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TECHNIQUES OF DETERMINATION SETTING TIME OF CEMENT SYSTEMS

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During concrete works in many cases one of the factors influencing manufacturing techniques of designs is the "setting time of cement system" parameter. In the work it is shown that today in different sources the term "end and beginning of setting" is interpreted in different ways. Methods of their determination are also various. So, it is possible to draw a conclusion that first of all it is necessary to concretize and tie the beginning and the end of a setting to internal processes in the cement-water system.

The regulating document used in Republic of Belarus and Russia GOST 30515-97 [1] enters such indicator of quality of cement as setting times. GOST 30515-97 [1] defines setting time of cement as the start and end of the cement paste setting in the normalized conditions. According to setting time cements are divided into:

– slow-setting — with the normalized starting time of a setting more than 2 h;

– normal-setting — with the normalized starting time of a setting from 45 min. to 2 h;

– fast-setting — with the normalized starting time of a setting less than 45 min.

At the same time the encyclopedia of terms, determinations and explanations of construction materials [2] defines the term "setting" as the irreversible process of slump loss of the cement paste up to transition to a firm condition characterized by indicators of the beginning and end of a setting.

According to [3] setting is the process during which rather fluid mix of cement with water gradually gets denser and obtains such initial durability at which its mechanical conversion becomes almost difficult and even impossible (at the end of a setting). Therefore the binders including cement should be characterized by such terms of setting which give the possibility to prepare mortar and concrete mixes and to use them. Prior to the beginning of a setting paste has thixotropic properties. As approaching the end of a setting cement paste or concrete mix becomes worse processed. Setting of cement in accordance to GOST 30515 97 [1] is called irreversible slump loss of the cement paste as a result of hydration.

Process of cement hardening according to the theory of hardening of binders, developed by the academician A. A. Baykov [4], is conditionally divided into three periods: preparatory, kolloidation and crystallization. In the preparatory period particles of cement are moistened with water and are dissolved from a surface; with time saturated solution is formed. During this period, lasted 1 … 3 h, cement paste is plastic and easily formed. During the period of kolloidation the concentration of new hydrated growths increases in the solution. The formed connections differ in smaller solubility from clinker minerals. Therefore the solution saturated in relation to initial connections is oversaturated in relation to new growths. Hydrated new growths in the form of the smallest colloidal parts — submicrocrystals — are allocated from solution, forming cement gel. Emergence of a large amount of gel leads to thickening of the cement paste which loses plasticity. The moment of thickening (setting) of cement paste comes in 3 ... 5 hours after mixing with water cement. Strength of the thickened paste during this period is still small. The beginning of a setting is characterized by formation of reversible coagulative and crystallizational structure of a cement stone when separate particles are linked in pitches, chains, spatial grids through liquid layers by van-der-vaals forces. The period of crystallization is characterized by further hydration of cement. Gel will gradually be transformed into crystal joints. The condensation and crystallizational structure of a cement stone with chemical bonds between particles is formed. Cement gel loses a significant amount of water, and there comes the end of a setting. The structure loses ability to dissolve and restore thixotropically after removal of mechanical influence.

Now the mechanism of hydration of cement components is a subject of discussions and disagreements [5]. The early theory of La-Chatelie says that hydration of cement happens through dissolution of waterless connections, which are followed by bounding and crystals setting of the hydrated connections. Mikhaelis considered that cohesion is the result of formation and the subsequent drying of gel. The idea of the topochemical or solid phase mechanism was developed.

The idea of the mechanism of hydration of C_3S, main phase of cement isn't still clear. It's offered to take into consideration some stages of process of hydration. 5 stages on a thermokinetic curve calorimetry in isothermal conditions are noted. At the 1^{st} stage there is jump of speed of a thermal emission, then the fall within 15–20 minutes. It is the preinductive stage. On the 2^{nd} stage the speed of reaction is very low. It is the inductive period, lasting several hours. It is supposed that the 1^{st} and 2^{nd} stages, which can be influenced by the means of additives, significantly influence the subsequent hydration of C_3S. At the 3^{d} stage the reaction goes actively, reaching a maxi-

mum by the end of the stage. Time of "the beginning of a setting" approximately matches with the time when the speed of reaction begins to increase greatly. Time of "the end of a setting" is the completion time of the 3^{d} stage. At the 4th stage speed of a thermal emission decreases and hydration of C_3S continues. On the 5th stage a small amount of products of hydration of C_3S is formed. According to Akhverdov N. I. [6] the term "the beginning and the end of a setting" can be nominally characterized by the beginning and the end of the induction period.

