

Maturity Testing of Lightweight Self-Compacting and Vibrated Concretes

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Abstract:

A series of laboratory tests were carried out to investigate the effect of temperature on the early-age strength development of lightweight self-compacting and vibrated concrete mixtures. These had been developed at Queen's University Belfast as part of a Technology Strategy Board funded project aimed at developing lightweight and low energy concretes. The new mixtures incorporated high volumes of pulverised fuel ash (PFA), ground granulated blast furnace slag (GGBS), and limestone powder (LSP). Activator, *i.e.* sodium sulphate, was used to improve the early age strength development of vibrated concrete mixtures proportioned with PFA and GGBS. For each mixture, concrete cubes were manufactured and cured under isothermal (20 °C, 30 °C, 40 °C and 50 °C) as well as adiabatic conditions. The temperature rise under adiabatic curing conditions was also

1 measured. The resulting isothermal strength data were analysed to determine the apparent
2 activation energies of the binders/mixtures used. The suitability of maturity methods for
3 predicting concrete strength development of these low energy lightweight self-compacting
4 and vibrated concrete mixtures under non-isothermal, *i.e.* adiabatic, curing was assessed.
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13 **Keywords:** Self-compacting concrete, lightweight concrete, activator, maturity functions,
14 activation energies, strength prediction.
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22 **1 Introduction**

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25 Novel low energy mixtures with self-compacting and/or lightweight properties were
26 developed at Queen's University Belfast as part of a Technology Strategy Board funded
27 project^[1-6]. These were intended for use by precast concrete manufacturers for products such
28 as coffered slab units for office buildings and individually cast voussoirs of the FlexiArchTM
29 bridge units^[7]. Products with low carbon footprint are sought after for the construction of
30 new buildings, which increases ratings of such buildings in environmental assessment
31 methods and rating systems, *e.g.* BREEAM^[8]. The new mixtures incorporated high volumes
32 of pulverised fuel ash (PFA) and ground granulated blast furnace slag (GGBS). Selected
33 vibrated mixtures, proportioned with PFA and GGBS, were activated with sodium sulphate in
34 order to improve their early age strength development. Such mixtures are more sensitive to
35 temperature than Portland cement mixtures. There was therefore the need to establish
36 whether maturity functions could be used to monitor early age strength development. These
37 could be used by the precast concrete manufacturer to (a) control the temperature of the
38 casting bed to the required one to obtain the early age strengths needed for lifting the units,
39 (b) identify strength variations along the depth of the element, since the heating was on the
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underside only, so as to avoid weak concrete at the top and subsequent failures during lifting, and (c) possible quality control assurance, *i.e.* for ensuring the strengths required are achieved, even in extreme cold weather situations, before lifting.

The need for estimating the effects of steam curing treatments on strength development led, in around 1950, to the development of maturity methods which aimed at accounting for the combined effect of time and temperature on the strength development of concrete. Carino^[9] has reviewed the historical development of maturity functions in great detail and only a summary of this is included here. It was proposed that the measured temperature history during the curing period could be used to compute a single number that would be indicative of the concrete strength. Saul^[10] called this single factor “maturity”:

$$M = \sum_t (T - T_0) \cdot \Delta t \qquad \text{Equation 1}$$

where: M is the maturity, °C-days,
T is the average temperature (20 °C for standard curing) over the time interval Δt , °C,
T₀ is the datum temperature, °C,
 Δt is the time interval, days.

This equation has become known as the Nurse-Saul function and it can be used to convert a given temperature-time curing history to an equivalent age of curing at a reference temperature as follows:

$$t_e = \frac{\sum (T - T_0)}{(T_r - T_0)} \cdot \Delta t \quad \text{Equation 2}$$

where: t_e is the equivalent age at the reference temperature, days,
 T_r is the reference temperature, °C.

Equivalent age represents the duration of the curing period at the reference temperature that would result in the same maturity as the curing period at other temperatures. The equivalent age concept, originally introduced by Rastrup^[11], is a convenient method for using other functions besides Equation 1 to account for the combined effect of time and temperature on strength development. Equation 2 can be written as:

$$t_e = \sum (\beta \cdot \Delta t) \quad \text{Equation 3}$$

where:

$$\beta = \frac{(T - T_0)}{(T_r - T_0)}$$

The ratio β , which is called the “age conversion factor”, is used to convert a curing interval Δt to the equivalent curing interval at the standard reference temperature.

