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# OPTIMUM HEAT TREATMENT CYCLE FOR CEMENTS OF DIFFERENT TYPE AND COMPOSITION

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#### ABSTRACT

Strength development of concrete is slow with respect to the impressive improvements associated with rapid and serial production of concrete in construction technology in recent years. In this respect, the necessity of having concrete reach a desired strength level by accelerating its hardening process in as short a time as possible is inevitable. Various methods have been developed and applied to accomplish this purpose. Heat treatment has an extensive application amongst these procedures. Cement as a binder has a great importance in heat treatment application due to its function in providing concrete with hardening and gaining strength. The primary factors determining the behaviour of cements subjected to heat treatment are fineness and composition of Portland cements, the type and amount of additive used in blended cements, and cycle parameters. The technical and economical success of heat treatment application depends on the objective and application of a suitable heat treatment cycle. In this context, heat treatment parameters for five cements of different type and composition, which are commonly produced in most countries, were determined. It is concluded that a treatment temperature of 80°C and a 4-hour initial curing before heat treatment is essential as well as reasonable for optimum heat treatment application to the cements investigated. © 1998 Elsevier Science Ltd

#### Introduction

Hardening process and strength gaining rate of concrete under accustomed conditions are slow with respect to the production rate of concrete plants in response to demand. In this respect, it is a natural aim to provide a desired strength level for concrete in a short time by accelerating its hardening process using various methods. Heat treatment is amongst the methods widely used for this purpose (1–3).

Heat treatment application is based on the principle of acceleration of the hydration reactions. With regard to this phenomenon, cement as a binder has great importance in heat treatment application due to its function in providing concrete with hardening and gaining strength. It has been stated that the primary factors determining the behaviour of cements

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under heat treatment application are fineness and composition of Portland cements, and type and amount of additive used in blended cements, and cycle parameters (4). As the evolution of hydration reactions at room temperature and the reaction products formed are dependent on these factors, the evolution under heat treatment application will first of all be dependent on the same factors. The effect of those factors on heat treatment application generally is known; however, there is an important difference of opinion associated with their optimum values. Both chemical and physical damage as the result of heat treatment may occur naturally in the material (5). It has been stated that heat treatment has a profound effect on the structure and properties of hardened cement paste, as crystal morphology and the amount of ettringite phase formed seem to be strongly dependent on temperature (6). Cases of expansion and crack formation in concrete that had been cured at temperatures over 70°C due to delayed formation of ettringite in the hydrated cement paste have been reported (7). Heat treatment also influences the pore structure of cement paste by increasing the proportion of large pores in the cement paste, and it promotes particle growth. This apparently reduces the modulus of elasticity (8). As a result, the ultimate strength of the heat-treated concrete elements will be lower than the standard cured specimens. Considering that these adverse effects occur as a result of heat treatment, it would be a more appropriate approach to determine a suitable heat treatment cycle for the cements considered.

# **Experimental Study**

## **Objective and Scope**

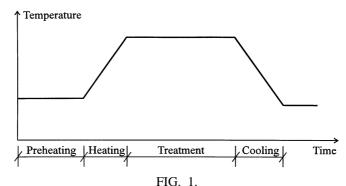
The technical and economical success of heat treatment application to accelerate the strength of concrete will first of all depend on the objective and application of a proper heat treatment cycle to cement. The objective of the investigation is to determine a suitable heat treatment cycle for cements of different type and composition, which are commonly used in most countries.

Practically, heat treatment usually is applied to concrete, reinforced concrete, or prestressed construction elements. However, as far as concrete is concerned, new parameters, such as type of aggregate, water-to-cement ratio, and additives, which have a large influence on heat treatment application, have arisen; therefore, the primary effect of cement cannot be elucidated. In this study, a suitable heat treatment cycle for cements being investigated is determined by evaluating the compressive strengths obtained from standard mortar specimens.

## **Experimental Program**

The heat treatment cycle applied is illustrated schematically in Figure 1. The treatment temperature and its duration, together with preheating duration, were ascertained, as these factors have a profound effect on the evolution of hydration reactions and the products formed. In this study, heat treatment cycles with a total duration of 22 hours are applied so as to minimize the degree of structural disintegration of the mortar specimens. The heating and cooling rates of 15°C per hour and the postcuring duration of 2 h were kept constant throughout the investigation.

The treatment temperatures were chosen to be 50°, 65°, 80°, and 95°C in 15°C increments.



Typical heat treatment cycle.

