Pergamon

Cement and Concrete Research 32 (2002) 1181–1188

CEMENT AND CONCRETE RESEARCH

Effects of dosage and modulus of water glass on early hydration of alkali–slag cements

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Received 30 April 2001; accepted 22 October 2001

Abstract

This paper examines the early hydration of alkali–slag cements activated with water glass with different *n* moduli and sodium metasilicate ($Na_2SiO_3 \cdot 5H_2O$) in solution at 25 °C. The early hydration of alkali-activated blast furnace slag cements has been studied using isothermal conduction calorimetry. The cumulative heat of hydration increases by increasing the *n* modulus as well as the dosage of water glass, but is still lower than that of Portland cement. The compressive strength of normal-cured water glass slag cements is higher than Portland cement mortars. Drying shrinkage of alkali–slag cements is considerably higher than that of Portland cement. Consequently, industrial use of alkali–slag cement needs better understanding of the hardening mechanism and requires further research based on presented observations and results. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Alkali-activated cement; Calorimetry; Hydration; Compressive strength

1. Introduction

The goal of sustainable development of the cement and concrete industry is very important, and can be reached if we make a serious effort for the complete utilization of the cementitious and pozzolanic by-products produced in thermal power plants and metallurgical industries [1].

Finely ground granulated slag has become a widely used and important ingredient as a supplementary binder in ordinary Portland cement (OPC). Since slag itself is nothing more than latent hydraulic binder, it must be activated in practice to react and provide strength. It is recognized that alkali additions can activate pozzolanic materials to set and harden in their own right, and there exists considerable scope for the development of OPC-free cementitious binders that may have improved mechanical and durability properties over OPC-based binders. Considering the energy-intensive manufacturing process of OPC, such binders could eventually have an important role to play.

Notwithstanding the fact that the Portland cement will, probably, remain the basic determining binding substance in the nearest discernable future, Glukhowsky et al. [2] set a task to develop new highly active mineral binding materials that would substantially enhance the strength of concrete, their frost and corrosion resistance, and other qualities. The essence of idea of creating such binders according to Glukhowsky et al. [2] consists of the exclusion from the cement composition of highly basic minerals (C_3A, C_3S, and C_4AF), whose hydration products either have no essential effect on the synthesis of hydrated cement strength (C_3AH_6) or enhance its reaction capacity with corrosion mediums (C_3AH_6, C_3AH_{13}, C_2SH_2, $Ca(OH)_2$) and in the introduction instead of alkali activators ensures hydration of the alumosilicate substance of the cement. Slag is usually activated chemically but this kind of activation can be improved if it is combined with mechanical (more finely ground) or thermal (increased temperature) activation. A two-component system, alkali–slag cements, consist of a cementitious component such as blast furnace slag and an alkaline activator. In spite of that blast furnace slag may show little or no cementing properties, alkali–slag cements can develop very high strength in the presence of a proper alkaline activator.

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PII: S0008-8846(01)00717-7

According to Glukhowsky [3], caustic alkalis (ROH), nonsilicate salts of weak acid (R_2CO_3, R_2SO_3, R_2S, RF, etc.), silicate salts type ($R_2O(0.5-2.5)SiO_2$), aluminates ($M_2OnAl_2O_3$), aluminosilicates ($M_2OAl_2O_3(2-6)SiO_2$), and nonsilicate strong acid salts (M_2SO_4) can be used as alkali components of the slag–alkaline cements. The early hydration and reaction kinetics of alkali-activated binders have normally been studied using isothermal conduction calorimetry [4,5]. McCarter and Ezirim [6] and McCarter et al. [7] focus on monitoring the change of electrical properties (conductance and capacitance) in the study of the early hydration reaction kinetics of alkali-activated slag cements. Zhou et al. [8] concluded that the hydration process of alkali–slag cements could be classified into five periods based on the heat evolution curves (initial, induction, acceleration, deceleration, and period of slow, continued reaction), which is similar to that of OPC, but the mechanisms are different in both cases. Shi and Li [9] and Shi and Day [10–12] found that all caustic alkalis and alkali compounds whose anions or anion groups can react with Ca^{2+} to produce Ca compounds that are less soluble than $Ca(OH)_2$ can act as activators of slags. They have also found that due to the variation of activator, the hydration of alkali–slag cements can be described by three models: Type I — one initial peak occurs during the first few minutes and no more peaks appear thereafter; Type II — one initial peak appears before the induction period and one accelerated hydration peak appears after the induction period; and Type III — two peaks (one initial and one addition initial) appear before the induction period and one accelerated hydration peak appears after the induction period.

