EFFECT OF MECHANICAL DISTURBANCE ON THE HYDRATION OF CEMENT SUSPENSIONS

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1. Practical problem

In the Laboratory of the Department of Hydraulic Engineering of the Budapest Technical University a test series was carried out in connection with shaft sinking. The problem was as follows:

In the course of shaft sinking often water bearing strata have to be crossed. Pumping may not be economical or possible any more. In such cases formation of a submerged cement plug at the given level of the shaft under construction may stop water flow from below, or at least reduce it to a rate that can be handled by continuous pumping. Aquiferous cracks of the rock are sealed by grouting through this hardened cement plug at the shaft bottom. Its quality — strength and impermeability — is primordial for the desired result, especially when an important water pressure is acting from below on the cement plug.

In general, the cement plug is prepared of a cement suspension conveyed through a vertical pipe either under pressure or by gravity to the pit bottom under water.

In shaft construction practice, variations in the quality of the cement plug have been observed, making determination of the necessary cement plug thickness incertain, leading to overdimensioning. In other cases, missed setting of the cement plug has also been observed, though no harmful waterchemical effects were present.

Therefore investigation into the circumstances of cement plug preparation and setting became necessary.

Model tests showed that quality variations of the cement plug prepared under water were not independent of the motion during grouting. Cement hydration begins already during the motion period; but no setting can occur, because the cement particles are in constant movement. There are two kinds of motion: primarily the technological procedure of grouting involves motion, then after standstill, cement particles in the grout settle, as an internal motion. Both types of motion may be considered as disturbances to setting, while the chemical process of hydration is left unhindered by motion and even intensified.

Beyond the actual technical problem justifying the investigation, similar or analogous phenomena may occur in other domains of practice, disturbing the setting motion.

The most important among them are:

a) In grouting works, grout or mortar conveyed by pipeline. and thereafter during the grouting process, is in movement for quite a long time, causing both hydration and development of texture to rather differ from those at rest.

b) In the "prepacked concrete" process, especially in mass concreting, the mortar is in motion until saturation of the individual sections. To determine both the volume to be grouted at once and the optimum effect of grouting under pressure, the relation between motion time and strength has to be known.

c) It is important as well to know the effect on strength of the mixing car time of ready-mixed concrete.

2. Disturbed setting phenomena

Hydration of cement is known to start immediately in the presence of water and to continue for a certain time. Hydrates of clinker minerals begin to form on the cement particle surfaces just upon contacting water, then the hydration proceeds inwards at a gradually slowing rate. So the hydrates forming on the particle surface are gel-like and are characterized by a large surface. The bond of the particles — i.e. the strength — relies primarily on large surfaces; namely the binding forces are essentially surface forces. A further determinant is the increase of density in a paste at rest, namely the hydration products occupy about twice as much place as the compact cement before hydration.

In concretes, where particles are densely adjacent, with an optimum hydration water there is a strong bond between cement particles, the increasing hydration process is accompanied by increasing concrete strength.

In high water/cement grouts, however, the cement particles are "swimming" in the carrier liquid; the individual particles are quite apart. Also here, the quantity of hydration products is increasing during hydration, but as the cement particles are far from each other, the cement sets as a lowdensity material and not even the whisker structure of the hydrates develops. The hydration bond between the cement particles develops here after a long time and in a rather loose form. This explains also for the well-known phenomena of protracted setting and strength decrease in case of high water/cement ratios. A cement suspension at rest — however high its water/cement ratio — sets sooner or later, because the cement particles settle and form a loose, but confined bulk; in this way hydration bond, even if loose, exists between the particles.

Cement particles kept in motion show, however, a phenomenon of a different tendency. Gel formation on the particle surface is started by hydration upon contact with water in this case too, but agitation of the suspension counteracts the bond between the particles throughout the motion. It depends on the duration and the character of the motion, whether at the time of stillstand, gel formation continues on the particle surface, or it is accomplished. If the hydration can continue, then further bonds between particles may form, but the hydration that has occurred during motion exhibits a minor effect and there is no substantial hardening but as result of the final stage of the process as a rule. For a motion of a given extreme duration, the entire particle bulk may become hydrated without bond between the cement particles. In such cases the end-product is a cement gel-slurry without strength.

During motion of the cement particles, side effects affecting the strength of the end-product may occur.

Motion, e.g. mixing intensifies "wetting" hence hydration — in fact, favourable for strength. Protraction of the process is, however, of negative effect with advanced hydration. Mechanical effect of the motion causes the particles to collide and involves abrading. This abrasion "uses up" primarily the new hydration products. Removal of the gel, formed in the initial stage of the hydration, is known to be advantageous for the strength. A further result of the abrading effect is, however, to decrease the quantity of "hydration products able to bond". The abrasion effect depends on the type and intensity of motion. A motion of such an intensity could be conceived as to continuously grind the cement particles, i.e. to increase the specific surface, resulting in increased strength, provided it is not maintained beyond a stage of rather advanced hydration.