The preheating and treatment durations were changed in 2-h increments, and 0-, 2-, 4-, and 6-h preheating durations were tested for each cement. All treatment temperatures were tested for 4-h preheating duration, and the temperature at which the 1-day compressive strength was maximum was determined to be 80°C. Heat treatment cycles were carried out with 0-, 2-, and 6-h preheating durations at that temperature, as listed in Table 1.

# Materials, Production, and Experimental Conditions

The cements studied, their physical properties, and oxide analysis, together with the potential compounds of Portland cements obtained using Bogue equations, are listed in Table 2.

The investigation was carried out in a room with an average temperature of  $20^{\circ} \pm 2^{\circ}$ C, where the materials were stored. The standard cured specimens were kept in the same room in a water tank at  $20^{\circ}$ C until the testing age. The specimens to be subjected to heat treatment at the end of the preheating duration appropriate for the associated cycle were immediately placed in a water tank at  $20^{\circ}$ C, and heat treatment was performed. The water temperature of

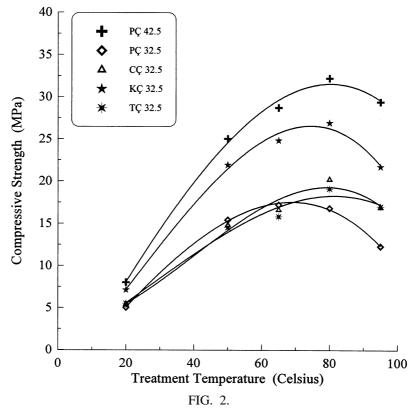
TABLE 1 Heat treatment cycles applied.

Cycle no.	Treatment temperature (°C)	Preheating duration (h)	Heating duration (h)	Treatment duration (h)	Cooling duration (h)
1	50	4	2	14	2
2	65	4	3	12	3
3	80	4	4	10	4
4	95	4	5	8	5
5	80	0	4	14	4
6	80	2	4	12	4
7	80	6	4	8	4

TABLE 2 Physical properties and chemical analysis of cements.

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Cement type	Turkish (EN 197-1)	PÇ 42.5 (CEM I)	PÇ 32.5 (CEM I)	CÇ 32.5 (CEM III)	KÇ 32.5 (CEM II)	TÇ 32.5 (CEM IV)		
Physical properties	Specific gravity (g/cm³) Fineness	3.04	3.09	3.02	3.05	3.05		
	200 μm, retained (%)	0.17	0.22	0.12	0.23	0.26		
	75 μm, retained (%)	2.71	12.71	10.17	9.72	12.18		
	Specific surface, Blaine (cm <sup>2</sup> /g)	3812	2751	2981	3861	3945		
	Water demand (%) Time of setting (Vicat)	29.5	25.0	27.0	26.5	26.5		
	Initial (hour: minute)	2:50	2:20	2:40	3:00	3:15		
	Final (hour: minute)	4:45	3:35	4:20	4:50	5:05		
Mechanical tests	1-day (MPa) 28-day (MPa)	8.0 47.0	5.0 39.8	5.4 34.1	7.1 41.9	5.5 31.7		
Potential compounds (%)	$C_2S$	7.1	47.9	_	_	_		
	$C_3S$	63.2	22.2	_				
	$C_3A$	6.6	14.1					
	$C_4AF$	10.0	7.9					
Chemical composition (%)	Silica (SiO <sub>2</sub> )	19.09	22.54	25.16	19.41	10.08		
	Alumina (Al <sub>2</sub> O <sub>3</sub> )	4.60	6.98	9.41	5.50	7.64		
	Ferric oxide (Fe <sub>2</sub> O <sub>3</sub> )	3.28	2.60	2.24	4.08	4.36		
	Lime (CaO), total	63.19	61.40	52.84	57.30	49.27		
	Magnesia (MgO)	1.79	1.40	4.46	0.77	1.40		
	Sulfur trioxide (SO <sub>3</sub> )	2.79	1.75	1.95	2.88	1.81		
	Loss on ignition	2.67	1.32	0.45	2.26	3.83		
	Insoluble residue	1.01	0.64	0.74	6.90	19.04		
	msoluble residue	1.01	0.04	0.74	0.70	17.04		

the tank was adjusted by a temperature programmer unit appropriate for the desired cycle, and the temperature was controlled continuously using a platinum resistance thermostat. The specimens were taken out of the moulds 2 h after the cooling period, then weighed and tested at the end of 24 h. In the meantime, the moulds were cleaned and prepared for the next



One-day compressive strength with respect to treatment temperature.

