THE INFLUENCE OF TEMPERATURE ON THE HYDRATION PROCESS OF CONCRETE EVALUATED THROUGH ULTRASOUND TECHNIQUE

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Abstract

An ultrasonic wave reflection test method was developed to characterize the state of early-age cement-based materials. It analyses the wave reflection from the interface between a well known material and the hardening concrete. Experimental results have shown that there is a distinct correlation between the development of the wave reflection and the development of mechanical properties of concrete. A large number of investigations have been carried out by the authors concerning the setting and hardening of cement-based materials.

The investigation presented deals with the influence of the temperature on the hydration process evaluated through ultrasonic techniques. Concrete specimen were produced and stored under controlled curing conditions at specific temperatures. Beneath the wave reflection method the ultrasonic wave velocity method measured in through transmission were performed. The obtained results were correlated with concrete technological properties of the specimen: setting time, compressive strength and degree of hydration.

It is obvious that the applied experimental technique is able to detect even small effects on the mechanical properties of the concrete due to the temperature during the hardening process. The accelerating influence of the temperature on the hydration process of cement can be detected with ultrasound.

1. Introduction

The progress of the strength in concrete is defined by the increase of its compressive strength with age [9]. It is governed by the development of the hardening process. The final strength is possibly reached only after years. Nevertheless the major part of strength development is finished at the 28th day after mixing. For the strength gain of normal concrete the strength gain of the cement matrix is dominant. Accordingly the development of the concrete strength is influenced in particular by the characteristics of the cement, the water cement ratio and the curing conditions, i.e. by the relative humidity and the temperature of the surrounding. The hydration and hardening can be accelerated by heating the fresh concrete immediately after
compacting. For that purpose the temperature of the concrete can be raised up to 85 °C. This affects not just the compressive strength but also the Young’s modulus and the degree of hydration.

The investigation presented here deals with the influence of the temperature on the hydration process evaluated through ultrasonic techniques. For this purpose two different ultrasonic test setups were applied. The ultrasonic wave velocity was measured in through transmission. The ultrasonic wave reflection method measured and analyzed the reflected part of the incident wave from the interface to the hardening early-age concrete.

The assessment of the mechanical state of the hardening concrete is of great interest. With this knowledge the date of the form removal can be optimized and the efficiency of pre-cast element productions improved. Non-destructive testing methods are useful, for the tests can be performed continuously without affecting the element.

2. Experimental Test Description

The L-wave reflection from the surface of the hardening concrete mixture is measured at different surrounding temperatures. The acquired data are correlated with obtained mechanical and chemical properties: compressive strength and degree of hydration. The tests are performed on concrete specimens comprised of ordinary type I Portland cement (CEM I 32,5R) and normal aggregates („Rheinkies“) with a maximum particle size of 8mm.

<table>
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<th>cement</th>
<th>water</th>
<th>aggregate 0-2mm</th>
<th>aggregate 2-8mm</th>
<th>grading curve</th>
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<td>0.5</td>
<td>2.42</td>
<td>1.83</td>
<td>B</td>
</tr>
</tbody>
</table>

A description of the specimen composition is shown in table 1. The test specimen were cured under isothermal conditions at constant temperatures of 20°C, 35°C, 50°C and 65°C, respectively. In order to eliminated the influence of the relative humidity of the surrounding on the results of the investigation the specimen were kept under conserved conditions throughout the duration of the experiments.

**L-wave velocity**

The L-wave velocity was measured in through transmission in the center of the specimen as shown in figure 1. L-wave transducers with a center frequency of 500 kHz were used.

![Figure 1: A schematic of the through transmission apparatus](image)
Wave reflection measurements
A longitudinal wave can propagate in a solid. Its velocity is given by

\[ V_L = \sqrt{\frac{E \cdot (1 - \nu)}{\rho \cdot (1 + \nu) \cdot (1 - 2\nu)}} \]  

(1)

where \( E \) is the Young’s modulus, \( \rho \) the mass density of the material and \( \nu \) the Poisson’s ratio.

At an interface between two materials with differing acoustic impedances one part an incident wave energy is transmitted through the boundary into the second material and the remaining part is reflected back into the first one. When the propagating wave is normally-incident upon the boundary the ratio of the amplitude of the reflected wave to the incident amplitude is given by

\[ R = \frac{\rho_2 V_2 - \rho_1 V_1}{\rho_2 V_2 + \rho_1 V_1} \]  

(2)

where \( R \) is the reflected wave, \( \rho_1 \) the density of the first material, \( V_1 \) the velocity of the first material, \( \rho_2 \) the density of the second material and \( V_2 \) the velocity of the second material.