This paper examines the effect of activator dosage on early hydration of alkali–slag cement activated with a third group of alkalis according to Glukhowsky [3], using a conduction calorimeter. Water glass with different *n* moduli and sodium metasilicate was used as alkaline activator. The strength development and shrinkage of alkali-activated slag were investigated too.

2. Experimental

2.1. Materials used

Granulated blast furnace slag (GBFS; SARTID, Smederevo) with chemical composition in accordance with Table 1 has been used in our experiments. The OPC from Beocin has been used as a binder reference. The slag was activated with water glass [$Na_2O(n)SiO_2$] and sodium metasilicate ($Na_2SiO_3 \cdot 5H_2O$) in solution. The *n* modulus of liquid water glass (the mass ratio between SiO_2 and Na_2O) was 3.01 with 9.62% Na_2O and 29.0% SiO_2.

NaOH was added to change the *n* modulus, and water glass with *n* moduli of 0.6, 0.9, 1.2, and 1.5 was used in experiments. The chemical composition of solid sodium metasilicate was 29.26% Na_2O and 27.98% SiO_2. It was found that the mass of Na_2O, instead of total mass of activators, correlated best with the physical properties of alkali–slag cements [3]. Accordingly, all activators were added as 3% and 4% by mass of Na_2O for the physicomechanical property examinations and 3%, 4%, and 6% by mass of Na_2O for the heat of hydration. Quartz sand (0–2 mm) was used as aggregate in the mortar specimens.

Table 1
Chemical composition (%) and physical properties of GBFS and OPC

Item	GBFS	OPC
SiO_2	38.26	18.67
Al_2O_3	9.32	5.42
Fe_2O_3	0.60	2.44
CaO	40.49	60.72
MgO	8.70	2.22
SO_3	0.47	2.86
Na_2O	0.40	0.52
K_2O	0.39	0.31
S	0.42	–
MnO	0.81	0.05
FeO	–	0.24
P_2O_5	–	0.05
IL	0.42	3.12
IR	–	3.00
Total	99.86	99.62
Density (kg/m^3)	2920	3150
Blaine fineness (m^2/kg)	428	351

2.2. Measurement of heat evolution rates

The evolution of heat of hydration was monitored using a Tonical Isothermal Conduction Calorimeter. The mass of slag used was 5 g and a water-to-slag ratio of 0.43 was used. OPC was used as the binder reference with water-to-cement ratio of 0.5. Measurements of heat of evolution were performed at a constant temperature of 25 °C. When thermal equilibrium was achieved, the slag and the solution containing the alkaline activator were mixed by injecting the solution into slag.

2.3. Mortar mix proportions, handling, and curing

Alkali-activated slag mortar was manufactured according to Yugoslavian standard JUS EN 196-1, with a water-to-binder ratio of 0.43 while the OPC mortar was manufactured with a water-to-cement ratio of 0.5. The activator was added by dissolving in mixing water. The weight ratio between binder and aggregate was 1:3. Mortar prisms with a dimension of 40 × 40 × 160 mm were manufactured for determining other physical properties. After curing, the compressive strengths of the specimens were tested at the different ages up to 90 days. The mortar specimens of alkali–slag cements were kept in a climate room with a temperature of 20 °C (68 °F) and relative humidity (RH) of 90% directly after manufacture and up to testing. The

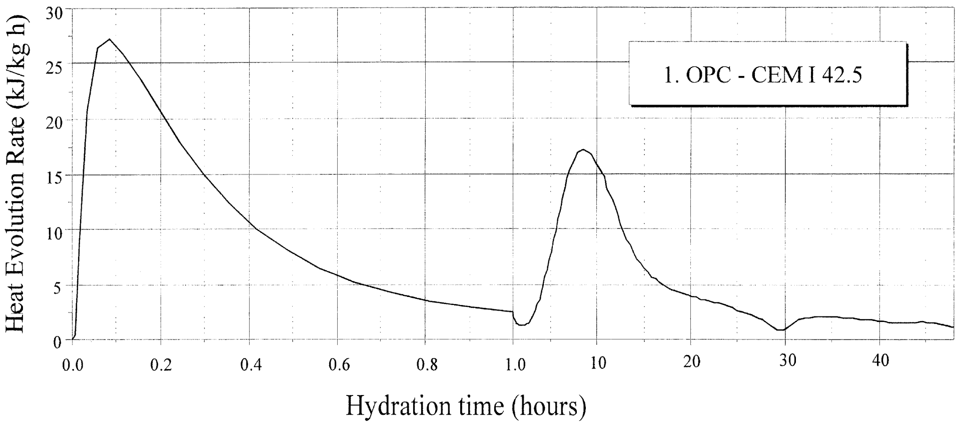

Fig. 1. Heat evolution rate of Portland cement, OPC-CEM I 42.5.