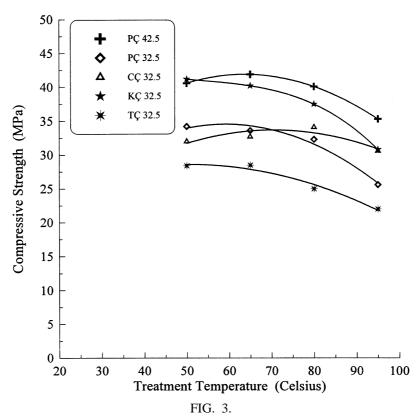
production in 2 h; thus, the program was performed so as to have the moulds used once a day. The specimens to be tested at the end of 28 days were kept in water at 20°C.

According to EN 196-1 (9), three standard mortar specimens measuring  $40 \times 40 \times 160$  mm for each cycle were produced. Following the heat treatment, the specimens were subjected to bend testing at the end of 1 day and 28 days. Compressive testing was performed using  $40 \times 40$ -mm steel plates on the half pieces of specimens obtained. The compressive strength for each age was determined as the average of six measurements.

Two series of mixtures were prepared to determine the compressive strengths at 20°C standard curing condition and to compare them with the results of heat-treated specimens. In each series, 1- and 28-day compressive strengths were obtained from three specimens; thus, for each age, compressive strength was determined as the average of 12 measurements.

## **Results and Discussion**

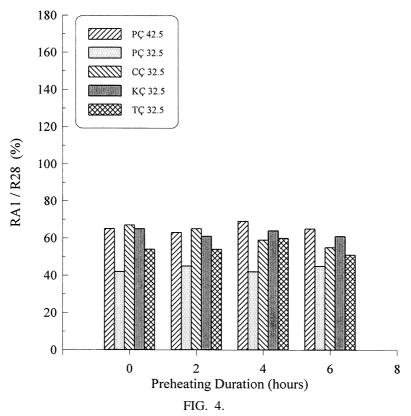
Figure 2 illustrates the variation of 1-day compressive strength of the heat-treated specimens with respect to treatment temperature. The 1-day compressive strengths of the cements that were not subjected to heat treatment are included in the relationship. It can be seen that a



Twenty-eight-day compressive strength with respect to treatment temperature.

65°C treatment temperature is more appropriate for PÇ 32.5 because of the higher 1-day compressive strength obtained. The difference observed for PÇ 32.5 can be attributed to the mineralogical composition of the cement, as the amount of  $C_3A$  is high (14%). Based on the evaluation, it was determined that the optimum treatment temperature is 65°C for PÇ 32.5 and 80°C for the other cements. Heat treatment with higher temperatures does not have a positive effect on strength; on the contrary, the strength decreases at temperatures over 80°C. It also has been observed that KÇ 32.5 exhibited good performance compared to cements other than PÇ 42.5. This behaviour is confirmed by the early higher compressive strength obtained at 20°C. Another reason for this could be the fineness of the cement (a rather high value of 3861 cm²/g). It has been concluded elsewhere (10) that composite cements containing slag, fly ash, or a natural pozzolan perform well under heat treatment, and they do not exhibit a loss in the 28-day compressive strength.

Figure 3 shows the variation of the 28-day compressive strength of the heat-treated specimens with regard to the treatment temperature. This figures shows that the 28-day strength decreases with respect to the treatment temperature, the difference in strength between 50° to 80°C is not discernible, and it increases at 95°C. Considering this propensity, it has been observed that treatment temperatures over 80°C are deleterious because the strength decreases. Therefore, the technical optimum treatment temperature is 80°C. How-

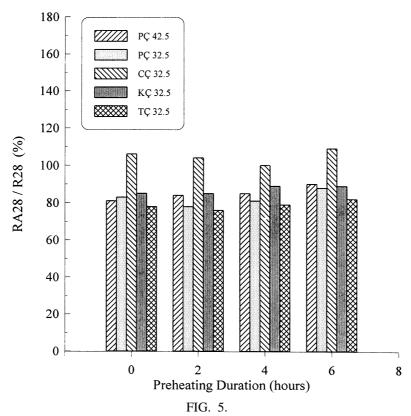


Proportions of RA<sub>1</sub>/R<sub>28</sub> at 80°C with respect to preheating temperature.

ever, if lower early compressive strength is satisfactory, a lower treatment temperature can be chosen for economical considerations. The decrease in long-term compression strength of CÇ 32.5 at 95°C is not so considerable with regard to those obtained from the other cements. One reason for this behaviour can be attributed to the contribution of slag present in the cement to the strength at higher temperatures. The other reason is that the strength gain at normal conditions is more pronounced in the long term compared to that obtained at early ages.