Hence the strength of a cement suspension kept in constant motion depends on the gel texture and position, rather than on the hydration rate. These circumstances do not provide for the gels to penetrate capillaries, therefore progress of the hydration is not accompanied by increasing density. This may be responsible for a low strength.

Among mechanical disturbances of the cement suspension setting, a type of disturbance, due to other than outside influences, namely to sedimentation, a natural motion of the suspension, has to be considered.

The cement grout of high w/c ratio shows sedimentation at rest, i.e. water bleeding. After standstill, the particles of the suspension — homogeneous during mixing — begin to settle. The sedimentation rate of the particles is proportional to the square of the particle size (Stokes theorem).

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During sedimentation the particles are in motion, though the suspension itself is already at rest. Duration of the particles' motion is a function of the rate of sedimentation and the path length. During sedimentation of the particles there is no setting but the hydration process is undisturbed.

During sedimentation along a long path, the particles segregate according to fractions, i.e. the cement particles settle in upwards decreasing size order in the space holding the cement slurry.

Sedimentation as a disturbed setting is a highly differentiated process, where also effects other than the quoted phenomena have to be taken into consideration.

From the Stokes theorem it follows that in a concrete hardened from a settled cement slurry, the particles settle according to fractions and so they harden. The coarser the particle, the longer is the path and the higher is the rate of sedimentation, but the shorter its duration. This is also true inversely, resulting in a prolonged disturbance of setting. This effect absolutely bears on the final strength.

3. Description of tests

The test program consisted of three test series (all involving cement C 500 of Tata).

The first test series cleared up phenomena occurring in the technological process of cementation of the shaft foot. This test series of no general interest aimed at analysing an actual technological process. It was important by having directed the attention to the phenomenon of disturbed setting and by being starting point for detailed tests of a more general character. However, some basic phenomena were already cleared up at this stage, therefore major items of the test series are presented in the following as an introduction.

An original scale model representing a shaft was tested in the arrangement seen in Fig. 1. Different density cement suspensions were fed by gravity into a water tank through an axially connected pipe. Another variable parameter was the distance between the pipe end and the bottom of the tank, resulting in different grades of turbulence of the introduced suspension.

After the introduced cement suspension settled, specimens were taken to determine the 7 days strengths. Compressive strength data were processed in function of the spatial position of the specimen. Fig. 2 shows the spatial position of sampling, Fig. 3 the compressive strength in function of both the spatial position and the density of the suspension. Fig. 4 shows relation of the density of the suspension to the 7 days compressive strength, omitting disturbances.

In the first test series the relation between strength and spatial position of the specimen probabilized the basic relation to be that between the strength



Fig. 1. Scheme and geometry of testing arrangement



Fig. 2. Sampling spots

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Fig. 3. Crushing strength as a function of the sampling position and the suspension density



Fig. 4. Effect of the cement suspension density on the crushing strength of the hardened cement (tube end at 4.0 cm from the bottom of the model)

and the time of setting disturbance by motion, a view supported by observation of both the turbulence for different feeding rates and the time necessary for reaching the standstill.

To enhance this effect, a further test was made by continuously blowing in air during 8 hours through the bottom of the model, to keep the cement suspension in motion. Fig. 5 shows that disturbance by air blowing decreases the strength of specimens taken of the same place in proportion to the disturbance time. Theoretically there exists a disturbance time where the strength is zeroed!



Fig. 5. Effect of time of disturbance by continuous air blowing on crushing strength

The second part of the test series strove after a more exact investigation of the disturbance effect.

It was desired to clear up the trend of phenomena occurring in the course of disturbed hydration in function of the kind, the intensity and the duration of motion, as well as to determine the critical time of disturbance where strength is completely zeroed. 7 days strength was tested.

Types of motion used as disturbance were as follows:

a) Constant air blowing: the grout was kept in constant motion by introducing compressed air. This motion, though of low intensity, caused agitation throughout the suspension. Of the suspension in motion, samples were taken hourly to make 2×3 specimens for the sedimentation tests. Crushing strength was tested on 7-day cubes of 7.06 cm edges. In the following tests, samples were taken and tested in the same way.

b) Air blowing cycles: the grout was brought into motion at determined cycles by compressed air, like under a). Three degrees of disturbance were applied: air was blown in for 2 minutes every 10, 20 or 30 minutes.

c) Continuous mixing: the grout was kept in constant motion by a

motor-driven laboratory mixer. Three test series were made, the mixing velocities being 1800 rpm, 360 rpm, and 60 rpm.

d) Mixing cycles: the suspension was brought into motion in given intervals by the equipment described under c), at a rate of 60 rpm; 3 degrees of disturbance were applied, i.e. mixing for 2 minutes every 10, 20 and 30 minutes.

e) Sedimentation: the cement suspension was poured into test tubes, without disturbance of the hydration, and exposed to disturbances according to a, b, c) and d) in turn. Sedimentation of the suspension was observed for 8 hours.