OPC specimens were kept in the water. The shrinkage was continuously followed up to 90 days. Immediately after stripping (after 1 day), the mortar prisms of alkali–slag cements were kept in climate room and the Portland cement prism in the water. The shrinkage that took place before stripping has not been studied. The length of prisms was then determined after 4, 7, 14, 21, 28, and 90 days of storage at 20 °C and 55% RH.

3. Experimental results and discussion

3.1. Effect of activator dosage on heat evolution rate

3.1.1. Portland cement + water

The heat evolution curves of Portland cement (Fig. 1) show that initial peak appears within first few minutes. *Preinduction* period signifies that immediately upon contact of cement with water, a rapid dissolution of ionic species into the liquid phase and the formation of hydrate phases occur. The rather intense liberation of heat with a maximum within a few minutes is due to the rapid hydration of C_3S and C_3A [13]. The heat evolution curves then decline to an *induction period* lasting from 1 to 2 h. In this stage, the hydration of all the clinker minerals progresses very slowly. The concentration of calcium hydroxide in the liquid phase reaches its maximum and starts to decline. This is followed by an *accelerated* hydration peak that results from the hydration of C_3S and formation of C-S-H phase and portlandite. The calcium sulphate becomes completely dissolved and the concentration of SO_4^{2-} in the liquid phase start to decline due to the formation of the AFt phase during the *deceleration period*. In the *postacceleration* period, the hydration rate slows down gradually and the rate of the hydration process becomes diffusion-controlled.

3.1.2. Slag activated with different-moduli water glass as well sodium metasilicate

Fig. 2 illustrates heat evolution rates of alkali–slag cements activated by different amounts of water glass with $n = 0.6$ at 25 °C.

The slag hydration process must be considered with regard to the oxygen forms present in slag, double-fixed in the $\equiv Si-O-Si\equiv$ bond, single-fixed in $\equiv Si-O-Me-$ bonds,

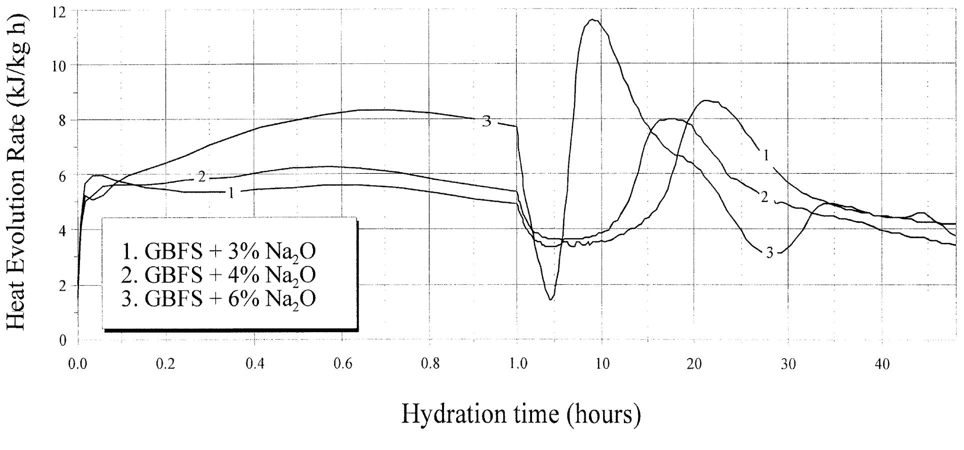

Fig. 2. Effect of water glass with $n = 0.6$ on heat evolution rate of alkali–slag cement.

and "free" in $-Me-O-Me-$ bond and the conditions of hydrate phase formation [14]. The reaction in aqueous medium proceed according to the following equations:

$$\equiv Si-O-Si\equiv + H{\cdot}OH \rightarrow 2\equiv Si-OH$$

$$\equiv Si-O-Me-O-Si\equiv +2\ H{\cdot}OH \rightarrow 2\equiv Si-OH = + Me^{2+} + OH^{-}$$

$$\equiv Si-O-Me{\cdot}\cdot\cdot O\cdot\cdot{\cdot}Me-O-Si\equiv + 3H{\cdot}OH \rightarrow 2\ \equiv Si-OH + 2\ Me^{2+} + 4OH^{-}$$

The presence of alkali cation, which at the stage of destruction–coagulation structure formation, first acts as destruction catalyst and then, with subsequent development of condensation structure alkali content decreases as a result of its bonding into new formations, which suggest that the alkaline component affects essentially the structure formation processes according to the following equation:

$$\equiv Si-OH + NaOH \rightarrow \equiv Si-ONa + H{\cdot}OH$$

These alkaline silicates may undergo reactions of ion interchange with divalent ions, forming $\equiv Si-O-Ca-OH$-type complexes [15]. Aluminia is present in similar formations, hydroxyl groups affect in the same manner the $Al-Si-O$ bond, giving rise to soluble aluminate anion $Al(OH)_4{}^{-}$ hydroxocomplex and surface groups $\equiv Al-OH$ [16].

