to a height where the energy is enough only for one fracture of the sample (Fig. 3). Block-diagram of impact strength determination using impact tester Charpy under the action of compressive and tensile forces is shown in Fig. 4.[10]

Special studies have shown that in the case of cement paste samples with the size of 10×10×80 mm, the hammer weight should not exceed 100 g. Dependence of energy of a hammer with the weight of 98.7 g on the position of additional scale is shown in Fig. 5.

Fig. 2. Cell for determining the compressive strength under wedging

Fig. 3. Impact tester with additional scale



a) b)

Fig. 4. Block-diagram of impact strength determination using impact tester Charpy under the action of compressive (a) and tensile (b) forces: hammer (1); supports (2); vertical metal liner (3); horizontal metal liner (4) and test sample (5)

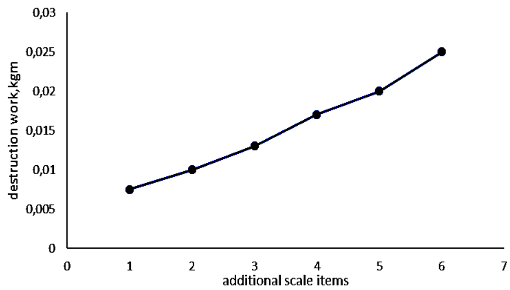


Fig. 5. Dependence of hammer energy on position of additional scale

Mechanical characteristics are presented as brittleness index K:

$$K = \frac{R_f}{R_c} \quad (1)$$

where R_f and R_c are impact strength at flexural and compression, respectively, J/cm^2.

Compressive strength of mortars was performed after 1 and 2 days. Impact strength of cement pastes was measured after 1, 2, 7, 28 days of curing. In order to compare the brittleness index results, the impact brittleness index activity (%) was used, where the impact brittleness index of paste with additive was compared to the impact brittleness index of control sample.

3. Results and Discussion

When determining the compressive strength by the method of wedging, a wedge acting under the influence of compressive force (with a small area of action) splits the sample into two parts with the same fracture surface. In this way the main disadvantage associated with the action of various destruction mechanisms inherent to the classical determination method of tensile strength and compression was eliminated. In order to inhibit the phenomenon of plastic deformation, which takes place in determining the strength *via* classical methods with low rate of load change, it is proposed to use brittle fracture at high load change rate.

Experimental results of strength are given in Table 2.

In accordance with Table 2, the brittleness index K is practically independent of the loading rate, and its value does not meet necessary condition:[3,6] $K = \frac{R_f}{R_c} < \frac{1}{8} < 0.125$

It means that despite the decrease in the K value over time, after two days the cement stone is not completely brittle. The wedge impact imprint on the surface also proves this fact. It can be argued that plastic deformation takes place at the first moment of load, and then a fragile fracture is observed in accordance with the theory of Griffits. At the same time, the increase in compressive strength under wedging leads to the value of K, which corresponds to the aforementioned condition. Thus, the accuracy of the method for determining the brittleness index K^2 and the impact test can be considered adequate, which allows the latter to be used to evaluate the activity of cement compositions.

It is known that the impact strength is considered to be one of the most commonly used mechanical properties in material science. Its advantage is simple production technology, low cost of equipment and maintenance. To the opinion of some researchers the method of determining the impact strength can be applied to assess the material rating, as well as for running control of its quality during continuous production.[11]

To verify the possibility of using impact test to evaluate the activity of siliceous additives, the basis of the composition was CEMI 32.5 R.

All additives were conventionally divided into two groups:

- group 1 - additives on the basis of finely ground glass obtained from waste glass containers;
- group 2 - pozzolanic additives.

Impact strength was determined using test beams, varying the amount of additives and setting time. The results are represented in Figs. 6 and 7.

Before all, it should be noted that regardless of the setting time, the additives of group 1 affect the brittleness index in different ways. For example, after one hour of setting, for colorless and brown glass (content of 6 %) the brittleness index decreases with respect to the control sample by 5 and 12.5 %, respectively, whereas the borosilicate glass, on the contrary, increases this value by 20 %. After seven days the brittleness index increases by ~18 and 36 % for the first two glasses and is practically not changed for borosilicate glass. After 28 days the index decreases by ~20–25 %. The increase in additives amount from 6 to 35 % decreases the brittleness index by ~10 %. The general tendency is the following: for additives (6 %) of group 1 the brittleness index increases during 7 days of setting with the subsequent decrease. After 28 days the index is almost equal for all additives.