Fig. 6. Effect of continuous mixing on crushing strength

For all tests cement C 500 was used and the described tests were carried out on suspensions of three different densities, so that the density effect could also be evaluated.

Test results are shown in Figs. 6 through 10, leading to the following conclusions:

Disturbance by mixing not only impedes the bond between the hydrating cement particles by keeping them in motion, but also abrades the forming hydrate film by successive collisions.

Air blowing essentially acts similarly as above, but the particles collide with much less energy so the effect of "grinding" is less pronounced.

Cyclical application of the above motions aimed at eliminating the "grinding" effect, i.e. at establishing the effect of "pure" setting disturbance, free from any side effect.

It may be concluded from the test series that in all four kinds of disturbance the effect on the strength is of the same order and tendency. For a disturbance of about 30 to 40 hours there is no setting, only a hydrated cement slurry develops. Strength loss is proportional to the disturbance time. As it was expected, the more intense disturbance of 1 or 2 hours — conceivable as wet milling — resulted in maximum strength. This effect did not appear for cyclic air blowing involving less mechanical work, and was moderate for cyclic mixing.



Fig. 7. Effect of cyclic mixing on crushing strength

The trend of intermediate strength values clearly reflects the effect of volume and intensity of the applied mechanical work: the more intense is the disturbance, the more marked the decrease in strength (mixing rpm, frequency of disturbance cycles).



Fig. 8. Effect of constant air-blowing on crushing strength

The scope of the *third part of the test series* was to investigate the sedimentation effect.

During the sedimentation process — the cement particles being in motion — hydration without setting occurs, thus, also this phenomenon can be conceived as a disturbed setting.



Fig. 9. Effect of cyclic air-blowing on crushing strength

The quoted experiments proved that a disturbed setting resulted in strength decrease. Moreover, after a certain disturbance time, the cement might completely hydrate without setting; instead of a hardened cement stone, a slurry consisting of hydrated cement particles would result. The



Fig. 10 Effect of disturbance method on crushing strength



Fig. 11. Crushing strength as a function of setting disturbance and sample spot, $\gamma = 1,3$ p/cm³. 1. mixing for 5 min.; 2. mixing at 60 rpm for 4 hours; 3. mixing at 1800 rpm for 4 hours



Fig. 12. Crushing strength as a function of setting disturbance and sampling spot $\gamma = 1.5$ p/cm³. 1. mixing for 5 min; 2. mixing at 60 rpm for 4 hours; 3. mixing at 1800 rpm for 4 hours

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Fig. 13. Crushing strength as a function of setting disturbance and sampling spot, $\gamma = 1.7$ p/cm³. 1. mixing for 5 min; 2. mixing at 60 rpm for 4 hours: 3. mixing at 1800 rpm for 4 hours

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tests also demonstrated intensity and duration of the disturbance to be related to the final strength.

Sedimentation as a disturbance to setting gave similar results. Tests combined the already reported types of motion, applied before sedimentation.

An accessory feature of the mentioned phenomenon — troubling somehow its effect — was the circumstance, well-known from the literature, that increase of the grinding fineness of cement resulted by itself in a higher concrete strength. This meant certainly an opposite effect, as the sedimentation lasted longer for the finer cement particles and so did disturbance of the setting.

Relevant tests were destined to clear up the resultant effect.

The test series consisted in pouring cement slurry of three different densities (1.3; 1.5; 1.7 p/cm^3) into a cleaved plexi tube of 50 mm dia. After a disturbing pretreatment, the sedimentation process in the plexiglass tube 2.50 m high has been recorded.

After the cement slurry has set, the plexiglass tube was opened, the cement concrete column cut into cylindrical specimens 75 mm high and tested for crushing strength at 7 days. Compressive strengths were evaluated in function of the distance from the tube bottom.

Test results are shown in Figs. 11, 12 and 13.

Protracted sedimentation can be stated to be analogous to mechanical disturbance and results in a strength loss proportional to the duration of the process.

Summary

The test series cleared up setting behaviour of cement suspensions upon disturbance by motion.

The chemical-physical process of hydration of a cement suspension in motion was found to continue undisturbed, and even accelerated by a more intensive motion, setting was getting ever weaker. After the motion stopped, the setting begins, provided duration and intensity of the motion are below a critical value. Even so, the strength is reduced in proportion to the duration and the intensity of the motion.

In a suspension kept in motion beyond a critical time -30 to 40 hours - there is no setting at all and a loose cement slurry structure, consisting of hydrated cement particles, develops.

The phenomenon of sedimentation inside the cement suspension in rest involves also disturbed setting. The cement particle motion during sedimentation has the same effect as an externally applied motion.

Test results explain for the backward phenomena occurring in shaft foot cementation (low-strength or unset cement plugs). At the same time, rules concerning grouting, slurry conveyance by pipeline and prepacked concrete emerge, of fundamental importance from quality aspects.

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