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The Effect of Elevated Temperature on Ordinary Portland Cements

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Abstract

A study was directed to analyze the influence of the different types of Ordinary Portland Cements (OPC) on the hardened cement paste exposed to high temperatures. Hardened cement paste specimens with three types of cement and nine water-cement ratios were investigated and heated to 11 heat steps at 28 days of age. Residual compressive strength and relative residual compressive strength were compared to the unheated results. It was shown that the expected range for the relative residual strength of the different types of OPC up to 400 °C is between 90 and 100%. From the local minimum at 150 °C to the maximum at 300 °C. The CEM I 52.5 type of cement shows the least favorable behavior to the increased (over 400 °C) temperature, according to the results of the residual compressive strengths curves and the newly introduced Integrated Temperature Endurance values after the first water-cement ratio (0.3). Nine water-cement ratios were tested with the favorable types of OPC (CEM I 42.5 and CEM I 32.5). At all water-cement ratios, the residual strength tendencies for the two types of cement were parallel. It has been observed that changes in the water-cement ratio have little to no impact on the relative strength curve's shape or the relationship between strength and the water cement ratio. As a result of our experiments applying simply Ordinary Portland Cement, it is obvious that the CEM I 32.5 type of OPC cement significantly outperformed the CEM I 42.5 type in regards of post-heating strength tendencies.

Keywords

cement, Ordinary Portland Cement, residual properties, compressive strength, integrated temperature endurance

1 Introduction

The world's most popular building material is concrete. It is the second most used natural resource on Earth after water as a composite material made of cement and aggregates [1]. Therefore, there has been a century-long interest in comprehending the behavior of this complex composite material. In addition to design and macrostructural information, it is crucial to understand how a material's microstructure responds to various environmental factors [2].

The heat of a fire is one of the most harmful situations that a concrete construction could encounter [3]. Since the 1940s, researchers have been examining how high temperatures affect concrete's mechanical properties [4, 5]. The internal composition and content of hardened cement paste have a significant impact on the characteristics of concrete exposed to high temperatures [3, 6–8]. To select the proper material and dose for diverse applications, a number of research programs have been conducted in recent decades to examine the generally used supplementary cementitious materials [9–14]. Early research on concretes made of Portland Cement or Portland Cement with little to no cementitious materials revealed about the same residual compressive strength over 400 °C [15]. The studies showed that the mixing ratios of the ingredients had an impact on the qualities of cement paste as well [3, 7, 8, 12, 13, 16–18].

According to Grainger's summary [19], the behavior at high temperatures is influenced by factors such as the ratio of water to cement, the quantity of calcium-silicate hydrates, the quantity of $Ca(OH)_2$, and the degree of hydration. Wang further emphasized that various cement pastes may behave differently in a fire [18].

To ascertain the effect of the oxide composition of Ordinary Portland Cements, Lublóy [7] conducted a study. The test variables included cement fineness (tree types), heat stages (five steps up to 800 °C), and oxide composition (four varieties). Research has demonstrated that the aluminate modulus and fineness of cement have a substantial impact on the material's fire resistance. Wang [18] tested Ordinary Portland Cement with three different water-cement ratios (0.23, 0.47, 0.71) with seven heat steps. The residual compressive strength results of pure Ordinary Portland Cements show 48%, 66%, and 62% of the initial compressive strength at 200 °C; and 23%, 11%, and 0% at 580 °C, respectively. However, the author's focus was to compare the pure Ordinary Portland Cement to mixtures that contained ground granulated blast furnace slag. The residual cement results from his studies were often better when blended types of cement were used.

The positive effect of the blending materials has been detected by several researchers. Abed and Lubloy [20] and Dias et al. [21] pointed out the positive influence of pulverized fly ash on the Portland cement matrix. Mohabbi Yodollahi and Dener [22] demonstrated that using an appropriate type of cement, residual compressive strength increase can be reached after exposure to elevated temperature.

Sabri et al. [23] investigated the impact of fly ash on hardened concrete that had spent two hours at 400 °C. The high amount of fly ash could enhance the qualities of concrete exposed to this moderately high temperature, according to both the mechanical and microstructural results. The most advantageous dosage of fly ash, based on their findings, was 30 m%, whereas the relative strength loss at 28 days of age was just 20%.

A study on metakaolin, silica fume, and fly ash-containing high strength hardened cement pastes was conducted by Abdelmelek and Lublóy [12]. The results revealed outstanding residual strength at the highest temperature, and the dosages tested varied between 615 m%. This study also showed that cement fineness affects the optimal dosage of cementitious components.