Alkaline activation of blast furnace slag is affected due to the high concentration of OH^{-} ions in the mix [17,18]. Due to the present of alkali (Na^{+} or K^{+}), even in small amount, the pH value in Portland cement is usually more than 13.0. Destruction of the dispersed phase of alkaline cement, in contrast to that of Portland cement, has its own peculiarities caused by the lower basicity of the initial minerals and the presence of high concentration of OH^{-} ions in the dispersion medium.

The small peak appears just after the addition of the activator solution, which is followed by a very diffuse peak during the preinduction period [10]. When the activator dosage is 3%, the initial peak and additional initial peak combine into one and occurs after 6 min. With increasing water glass dosage, initial peaks appear earlier (5 and 3 min), additional initial peaks are higher and more diffuse than initial peaks and appear after 30 and 40 min, respectively. The first destruction step under high concentration of OH^{-} can be described as the breaking of $Me-O$ ($Me\equiv Ca,Mg$), $Si-O-Si$, $Al-O-Al$, and $Al-O-Si$ of the slag grain. Since the $Me-O$ bonds are much weaker than $Al-O$ and $Si-O$ bonds, more Ca and Mg enter into water than Al and Si, and the Si/Al-enriched layer forms quickly on the surface of slag [19,20]. The phase structure of the slag provided channels for OH^{-} ions to enter the bulk to complete the hydration process. Besides, the dispersed medium has a high concentration of $[SiO_4]^{4-}$ from water glass, and the first step may correspond to the hydration and hardening of the water glass or reaction between $[SiO_4]^{4-}$ and Ca^{2+} dissolved from the surface of slag grains. The initial peak corresponds to the wetting and dissolution of slag grains and adsorption of some ions onto the surface of slag grains, and the second peak to the formation of precipitate layer at the slag particle surface [10]. Hydration products have formed in the space between the slag grains, originally occupied by alkaline solution, indicating their formation by dissolution and precipitation mechanism at early ages [21]. The higher the water glass dosage is, the higher are the combined and accelerated hydration peaks. The accelerated hydration peaks for 3%, 4%, and 6% activated slag were higher than the corresponding combined preinduction peaks. The precipitation of C-S-H gel results in a very long induction effect. All curves show an appreciable induction period and 22 h is almost on the top of the accelerated hydration peak for alkali–slag cement activated with 3% water glass. The longer induction period, varying from about 5 to 14 h, is observed before the appearance of the accelerated hydration peak. The higher the activator dosage, the shorter is the induction period. When the slag grains were

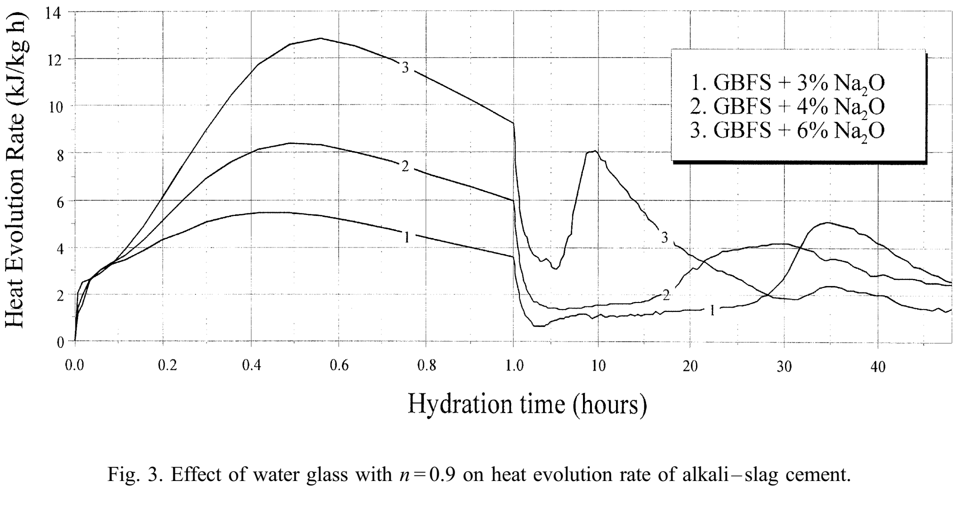

Fig. 3. Effect of water glass with $n = 0.9$ on heat evolution rate of alkali–slag cement.