Figure 4 illustrates variation of the proportions of the 1-day compressive strengths of specimens heat-treated at the optimum temperature of 80°C to the standard 28-day compressive strengths with regard to preheating duration. The cements other than PÇ 32.5 at optimum treatment temperature reached 60% to 70% of their 28-day compressive strength level at the end of 1 day, which is about the 7-day compressive strength level requested by the associated standards for the cements under investigation.

A general evaluation associated with Figure 4 is that early compressive strength decreases when a heat treatment cycle with a 6-h preheating duration is applied. Therefore, a preheating duration between 0 and 4 h was found to be optimum, as the decrease in strength is not



Proportions of  $RA_{28}/R_{28}$  at  $80^{\circ}C$  with respect to preheating temperature.

significant between the 0- to 4-h period. To observe this clearly, variation of the long-term strength with regard to preheating duration should be taken into account.

The physical damage that occurred in the heat-treated concrete elements usually manifested as loss in compressive strength at later ages (5). One way to minimize the physical damage is to have construction elements of sufficient strength before heat treatment is applied. This can be achieved by applying a sufficiently long preheating duration, which is one of the parameters determining heat treatment application. On the contrary, a heat treatment cycle with a short preheating duration has an adverse effect on the residual volume increase of the material. This will affect greatly the quality of the construction element at later ages. In this respect, in the investigation, it was necessary as well as useful to have a 4-h preheating duration for practical purposes (11).

Figure 5 indicates the variation of the proportions of the 28-day compressive strengths of specimens heat-treated at the optimum treatment temperature of 80°C to the standard 28-day compressive strengths with respect to the preheating duration. It can be seen that a 6-h preheating duration is useful for long-term strength gain. The difference in strength between 0 and 4 h is insignificant. Although technically there is no numerical difference, a 4-h preheating duration obviously will be more economical. With regard to concrete, however,

it is obvious that a longer preheating duration will be more appropriate to minimize the risk of residual disintegration of the material. With regard to this point, when evaluation of both with respect to 1- and 28-day compressive strengths is performed, the reason heat treatment application with a 4-h preheating duration is preferred becomes clearer.

PÇ 42.5, KÇ 32.5, and TÇ 32.5 all comply with the general interpretation done previously, as a heat treatment cycle with a 4-h preheating duration seems to be suitable. However, considering the standard 1-day compressive strength at  $20^{\circ}$ C, it can be seen that the strength gain of PÇ 32.5 and CÇ 32.5 compared to other cements is rather slow. Regarding this point, it is normal for these cements to prefer a maximum treatment duration (therefore, a minimum preheating duration) in heat treatment application.

As can be seen in Figure 5, there is no strength loss for CÇ 32.5 subjected to heat treatment application with a treatment temperature of 80°C and a 4-h preheating duration. The strength loss is about 15% for PÇ 42.5, which is close to the 20% for PÇ 32.5 and TÇ 32.5; it is about 10% for KÇ 32.5. Based on the evaluation given previously, the most important point that can be drawn is that heat treatment does not have an adverse effect on long-term strengths of cements containing slag. Moreover, there even seems to be a strength gain for heat-treated cements with respect to the standard strength. Similar observations have been reported in the literature (12).

The temperature at which the early strength gain is the highest is 80°C. However, this does not necessarily mean that a heat treatment application at a temperature of 80°C should be applied to the all cements. The treatment temperature can be adjusted according to the targeted 1-day strength level. It is obvious that heat treatment application at a lower treatment temperature is more economical and energy saving. In choosing treatment temperature, technical priorities and economical priorities should be taken into account, and it is not necessary to choose higher treatment temperatures than necessary for heat treatment application.

### **Conclusions**

The cements considered in this study are all responded well to heat treatment application according to the strength level reached at the end of 1 day. The strength level that cements reached at 24 h is about 70% of their standard 28-day compressive strength. This is adequate for prestressed concrete elements as well as for prefabrication purposes.

Except for PÇ 32.5, the heat treatment temperature at which the early strength becomes maximum is 80°C. Treatment temperature of about 65°C seems to be more suitable for PÇ 32.5. Higher temperatures are harmful because of adverse effects on both early and long-term strengths. If the targeted strength level is found to be sufficient, lower temperatures in the range of 50° to 70°C can be used for economical reasons.

The necessity of having a preheating duration useful in practice to minimize physical damage that might occur in the material because of the adverse effect of heat treatment, especially with respect to long-term strength, is obvious. Based on the findings obtained, it is thought that a preheating duration of about 4 h is appropriate. This duration may be shorter for CÇ 32.5, which gains strength slowly. It is proper to determine an optimum preheating duration considering the actual production conditions.

It is useful for heat treatment duration be limited to the amount of time necessary for an

economically targeted early strength level to be reached, as the production cost associated with heat treatment application cannot be ignored.

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