In a series of tests, heterogenous cements, including pozzolana and fly ash, were used, according to Lublóy et al. [13]. On the 28th day, an electric furnace was used to test the fire resistance up to 800 °C using a total of five types of different blended cements with the same water-to-cement ratio. The outcome indicated that the thermal stability of the cement matrix is marginally improved by flay ash and pozzolana.

Maheswaran et al. [24] looked at how ternary-blended cement pastes and mortar characteristics were affected by high temperatures. As research variables, they employed nanosilica (1.53%), silica fume (36%), and lime sludge (020%). The results demonstrate that adding lime sludge and silica fume cement composites increases compressive strength significantly, up to 26%, compared to control

mortars at ambient temperature and after two hours of exposure to 500 °C. However, spalling of mortars occurs at 800 °C after 28 days of water curing. At all temperatures, the residual compressive strength of lime sludge and nanosilica cement composites decreases noticeably, although spalling does not occur.

Glass powder's impact on the characteristics of cement pastes that have hardened is tested by Barkauskas et al. [25]. With constant types of cement and constant ratios of water to fines, seven compositions of cement paste mixes were used, each containing varying percentages of crushed glass (030%). Tests on density, ultrasonic pulse velocity, Xray analysis, and compressive strength demonstrated that the mixture containing 5% crushed glass is stronger and denser.

El-Gamal et al. [26] showed that ceramic waste could be used as cementitious material when coupled with a small amount of carbon nanotubes. The experiment showed that the best option available for regular Portland cement is 10% ceramic waste and 0.05% carbon nanotubes. The samples were heated for three hours to a maximum of 800 °C.

To determine the impact of the aerogel addition on the performance of cement-based mortar at high temperatures, Stefanisou and Pachta [27] carried out a research. Aerogel (only 0.5 m%) was utilized in conjunction with a mixture of CEM I 42.5 type cement and siliceous sand in addition to 20 m% of perlite. It was possible to observe the physical and mechanical characteristics of specimens after exposure to the heat of 5 heat steps, even 1000 °C. The findings demonstrated that while cementitious materials somewhat decrease capabilities at normal temperatures, they enhance volume stability and mechanical properties at 800 °C and higher.

At ambient temperature, Vaiciukynienė et al. [28] investigated the effects of perlite (pure, zeolitized, and calcium altered) on the characteristics of blended cement. The outcome demonstrated that the cement matrix's compressive strength might be enhanced by the hydraulic characteristics of perlite.

Despite the benefits of blending materials, Ordinary Portland Cement remains one of the most commonly used building materials at the moment [29]. It is crucial to comprehend how high temperatures affect the matrixes of Ordinary Portland Cement, particularly the residual compressive strength. We didn't have the opportunity to compare additional mixed materials until after these fundamental tests, looking at how they affect the cement matrix favorably or unfavorably. It is vital to recognize the physical and chemical changes in the concrete in order to successfully mitigate the effects of the heat on the structural elements. Increasing the temperature, water discharges; first, the ettringite and monosulphate dehydrate between 50 °C and 110 °C [3, 17, 21], followed by the Ca(OH)₂, the CaCO₃ and the substituent of the concrete dehydrate (i.e., CSH) [3, 11, 14, 17, 30] from the pure Ordinary Portland Cement pastes matrix as shown in Fig. 1.

The studies on the effects of various Ordinary Portland Cement products on the residual characteristics of hardened cement pastes are presented in this paper. Three distinct types of Ordinary Portland Cement were examined during the research. The types of Ordinary Portland Cements [31] used in the tests and the water-to-cement ratios were the test variables. The residual compressive strength was the primary attribute that was examined.

2 Tests on hardened cement pastes

To investigate the effect of hardened cement of different types (strength classes, composition, specific surface) of Ordinary Portland Cement series of tests were carried out. Our research sought to identify the physical and mechanical changes brought on by thermal effects in hardened cement pastes.

2.1 Experimental recipes

A constant water/cement ratio of 0.3 was used to compare the three primary cement kinds (CEM I 52.5 N, CEM I 42.5 R, and CEM I 32.5 R(S)). Table 1 displays the composition and other main characteristics of the cements that were used.



Fig. 1 Chemical changes in the cement matrix

Following that, as shown in Table 2, CEM I 42.5 R and CEM I 32.5 RS were evaluated using nine different water/ cement ratios. To adjust the consistency, a superplasticizer was applied in each case.

2.2 Tests

Cube specimens of 30 mm edge length were made:

- 1. Casting the specimens with the standards molds.
- 2. The specimens were demolded and emerged in water 24 hours after casting.
- 3. Water curing last from the 2^{nd} to the 7^{th} day.
- 4. After the seventh day, the samples were removed from the water and kept in a laboratory air environment (20 °C and 45 %RH) until the day of the test. This type of storage complies with Hungarian standards. Moreover, additional drying of the specimens before heating was not necessary due to the air in the laboratory's drying condition [3, 12, 14, 32].
- 5. All the tests started on the 28^{th} day, and it took 2–4 days.