The kinetics of structuring of the cement paste is characterized by reversible and irreversible processes depending on properties of the bonds of the crystal hydrate of complexes. If by the term "the beginning of setting" is meant an initial stage of forming of a microstructure of the cement paste, during which it has the reversible thixotropic properties, contributing not only to initial reduction, it is possible to tell that such phenomenon corresponds to the end of adsorption of liquid of all hard phase. Therefore, this should be corresponded by the structure of the cement paste which is created at a final stage of the induction period.

The concept "the beginning of a setting" is characterized by rheological properties of cement mixture and is only on average connected with internal physical and chemical transformations. All internal processes after the beginning of interactions of cement with water go cyclically as cement and water are heterogeneous in composition [7]. Therefore, even under production conditions in the work shop the time of the beginning of treatment during molding designs from several portions of the concrete mixtures, that shut at different times, (to determine the moment of the beginning of setting) is an intractable problem. Moreover the usage of all offered methods of active and passive impacts, even such low-power-intensive as magnetic handling and especially electrohandling, in technology of monolithic concrete is rather problematic.

In case of construction of monolithic designs usually several batches are loaded into means for delivery of concrete mixture. During transportation, because of various impacts there is a consolidation of all batches in single mass. For average mass even if it satisfies all requirements of technology, the concept of the moment of a mixing becomes abstract, but not the initial datum point in the transformations happening in already difficult cement system. In all considered handling types the moment of the beginning of impact is connected with the certain interval of time, which counting is conducted from the beginning of mixing.

Stacking of concrete mixture is performed by three methods: with sealing, casting (concrete mixtures with supersofteners) and pressure tight packing. For each method of packing the basic rule should be followed - the new portion of concrete mixture shall be laid prior to the beginning of cement setting in earlier placed layer [8]. According to foreign researchers [9–13] terms of cement setting can be determined by means of Gillmore apparatus (ASTM C 266) [9] and Vicat apparatus (ASTM 191) [10].

Рис. 1 Vick's device

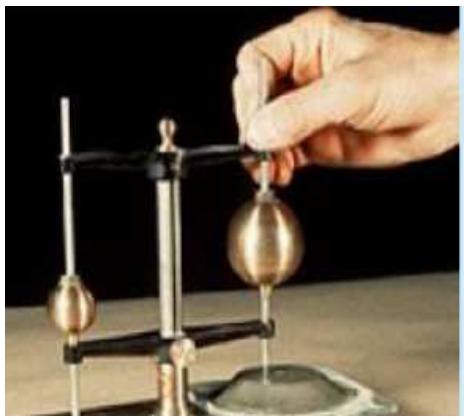

Рис. 2 Gilmore's device

Table 1 – The results of determination of setting time by means of Vicat apparatus and Gillmore apparatus

Setting time	Vicat apparatus	Gillmore apparatus
Portland cement (ASTM C 150) [11] the beginning setting (ht., min.)	0: 45	6:15
the end of setting (hr.,min.)	1:00	10:00

Working procedure with the help of Gillmore apparatus is the following: on flat pat from the cement paste with the diameter of 75 mm and with the thickness 12,5 mm, formed on glass, a prick with a needle is put. To determine the time prior to the beginning of a setting the needle with the weight of 113,4 g and with a diameter of 2 mm is used, however for determination of time until the end of a setting the similar needle has the mass of 453,6 g and the diameter of 1 mm. The time prior to the beginning of a setting is determined as time when the

first needle of Gillmore doesn't leave a noticeable prick, and the time until the end of a setting — when the same occurs with the application of the second needle. All standard cements must have the time prior to the beginning of a setting not less than 60 min., and the time until the end of a setting — no more than 10 h; when applying a Vicat needle — 45 min. and 8 h respectively.