For additives (6 %) of group 2 the brittleness index increases by ~38–40 % with an increase in setting time from 1 to 28 days. The increase in the additive amount to 10–35 %, as in the previous case, leads to the increase in the brittleness index.

So, the impact strength method gives an opportunity to observe the dependence of the brittleness index on the chemical composition of additives and setting time, which means the possibility of its application for the evaluation of the activity of cement blends with siliceous additives.

A rather high brittleness index after 24 h is most likely associated with the presence of liquid phase in structural pores.[12] This fluid plays the role of buffer at high load rates.[13,14] The results presented in Fig. 8 also confirm this fact.

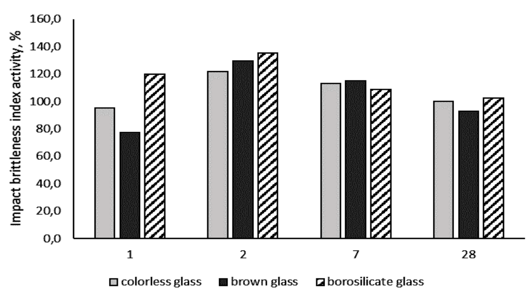
Table 2. Brittleness of samples from cement paste CEMI 32.5R determined by different methods

Method	Rate of load change	Strength after 1 day, MPa		Brittleness, K	Strength after 2 days, MPa		Brittleness, K
		R_f	R_c		R_t	R_c	
EN 196-1	1mm/min	1.38	3.36	0.41	3.73	13.42	0.28
EN 196-1*	1mm/min	1.38	41,8	0.03	3.73	162.2	0.02
Impact strength, J/cm^2	5.25 m/s	0.093	0.23	0.40	0.09	0.24	0.37

Note: *compressive strength was determined by the method of wedging.

Table 3. Composition of cement blends

Additive	Amount of additive, %	Water demand, %	Composition of blend, g/500g		
			Cement*	Additive	Water
Colorless glass	6	34.0	470	30	132
Colorless glass	35	34.0	325	175	144
Brown glass	6	34.0	470	30	132
Brown glass	35	34.0	325	175	144
Borosicate glass	6	28.5	470	30	131
Borosicate glass	35	28.5	325	175	134
Silica fume	6	48.4	470	30	136
Silica fume	10	48.4	450	50	142
Synthetic silica	6	80.0	470	30	200
Synthetic silica	10	80.0	450	50	260



a)

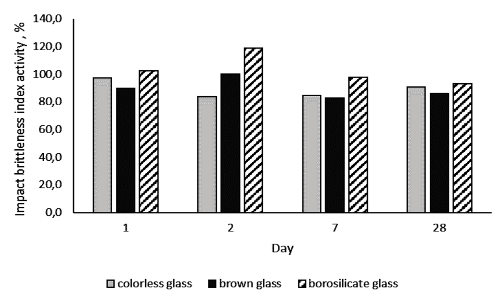


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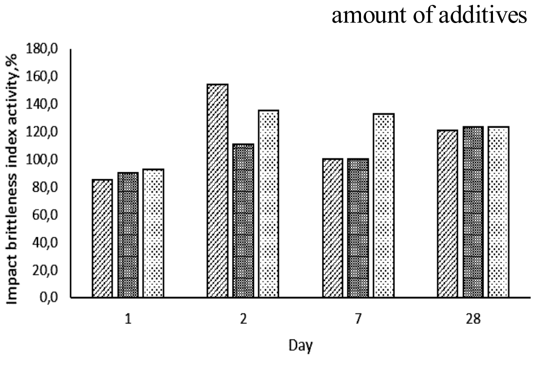
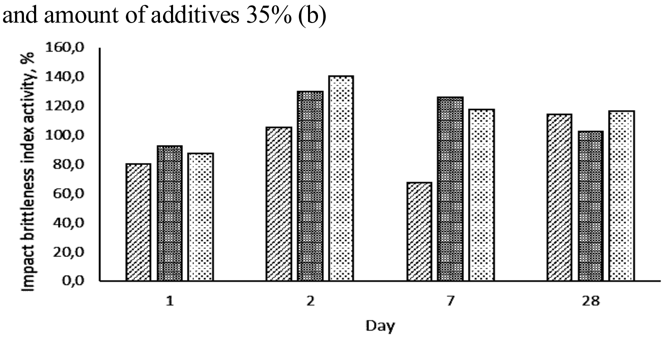
Fig. 6. Dependence of impact brittleness index on the amount of additives of group 1 and setting time: amount of additives 6% (a) and amount of additives 35% (b)



a)



b)