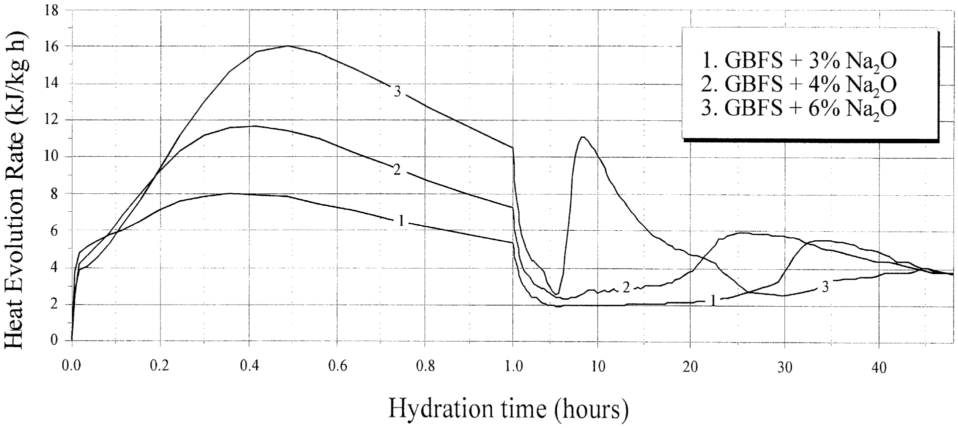

Fig. 4. Effect of sodium metasilicate on heat evolution rate of alkali–slag cement.

coated by a hydrate layer, the hydration kinetics was controlled by a diffusion process [4] until the completion of the reaction, the diffusion process being affected by the degree of structural defectiveness [16].

The magnitude of accelerated hydration peak of the cement with 6% of water glass is higher than that with 3% and 4% but still lower than the accelerated peak of Portland cement.

Fig. 3 illustrates heat evolution rates of alkali–slag cements activated by different amounts of water glass with $n = 0.9$ at 25 °C.

The small peak appears just after the addition of the activator solution, which is followed by a diffuse peak during the preinduction period. When activator dosage is 6%, the initial peak appears first and occurs after 2 or 3 min. With increasing water glass dosage, the initial peak appears earlier and additional initial peaks were higher and less diffuse. The higher the water glass dosage, the higher are the combined and accelerated hydration peaks. The precipitation of C–S–H gel results in a very long induction effect. All curves show an appreciable induction period and 35 h is almost on the top of the accelerated hydration peak for alkali–slag cement activated with 3% water glass. The longer induction period, varying from about 6 to 27 h, is observed before the appearance of the accelerated hydration peak. The higher the activator dosage, the shorter is the induction period. In contrast to slag paste activated with water glass with $n = 0.6$, the accelerated peak with $n = 0.9$ is lower than the additional initial peak during the preinduction period.

Fig. 4 illustrates heat evolution rates of alkali–slag cements activated by different amounts of sodium metasilicate $Na_2SiO_3x5H_2O$ at 25 °C. Compared with slag paste activated with $n = 0.9$, very similar features of heat evolution rates can be observed. Slightly higher effectivity in sodium metasilicate-activated paste during preinduction is visible. All curves continued to have a trend of lower induction period peaks compared with additional initial peaks during preinduction period.

Figs. 5 and 6 illustrate heat evolution rates of alkali–slag cements activated by different amounts of water glass with $n = 1.2$ and $n = 1.5$, respectively, at 25 °C. From the Figs. 5 and 6, the trend of magnifying the additional initial peaks by increasing the modulus as well as the dosage of water glass is observed. The higher the water glass dosage, the higher are the combined and accelerated hydration peaks. The precipitation of C-S-H gel results in a very long induction

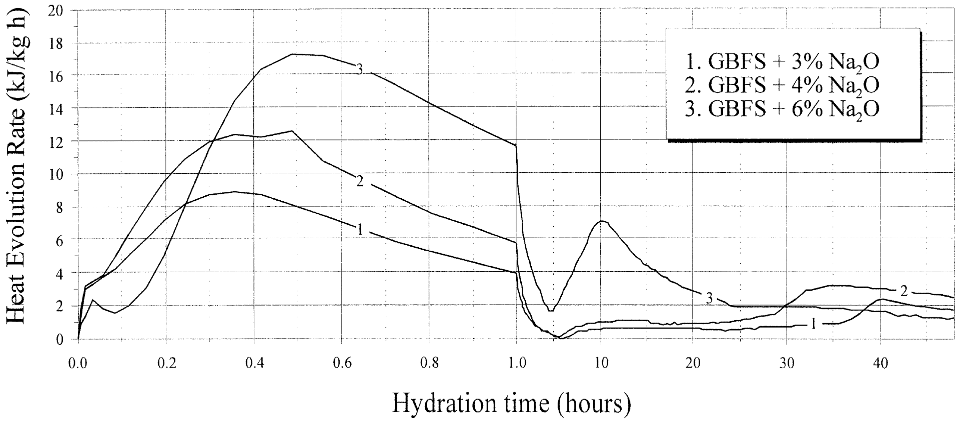

Fig. 5. Effect of water glass with $n = 1.2$ on heat evolution rate of alkali–slag cement.