In order to eliminate the influence of the measuring device and the surrounding like the coupling of the transducer the measurements at the hardening concrete are normalized on that at air and we obtain

\[ WRF = \frac{R_{\text{CONCRETE}}}{R_{\text{AIR}}} \]  

(3)

where \( R_{\text{CONCRETE}} \) is the wave reflection at the interface acrylic glass – hardening concrete and \( R_{\text{AIR}} \) the wave reflection at the interface acrylic glass – air. For this purpose a wave reflection measurement was performed on the empty mold before filling the concrete in. Successively the measurements at the concrete were started and the WRF calculated automatically. The test setup for the WRF measurements is shown in figure 2.

Typical WRF graphs can be seen in figure 3. At first the WRF curve decreases, reaches a minimum, increases again and levels off at a certain value. After mixing the acoustic impedance of the concrete is lower than that of acrylic glass. It increases with the hydration process of the cement. At the minimum of the WRF curve the acoustic impedance of the concrete is equal to that of the acrylic glass. Afterwards the acoustic impedance of the concrete exceeds that of the acrylic glass.

L-wave transducers with a center frequency of 200 kHz were used. They were coupled on the acrylic glass plate with a thin layer of VASELINE®. The mold was glued on the acrylic glass plate. The test setup was stored in the oven at the specific temperature for several hours before the concrete was filled in. After filling in the concrete and compacting it a glass plate was glued on top of the mold in order to achieve a conserved condition. The interval for the data collection was set to 20 minutes.
Dynamic Young’s modulus
The dynamic Young’s modulus in the center of the specimen was calculated from the L-wave velocity measurements. It is given by

\[ E_{dyn, VL} = \frac{V_{L,C}^2 \cdot \rho_C \cdot (1 + \nu) \cdot (1 - 2\nu)}{1 - \nu} \]  

where \( V_{L,C} \) is the L-wave velocity inside the hardening concrete, \( \rho_C \) is the mass density of the hardening concrete and \( \nu \) is its Poisson’s ratio. For the calculations \( \rho_C = 2.24 \text{ kg/dm}^3 \) and \( \nu = 0.2 \) were used.

Pin Penetration resistance
The mechanical stiffening was measured according to DIN EN 480-2. The pin penetration resistance test determines the time at which initial and final set occurs.

Compressive strength
For the compressive strength test according to DIN 1048 specimen with the size of 150 mm x 150 mm x 150 mm were produced. At each test series the tests were started after the final set. Immediately after the specimen were taken out of the oven they were cast out and the tests were performed.

Degree of hydration
Cement and water react throughout the hydration process to CSH-phases. At the same time pores of different sizes are built up. The water fractions can be divided in two parts:

1) the chemically combined water and
2) the free water.

It is assumed that those parts of water inside the specimen which are not evaporated by heating the sample up to 105°C are chemically combined. They can be driven out of the sample by
increasing the temperature up to 950°C. The amount of chemically combined water is an
indicator for the degree of hydration of the specimen at a certain time [5].
At the investigation presented the degree of hydration of the specimen was measured by the
CWA analysis. It determines the CO2 – and H2O – content as percentage of the investigated
powdery sample.
As an acceptable approach the degree of hydration $\alpha$ can be described as the ratio of the
amount of chemically combined water at a certain time $t$ to that amount of water which can be
combined chemically at complete hydration. It is given as

$$\alpha_t = \frac{w_{n,t}}{w_{n,max}}$$

where $\alpha_t$ is the degree of hydration at a certain time $t$, $w_{n,t}$ the fraction of non-evaporable water inside the cement at a certain time $t$ and $w_{n,max}$ the fraction of non-evaporable water inside the cement which is combined
chemically at complete hydration. Both values $w_{n,t}$ and $w_{n,max}$ are given as percentage
by weight.

3. Experimental Results

Wave reflection factor (WRF)
The development of the WRF at all four temperatures is shown in figure 3. The higher the
surrounding temperature is, the faster does the concrete mix reach the acoustic impedance of
the acrylic glass correlating with the minimum of the WRF curve. In addition the WRF curve
increases faster and reaches higher maximum value.