The specimens' mass and size were measured before the heat test. The effect of the heat shock was then modeled by placing them in an electrical furnace that had been reheated. In each case, the test lasted 120 minutes. The specimens'

Table 1 Main properties of the used Ordinary Portland Cements

Properties		CEM I 32.5 RS	CEM I 42.5 R	CEM I 52.5 N
Density	g/mℓ	3.23	3.06	3.17
Specific surface	m²/kg	304.0	353.0	452.0
Compressive strength, according to the Standard, 28 days	MPa	43.7	54.5	63.2
	C ₃ S	70.7	65.5	67.7
Clinker composition	$\beta C_2 S$	5.0	8.7	9.1
(m/m%)	C_3A	0.0	7.0	12.5
	C_4AF	18.7	9.6	0.9
Free CaO	m/m%	0.37	0.52	0.51
The heat of hydration, 28 days	J/g	384.0	406.8	447.0

 Table 2 Water/cement ratios of Portland cement pastes (CEM I 52.5 N;

 CEM I 42.5 R and CEM I 32.5 RS)

water/ cement ratio	0.120		0.375	
	0.160	0.200	0.462	
	0.195	0.300	0.545	
	0.240		0.750	
tested cement types	CEM 1 42 5	CEM I 52.5	CEM L 42.5	
	CEM I 42.5	CEM I 42.5	CEM 1 42.5	
	CENI I 52.5	CEM I 32.5	CEM 1 52.5	

mass and size were once more measured after they had cooled, and the compressive strength was then evaluated. The results were compared to the preheating parameters. 60 (in some cases 120) specimens (Fig. 2) were used for each series. 10 (20) specimens were stored in laboratory conditions. A set of 5 (10) pieces were heated to 10 different temperatures (50, 100, 150, 200, 300, 400, 500, 600, 750, and 900 °C) to determine the behavior both in the lower and upper heat ranges.

In order to evaluate the effect of the cement properties of the hardened cement paste exposed to high temperature, temperature vs. strength curves, temperature vs. relative strength curves, water/cement ratio vs. strength curves, and the *ITE*, i.e., Integrated Temperature Endurance, the definite integral of the relative strength curves (i.e., area below them), was inducted [9, 12, 14].

3 Results

3.1 Comparison of the three major cement types with constant water/cement ratio

The primary objective was to compare the three main types of Ordinary Portland Cement. Three series were created using an increased number of specimens (120 pieces each) and a constant water/cement ratio (0.3).



Fig. 2 One series of specimens

The series' residual compressive strength is represented in Fig. 3. Because of its constant strength up to 600 °C, it is clear from this first diagram that the CEM I 32.5 RS cement will behave the best. Notwithstanding the fact that the other cement types have higher initial strengths, the earlier starting of the radical strength-decrement makes these cement types' properties less favorable.

It is practical to introduce the relative values of the residual strength results for a better comparison of the habits of the curves. Each result was based on its own series' values under laboratory conditions. The initial strength discrepancies vanished after this phase, and it was now possible to compare the curves more sophisticatedly. The output of this technique is displayed in Fig. 4.



Fig. 3 Residual compressive strength, 3 types of cement, w/c = 0.3



Fig. 4 Relative residual compressive strength, three types of cement, w/c = 0.3

It is evident that CEM I 52.5 types of Ordinary Portland cement have some advantageous characteristics at low heat ranges. However, the results dropped off more quickly than the other curves after reaching their maximum at 300 °C. At 500 °C, the relative residual strength was about the same as it was at the highest temperature. These tendencies made the CEM I 52.5 types of Ordinary Portland Cement inapplicable for further research.

The behavior of the CEM I 32.5 and CEM I 42.5 types of Ordinary Portland Cements was more comparable, although the CEM I 32.5 presents a considerably higher residual strength characteristic up to 600 °C.

Regardless of the aforementioned differences, every curve demonstrates that the residual strength from the specimens heated to 50 °C is greater than the strength of the reference value. It was followed by a drop in temperature between 100 and 150 °C. Each series experienced a second significant strength increase between 200 and 400 °C, achieving a maximum level of 120 to 130% at 300 °C. The post-hydration action of the discharged crystallin waters is what caused this increase. Ca(OH)₂ dehydrates at temperatures above 400 °C, which results in a notable loss of strength. At the highest temperature step (900 °C), each series of pastes has approximately the same results, even if the cement type is important for the behavior of the hardened cement paste's result in the range of 400800 °C (ca. 20%).