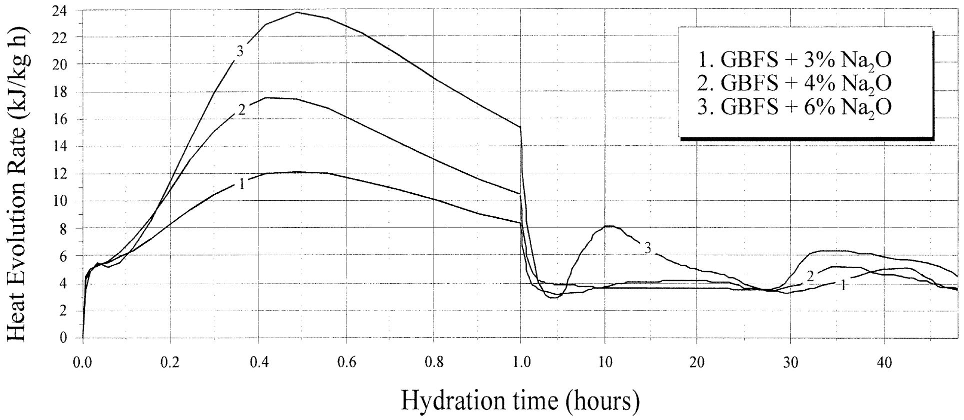

Fig. 6. Effect of water glass with $n = 1.5$ on heat evolution rate of alkali–slag cement.

period and the top of the accelerated hydration peak with 3% of water glass with moduli of 1.2 and 1.5 is about 40 and 43 h, respectively.

The longer induction period, varying from about 5 h, for slag activated with 6% of water glass with moduli of 1, 2, and 1.5, to 36 h, is observed before the appearance of the accelerated hydration peak. The higher the activator dosage, the shorter is the induction period.

3.2. Effect of activator dosage on cumulative heat of hydration

The effect of activator dosage on cumulative heat of hydration of alkali–slag cement at 25 °C is illustrated in Figs. 7–9. Increasing the modulus of water glass as well as dosage shows an increase in cumulative heat of hydration.

During the first 48 h, water glass with different moduli as well sodium metasilicate-activated slag releases less heat than Portland cement at a given isothermal temperature for all activator dosages. Thus, for alkali–slag cements activated with water glass or sodium metasilicate, the contribution of heat of hydration depends on the nature of slag, the modulus, and the dosage of activators used. According to results obtained, some authors [8,10] reported that alkali–slag cements cannot be regarded as low-heat cements. Our experiments did not include dosage over 6% Na_2O by the mass of slag.

3.3. Effect of modulus and activator dosage on physicomechanical properties of alkali–slag cements

The *strength development* of alkali–slag cement mortars activated with different moduli (n) and dosage of water glass and sodium metasilicate was studied using Portland cement as control. Considering all factors affecting strength development of Portland cement, the nature and the dosage of activators distinguish itself playing the crucial role in determining the strength of alkali–slag cements. Many results confirm the effect an alkaline activator has on strength development and that it may be different for slag of different origins [22,23]. The growth in compressive strength between

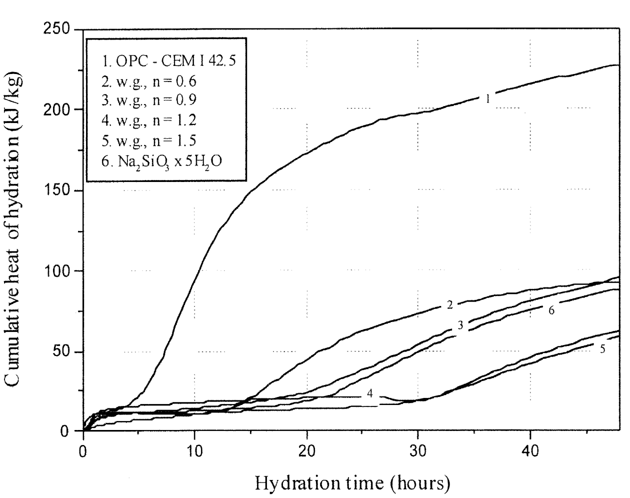

Fig. 7. Effect of water glasses with different n moduli and sodium metasilicate in the dosage of 3% Na_2O of slag weight on cumulative heat of hydration of alkali–slag cement compared to Portland cement at 25 °C.