![Figure 3: Development of the Wave Reflection Factor (WRF) at different surrounding
temperatures](image)

The initial set (red circle) and the final set (blue circle) occur earlier with increasing
surrounding temperature. The correlation of the WRF and the pin penetration resistance is
investigated and developed in [12]. The beginning of the acceleration period accompanied by the built up of CH and C-S-H phases starts earlier. Plotting the initial and final set of the concrete at the specific surrounding temperatures it can be seen that the greatest accelerating effect of the temperature on the setting of cement is from 20°C to 35°C. (Fig. 4)

![Figure 4: Initial and final set of concrete at different surrounding temperatures](image)

**Dynamic Young’s modulus**
The development of the dynamic Young’s modulus inside the specimen is shown in figure 5.

![Figure 5: Development of the dynamic Young’s modulus at different surrounding temperatures](image)

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Temperature has an accelerating influence on the hydration process of the cement. The gain of dynamic Young’s modulus occurs faster. However after about 4 days the specimen have reached the same value no matter which temperature they were exposed to. Subsequently the ranking turns reverse. The statement in the literature [9] saying that 28 days after mixing the specimen cured at higher temperatures have lower Young’s modulus was confirmed in our tests.

Figure 6: Correlation of the WRF and the dynamic Young’s modulus

A linear correlation of the dynamic Young’s modulus and the WRF is detectable for all investigated temperatures. (Fig.6) With increasing temperature the dynamic Young’s modulus inside the specimen is lower for the same WRF. The difference of the acoustic impedance of the surface of the hardening concrete to its volume increases with the surrounding temperature.

Compressive strength

With increasing surrounding temperature the gain of the compressive strength is accelerated in the first days. (Fig. 7) However later on the specimen exposed to lower temperature reach higher compressive strength. According to the height heating of the concrete leads to distinct enduring internal damages which cause the decline of the compressive strength. The obtained results correlate with the conclusions in the literature [9].
The relationship between the WRF and the compressive strength for different temperatures is shown in figure 8.

The slope of the curve decreases with increasing temperature. The same compressive strength is achieved at higher WRF values with increasing surrounding temperature. An exponential relationship between the WRF and the compressive strength can be approximated.
Plotting the WRF – compressive strength relationship for all investigated temperatures at different times after mixing a linear trend can be seen at any graph. (Fig. 9)

Figure 9: Relationship between WRF and compressive strength for different surrounding temperatures at different times after mixing

**Degree of Hydration**

![Figure 10: Development of the degree of hydration at different surrounding temperatures](image)

The hydration process is due to the reaction of cement particles with water. During the hydration process the hydration products of the origin cement particle grow inwards as well as outwards. Their growth into the interspaces between the single particles evokes an interlocking of the hydration products and leads subsequently to an increase of the degree of hydration and
strength gain. Temperature has a significant influence on the speed of the chemical combination of water. The development of the degree of hydration inside the specimen for different surrounding temperatures is shown in figure 10.

The increase of the degree of hydration of the cement occurs faster. The temperature accelerates the chemical combination of water. The relationship between the WRF normalized at the maximum value at which it levels off at each temperature and the degree of hydration is shown in figure 11. With increasing surrounding temperature a higher degree of hydration is reached for the same normalized WRF value. This indicates also a peripheral zone effect as was detected above.

![Figure 11: Relationship between the normalized WRF and the degree of hydration at different surrounding temperatures](image)

These results demonstrate that the temperature of the surrounding has a significant influence on the ultrasonic measurements of concrete properties. At the interpretation of the collected data these results have to be paid attention to.

4. Conclusion

Based on the presented research the following conclusions can be drawn:

1) With increasing temperature the WRF curve is compressed.
2) The greatest accelerating effect of temperature on the setting of cement is from 20°C to 35°C. Further increase of temperature shows no significant acceleration.
3) The WRF and the dynamic Young’s modulus are linearly correlated for the investigated temperatures.
4) The difference of the acoustic impedance of the surface of the hardening concrete to its volume increases with the surrounding temperature.
5) The WRF and the compressive strength are approximately exponentially related.
6) At any time after mixing the WRF – compressive strength – relationship is linearly related with increasing temperature.
7) With increasing surrounding temperature the degree of hydration is higher for the same normalized WRF value.
References