The Integrated Temperature Endurance (ITE) number, which is the integrated area below the residual compressive strength curves, provides another way to evaluate the data obtained from the compressive strength measurements [9, 12, 33]. The chained trapezoidal rule method was used to calculate the definite integrals. The Integrated Temperature Endurance unit is $[\% \times ^{\circ}C]$ due to the diagram's axis unit of measurements. A 2D curve could be reduced to a single integer using this type of definite integral, and the conclusions drawn using the same procedure can be contrasted. The calculation of these definite integrals from the whole curve (i.e., 20–900 °C) or from a particular range of results (e.g., 150–300 °C) is obvious.

Both the total Integrated Temperature Endurance of the curves and the specific 300900 °C findings are displayed in Fig. 5. The calculated area technique balanced the positive and negative compressive strength alternations. Because the CEM I 52.5 Ordinary Portland Cement has higher relative residual compressive strength values in the low-temperature range, their severe losses in the mid and high temperature regions don't create as much of a reduction in the Integrated Temperature Endurance.



Fig. 5 Integrated Temperature Endurances, 3 types of cement, w/c = 0.3

The high heat range (300900 °C) ITE measurements highlight the significant differences between the CEM I 42.5 and CEM I 52.5 types.

3.2 Comparison of the strength of hardened cement pastes specimens with different watercement ratios As was mentioned in the preceding paragraph, the behavior of CEM I 42.5 and CEM I 32.5 Ordinary Portland Cements is essentially the same for constant watercement ratios.

The next phase of our experimental program was to ascertain whether the watercement ratio could have an impact on the residual behavior. The CEM I 52.5 was excluded from this section of the investigation due to the aforementioned unfavorable characteristics.

The rule of the relationship between strength and water/ cement ratio [34], without reference to the actual temperature level, has been shown to be unchangeable after analysis of our experimental results (Figs. 6–9), notably below 500 °C. Because the curves generally affine to the curve of the unheated specimen, the residual strength decreases at 500 °C.

The variety of findings makes it possible to establish links between the water/cement ratio and residual strength at all heat stages. Figs. 10–11 the different iterations of the well-known curves [35, 36].

In terms of all water/cement ratios, both overall values and the specific (300–900 °C) component, CEM I 32.5 cement is equal to or better than CEM I 42.5, as shown in Fig. 12.

4 Conclusions

In this paper, our experimental results of residual compressive strength and the Integrated Temperature Endurance of hardened cement pastes consisting of different kinds of



Fig. 6 Residual compressive strength, CEM I 32.5, different water cement ratios



Fig. 7 Relative residual compressive strength, CEM I 32.5, different watercement ratios



Fig. 8 Residual compressive strength, CEM I 42.5, different watercement ratios



Fig. 9 Relative residual compressive strength, CEM I 42.5, different watercement ratios



Fig. 10 Residual compressive strength in the function of watercement ratio, CEM I 32.5, different heat steps



Fig. 11 Residual compressive strength in the function of watercement ratio, CEM I 42.5, different heat steps

Portland types of cement were summarized. The results of almost 1300 specimens were evaluated in this research.

The main objective of the current research was to examine the fire resistance of the common pure types of Ordinary Portland Cement, such as CEM I 32.5 RS, CEM I 42.5 R, and CEM I 52.5 N. Nine heat steps and one water-cement ratio were applied to all three types of cement for the preliminary comparison.

Hereafter, the two better types of cement were further tested with an additional eight (altogether nine) water-cement ratios and the nine heat steps. All specimens were cubes with 30 mm edge length, and the tests started on 28th day.

The following finding can be drawn from our research:

- The relative residual strength of the different types of Ordinary Portland Cement (CEM I 52.5 R, CEM I 42.5 R, and CEM I 32.5 RS) has been estimated to be between 90 and 100% up to 400 °C.
- 2. Due to the crystallin water's post-hydrating effect, there is a significant strength increase from the local minimum at 150 °C to the maximum at 300 °C.
- 3. CEM I 52.5 type of Ordinary Portland Cement is rarely advised for use in fire resistant concrete alone due to the rapid strength loss at higher temperature ranges. Consequently, this type of cement was removed from the following researches.
- 4. Additionally, it has been found that neither the shape of the relative strength curve nor the relationship between strength and the watercement ratio are appreciably affected by changes in the watercement ratio.

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Fig. 12 Integrated Temperature Endurance in the function of watercement ratio, different heat ranges

5. The CEM I 32.5 type Ordinary Portland Cement shows comparatively favorable results in all heat ranges if evaluating the Integrated Temperature Endurance's data for both the full range and the specific heat ranges.

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