Functions described above are for calculating a maturity index (temperature-time factor or equivalent age) based on the temperature history of the concrete. Several functions have also been proposed to relate concrete strength to the maturity index^[12-19]. The following S-shape function proposed by Carino^[20] (Equation 4) is the one recommended in the ASTM

Standard^[21] procedure. Regression analysis is needed to provide for each curing temperature the rate constant, k_T , the ultimate strength, S_u , and the setting time, t_0 , of the mortar mixture. In order to calculate the apparent activation energy, E_a , the ASTM Standard's^[21] recommendation is to plot $\ln(k_T)$ against $1/T_{abs}$ (given in 1/Kelvin), where T_{abs} is the absolute curing temperature. The slope of the trend line is equal to $-Q$ and the activation energy for the mixture will be equal to $Q \cdot R$, where R is the universal gas constant equal to $8.31 \text{ J/K} \cdot \text{mol}$.

$$S = \frac{S_u \cdot k_T \cdot (t - t_0)}{1 + k_T \cdot (t - t_0)} \quad \text{Equation 4}$$

where: S strength at age t , MPa,
 S_u ultimate mortar strength at temperature T , MPa,
 k_T rate constant at temperature T , 1/day,
 t test age, days,
 t_0 age at which mortar strength development is initiated at temperature T , days.

The equivalent age is then related to Q based on the following equation:

$$t_e = \sum e^{-Q \left(\frac{1}{T_a} - \frac{1}{T_s} \right)} \cdot \Delta t \quad \text{Equation 5}$$

where: T_a average temperature of concrete during time interval Δt , K,
 T_s specified reference temperature, K.

Apparent activation energies can be determined using “equivalent” mortar specimens, as described in ASTM Standard C1074-98^[21] and the results applied to the concrete under

1 investigation. Values for activation energies reported in the literature^[20] range from 33,500
2 J/gmol to 63,600 J/gmol, see Table 1. All of these concrete mixtures had normal weight
3 aggregate and not lightweight aggregate. According to ASTM Standard C1074-98^[21], tests
4 can be performed on mortar specimens and the results applied to the concrete under
5 investigation. The equivalent mortars need to have the same water-binder ratios and
6 superplasticiser dosages as the concretes. The sand/binder ratios also need to be equal to the
7 coarse/binder ratios of the concretes. These requirements are to ensure that the strength
8 development of the mortar specimens is similar to that of the corresponding concrete
9 mixtures. Unfortunately, equivalent mortar specimens cannot be used for lightweight
10 concretes as their densities will be much different and this will affect the strength
11 development. Activation energies need therefore be determined on concrete cube specimens.
12 The volume of concrete is considerable in this case and it is not surprising that (a) the
13 equivalent mortar method is favoured, and (b) no data for activation energies has been found
14 in the literature for lightweight aggregate concretes.

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36 Therefore, the aim of this project was to determine activation energies for lightweight self-
37 compacting and vibrated concretes and to determine the suitability of maturity methods for
38 predicting concrete strength development under non-isothermal (adiabatic) curing.

39 40 41 42 43 44 45 46 47 **2 Material and Experimental Procedures**

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50 Eight concrete mixtures were prepared according to mixture proportions derived at Queen's
51 University Belfast (QUB)^[1-3] in close collaboration with the industry and these are shown in
52 Table 2. For each concrete mixture, two batches of concrete (90 litres each) were
53 manufactured.

2.1 Materials

A single batch of Portland cement (PC) CEM I 42.5N was used throughout. When required, PC was partially replaced with pulverised fuel ash (PFA), ground granulated blast furnace slag (GGBS) or limestone powder (LSP). The fine aggregate was a very fine sand where 69% of the particles passed through the 600 µm sieve. The coarse aggregate was all but for one mixture sintered fly ash lightweight aggregate (Lytag), size 4 to 14 mm. One mixture used normal weight aggregate in the form of granite with a maximum aggregate size of 20 mm. All aggregates were air-dried before use. The moisture content was determined by oven drying a sample and then allowance was made for absorption when calculating batch weights for mixing. Two superplasticisers were used: SP1, a superplasticising and accelerating concrete admixture based on polycarboxylate polymers and SP2, a third generation synthetic polycarboxylate polymer-based superplasticiser. The specific gravity of SP1 was 1.08 and that of SP2 was 1.10, whilst their solid contents were equal to 40% and 35%, respectively. A sodium sulphate activator was used for some of the mixtures.

2.2 Mixing, Casting, Curing and Testing of Concrete Specimens

Materials for concrete mixtures were weighed and placed in a 0.1 m³ capacity horizontal pan mixer. For vibrated concrete mixtures the coarse aggregate and sand were first placed in the mixer with half the water, mixed for 30 second and left to stand for 10 minutes. Cement and other powders, *i.e.* PFA or GGBS, were then added followed by the remaining water. In the case of the activated mixtures the remaining water contained the dissolved sodium sulphate. These were then mixed for 2 to 3 minutes before adding the superplasticiser. The specimens were compacted in two layers and subsequently wrapped in polythene sheet and transferred