The technique of work with Vicat apparatus is similar to the described above; only the weight and the diameter of a needle and the sizes of a sample from the cement paste are various. The moment of the beginning of a setting is determined at free lowering of a needle. The needle is immersed into paste every 10 min., moving a ring after each immersion so that the needle doesn't get to the former place. In this method the time prior to the beginning of a setting is fixed when the needle passes into paste to the depth of 25 mm, and the time until the end of a setting is fixed when a needle doesn't get into cement paste considerably. The beginning of a setting of the cement paste is considered as the time which passes from the beginning of a mixing (the water sticking moment) to the moment when the needle doesn't reach a plate on 2-4 mm. The end of a setting of the cement paste is considered the time from the beginning of a mixing to the moment when the needle falls to dough no more than by 1-2 mm. As it is seen from table 1, the beginning and the end of a setting on ASTM C 150 [11], considerably differ from each other.

According to the Canadian CSA CAN 3-A5 [12] standard the determination of the time prior to the beginning of a setting is only provided. The usage of Vicat apparatus is stipulated also in the British standard of BS 12:1978 [13]. For testing cement mortar is used. Penetrometer is applied to determine the time before the beginning and the end of a setting. The force required for penetration of the needle to a depth of 25 mm is determined. The force per unit area, created by a needle and maintained by a paste surface is called penetration resistance. The time prior to the beginning of a setting is the time from the moment of contact of cement with water , which is necessary preparation of the cement mortar, sifted from concrete to the achievement of resistance of a penetration of 3,5 MPa. The time until the end of a setting is the quantity equal to 27,6 MPa.

Researchers in Russia and the Republic of Belarus also use Vicat apparatus in accordance with ГОСТ 56587-2015 and ГОСТ 310.3-76 for determination of terms of a setting respectively [14, 15].

In the patent of the Russian researchers Bulat A. D. and Tsaryov A. M. [16] the method of control and management of setting terms, stages and processes of structuring of mortar and concrete mixtures is offered. The invention can be used in production of concrete and reinforced-concrete workpieces of the increased durability and in construction of monolithic buildings and constructions. The technical result is a possibility of determination of time of the beginning and end of a setting, maintenance of functional inspectation of a condition and formation of stages of hardering of mixtures on the basis of cement.

As an analog for determination of the beginning and the end of a setting the known penetrometry method of Vicat performed according to GOST 310.3-76 according to item 2 is used. The needle of the device of Vicat is immersed in cement paste every 10 minutes. Under the conditions of mixture hardering the quality of control sharply decreases. The beginning of a setting of the cement paste is considered at he time which passes from the beginning of a mixing until the moment when the needle doesn't reach a plate on 2–4 mm. The end of a setting of the cement paste is considered as the time from the beginning of a mixing to the moment when the needle falls to dough no more than by 1–2 mm. At the same time if the beginning of a setting of the cement paste to some extent corresponds to the beginning of the induction period, then the end of a setting doesn't correspond to the end of the induction period, it can't be precisely determined. In case of setting cement paste hardens and it is impossible to lower a needle into paste. Besides, this method isn't suitable for concrete mixture at all, as the penetration of a needle into concrete mixture is interfered, besides, by filler grains. The device has a big instrument error, and thus low accuracy of determination of stages of hardening. If the beginning of a setting of the cement paste can be determined with a low error using this device, then this method isn't suitable for control of the termination of a setting and stages of hardening of mixture.

The closest method according to technical solution as a way of determination of terms of a setting of concrete mixture and control of kinetics of concrete hardening is the method of conductivity change with connection to the power supply [6]. In N. I. Akhverdov's experiments portland cement is used, to which the distilled water was added so that to exclude the distorting influence of the salts dissolved in it. Cement paste of various consistences was placed in dielectric bowls. The results of experiments by means of Vicat apparatus and a method of conductivity allow to assume that they characterize various properties of the cement paste: the first allows to judge indirectly the mechanical durability of a macrostructure of the cement paste, and the second allows to judge the physical and chemical transformations causing formation of its microstructure.

Analyzing the existing techniques of determination of terms of a setting of cement systems, it is possible to draw a conclusion that they have disadvantages and in them there is no unambiguous definition of the term of the beginning and the end of a setting. Also the beginning and the end of a setting are not connected with the electrochemical processes happening in cement system when cement is mixed to water.

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