Fig. 7. Dependence of impact brittleness index on the amount of additives of group 2 and setting time: amount of additives 6% (a) and amount of additives 35% (b)

After 1 h of saturation with water only surface layers of cement samples were saturated. Their water saturation value is ~10 %, while the increase in impact strength varies within 5–7 %.

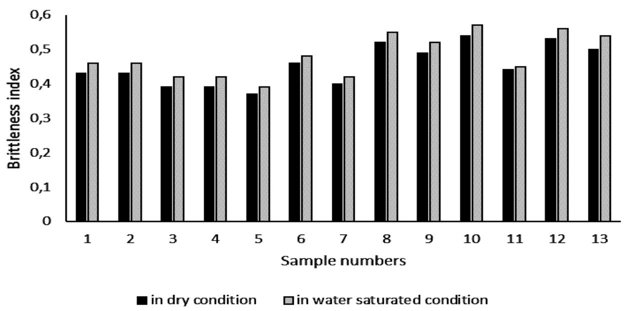


Fig. 8. Brittleness index after 28 days of setting under dry condition and after saturation with water

Thus, the brittleness index after 1 h of setting reflects not only the structural strength of the cement paste samples, but also indicates the presence of liquid phase in the material pores, which contributes to the increase in the index value.

A slight difference in the values of the brittleness index of the samples after one day and 28 days allows us to adopt a method for determining the impact strength, as accelerated procedure. This method is suitable to determine the brittleness index of the samples with siliceous additives, *i.e.,* it may be an evaluation criterion of additives activity.

4. Conclusions

Based on the performed studies, it can be argued that the method for determining the impact strength can be used to evaluate the activity of siliceous additives in cement blends with minor changes to the existing method (PN-ENISO 148-1:2017-02 or ISO 14556:2015-12), which is used for metals and plastic products. The advantages of the developed method, in comparison with the other ones, are simplicity of definition, low cost of equipment and operation. Taking the brittleness index as an evaluation criterion, the time of determination may be reduced to one day. This allows to use the developed method for systematic control of activity of cement blends with different chemical composition according to EN-PN 197.I:2012 standard.

The fragility index depends on the type and amount of additives introduced into the cement composition. The additives of the second group are more effective than those of the first one. In the first group, borosilicate glass is the most effective, which may be associated with the change in coordination of boron. In addition, the increase in additives amount from 6 to 35 % decreases the brittleness index by ~10 %. A similar picture is observed for additives of the second group. This suggests that regardless of the type of additives, replacement of cement by active Dilution helps to increase the compressive strength of cement compositions.

A characteristic feature of the method for determining the impact characteristics is that the saturation of the structural pores of the cement stone with water leads to an increase in brittleness due to amortization and a decrease in the propagation velocity of the shock wave in the material.

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ВИЗНАЧЕННЯ КРИХКОСТІ ЦЕМЕНТНИХ СУМІШЕЙ З КРЕМНЕЗЕМИСТИМИ ДОДАТКАМИ

Анотація. *З використанням методу ударної в'язкості досліджено крихкість цементних сумішей з кремнеземистими додатками. Показані перевагами розробленого методу: простота визначення, низька вартість обладнання та експлуатації. Досліджено додатки на основі тонкомеленого скла, одержаного з відходів скляної тари, та пуцоланових добавок, таких як зола виносу, кремнезем та синтетичний кремнезем. Встановлено, що збільшення кількості додатку по відношенню до цементної маси зменшує крихкість на ~ 10% після 28 днів твердження. Додавання 35% меленого скла знижує крихкість цементної пасти, тоді як 10% пуцоланового додатку збільшують її протягом 1–28 днів. Показано, що визначення крихкості за допомогою ударної в'язкості можна використовувати для оцінки активності кремнеземистих додатків у цементних сумішах.*

Ключові слова*: цемент, міцність на стиск і розтяг, додатки, крихкість.*