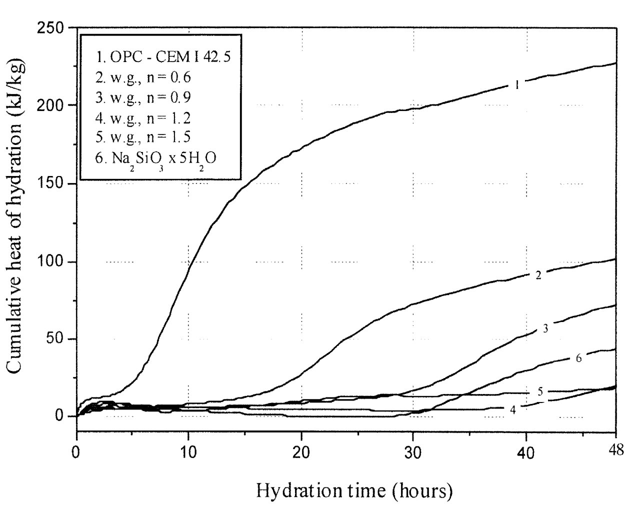

Fig. 8. Effect of water glasses with different n moduli and sodium metasilicate in the dosage of 4% Na_2O of slag weight on cumulative heat of hydration of alkali–slag cement compared to Portland cement at 25 °C.

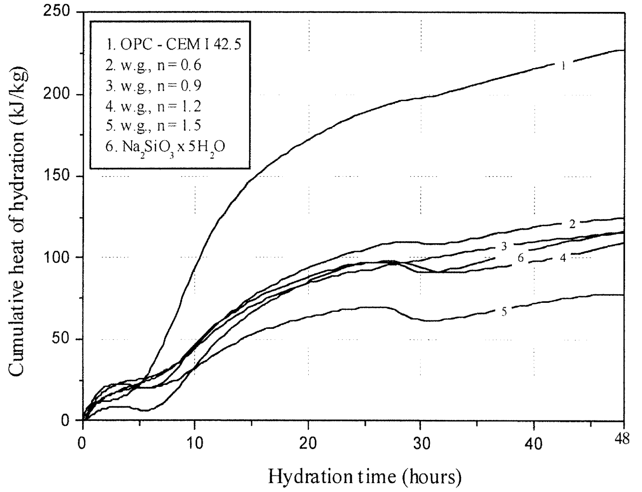

Fig. 9. Effect of water glasses with different *n* moduli and sodium metasilicate in the dosage of 6% Na_2O of slag weight on cumulative heat of hydration of alkali–slag cement compared to Portland cement at 25 °C.

different alkali-activated slag mortars and OPC is compared in Figs. 10 and 11.

All alkali–slag cements show higher ultimate strengths than Portland cement, but only the cement activated with 4% of water glass by weight of Na_2O ($n = 0.6$) show higher strength at early age — after 1 day. The cements with higher dosage of water glass show rapid strength gain after 3 days. The lower initial strengths of alkali–slag cements can be partly attributed due to length of induction period as mentioned before. More work is needed to establish relationships between the strength of alkali–slag cements and various factors.

The *shrinkage* in the water glass as well sodium metasilicate-activated slag mortars that were air-cured is considerably higher than that of corresponding prisms with OPC. Water glass with a high *n* modulus gives greatest shrinkage, followed by water glass with lower *n* modulus and OPC, respectively, as shown in Fig. 12. Some authors reported that alkali-activated slag concretes based on water glass often undergo greater drying shrinkage due to formation of silica or silica-rich gel during hydration [24–26]. According to Glukhowsky at al. [27], the initial phases formed in alkali-activated slag are rich in silica hydrosilicates and silica acid. The silica acid can be polymerized into silica gel. The ease of the silicic acid to condense is increased at alkaline pH where it is slightly dissociated or in molecular state and occurs within only a few minutes. A silica gel has a high water content and begins to shrink, expelling water from the mass. The shrinkage begins with this syneresis process, when free liquid enmeshed in the gel is spontaneously expelled. Syneresis comes to an end when the water content of the gel is still high, about 90%. On further drying, the structure of the gel continues to shrink and number of particle-to-particle bonds in the structure gradually break.

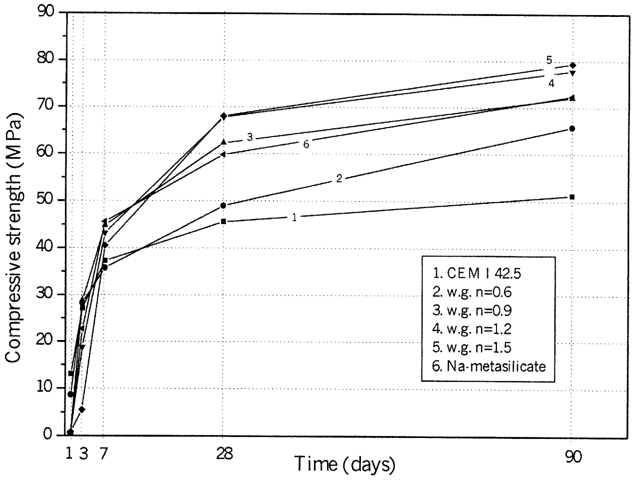

Fig. 10. Effect of water glasses with different *n* moduli and sodium metasilicate in the dosage of 3% Na_2O of slag weight on compressive strength of mortar compared to Portland cement.

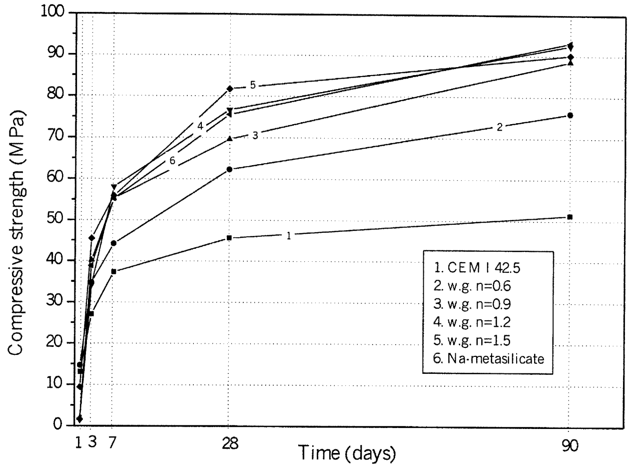

Fig. 11. Effect of water glasses with different *n* moduli and sodium metasilicate in the dosage of 4% Na_2O of slag weight on compressive strength of mortar compared to Portland cement.

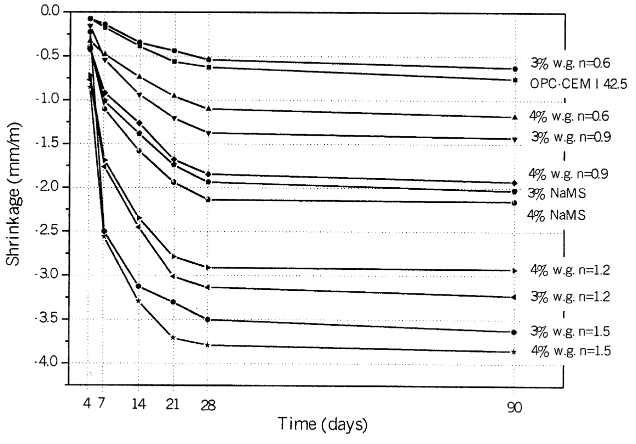

Fig. 12. Effect of water glasses with different *n* moduli and sodium metasilicate at the dosage of Na_2O by the mass of slag on shrinkage of alkali–slag cement compared to Portland cement.

4. Conclusions

Hydration process of alkali–slag cements and OPC are similar to each other, but mechanisms of hydration are different.

- Water glass-activated slag displays two heat evolution peaks during preinduction period, and by increasing the modulus of water glass, the value of additional initial peak increases and induction period lasting increases as well.
  By increasing the dosage of activator, the induction period decreases.
- Cumulative heat of hydration increases by increasing the *n* modulus as well as dosage of water glass, but is still lower than that of Portland cement.
- Alkali–slag cements activated with water glass with moduli between 0.6 and 1.5 in appropriate dosage show higher ultimate strengths than Portland cement.
- Drying shrinkage of alkali–slag cements is considerably higher than that of Portland cement and increases by increasing the modulus as well as the dosage of water glass.
- The alkali–slag cement, as low-cost product and with the possibility of using by-products and wastes in alkali-activated slag mixes, provides a solution to the present ecological problem.

Acknowledgments

The authors thank the Republic of Serbia Ministry of Science, Development and Technology for funding this research through Project No. 02 E 41. Thanks to Dr. M. Komljenovic for useful discussion during paper preparation. Thanks to Mr. Z. Slovic and Mrs. E. Ostojic for the blast furnace slag supply and Mr. S. Dakovic and N. Dacic for water glass and sodium metasilicate supply.

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