1 to curing tanks/environmental chamber. For self-compacting mixtures the coarse aggregate
2 and sand were first placed in the mixer with 2/3 of the water and mixed for 2 minutes. GGBS
3 or LSP were then added and mixing continued for another 1 minute. Cement was
4 subsequently added and mixing continued for a further 1 minute before adding the remaining
5 water and superplasticiser. The concrete was mixed for a further 2 minutes.
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14 The 100 mm size concrete cubes were manufactured and water cured under isothermal
15 conditions (20 °C, 30 °C, 40 °C and 50 °C). Another set of 100 mm size cubes was cast for
16 water curing under adiabatic conditions. In addition, an “adiabatic” concrete specimen was
17 cast in a plywood 240 mm size cube mould lined with 20 mm expanded polystyrene to
18 further insulate it and heavy duty polythene to prevent moisture loss. This “adiabatic”
19 specimen (240 mm size cube) was then placed in an environmental chamber which was set
20 up in the way described below so that it would follow the adiabatic temperature history of the
21 concrete.
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37 For each curing regime, 3 replicate cubes were tested at the following ages: 3 hours, 6 hours,
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45 **2.3 Adiabatic Temperature Measurements**

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48 The adiabatic temperature rise due to hydration of cement is the temperature rise which will
49 occur if fresh concrete is stored in a perfectly insulated environment, *i.e.* one from which no
50 heat loss can occur. To achieve this state it is necessary to either heavily insulate the concrete
51 or alternatively to ensure that the environment in which the concrete is stored is at the same
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1 or nearly the same temperature as the concrete. The latter approach was adopted in this
2 research programme, see Figure 1.
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7 Following test set-up was used^[22]. Two copper/constantan thermocouples were inserted,
8 through a hole in the top of the mould, in the “adiabatic” concrete specimen (240 mm size
9 samples). Two more copper/constantan thermocouples were used to monitor the temperature
10 of the curing tank. The thermocouples were all connected to a computer which not only
11 recorded the temperatures but also was set to activate the cabinet when the temperature
12 difference between the curing tank and the concrete was 1 °C. It can be assumed, based on
13 the fact that there was no temperature drop after the maximum had been reached, that there
14 was only very little heat loss and no adjustment was needed for the results.
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29 **3 Results and Discussion**

30 **3.1 Activation Energies**

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32 The concrete compressive strength test results for all 4 curing temperatures, *i.e.* 20 °C, 30 °C,
33 40 °C and 50 °C, were plotted as shown in Figure 2. The parameters S_u , t_0 and k_T in
34 Equation 4 were obtained by regression analysis using a commercially available statistical
35 analysis software package called SigmaPlot^[23]. Table 3 shows these three parameters for
36 each concrete mixture and for each curing temperature.
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50 The datum temperature of each mixture was not estimated by plotting the k_T values against
51 the curing temperature. This requires a trend line to be fitted and the datum temperature
52 deduced from its intercept at the x-axis. However, in this study the datum temperature
53 determined with this procedure has been shown not to be reliable and, therefore, the value of
54 -11 °C was used instead for strength predictions. This is the average of what has been
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recommended in literature, *i.e.* usually taken to be between -10 °C and -12 °C
Plowman^[9, 10, 12].

In order to calculate the apparent activation energy, E_a , the ASTM Standard's recommendation is to plot $\ln(k_T)$ against $1/T_{abs}$ (1/Kelvin), where T_{abs} is the absolute curing temperature, see Figure 3. The slope of the trend line is equal to $-Q$ and the activation energy for the mixture will be equal to $Q \cdot R$, where R is the universal gas constant equal to 8.31 J/Kmol. Activation energies determined are shown in Table 4.

Activation energy values were found to vary approximately from 20 to 42 kJ/mol. The higher the activation energy the higher was the effect of temperature on the strength development of the concrete mixtures.

Neat Portland cement mixtures, *i.e.* CEM I, are expected to be at the lower end of the range of activation energies, *i.e.* around 20 to 27 kJ/mol. Values found in the literature, see Table 1, increase confidence in these values. Higher values reported seem to be associated with high strength concrete mixtures with low water-cement ratios. Although it is generally accepted^[24] that activation energies may not be affected by the water-cement ratio, there is information in the literature implying that this may only be true for normal strength concretes^[25].

Activation energies for PFA mixtures quoted in the literature (Table 1) vary a lot, *i.e.* from 20 to 37 kJ/mol. The higher values appear to be from research carried out in USA and may be associated with high calcium content of PFA which is not available in the UK.

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Activation energies for GGBS mixtures have been reported to be at the high end of the range, *i.e.* from 40 to 60 kJ/mol (Table 1). It is worth noting that the values determined in this programme of work are at the lower end of the range reported in the literature despite one of the two GGBS mixtures having a chemical activator. The activation energy of the activated GGBS mixture was actually found to be lower than the one without it.

3.2 Strength Prediction of Adiabatically Cured Concretes

The strength development of concretes under adiabatic curing regimes is challenging to predict. The temperature histories, see Figure 4, are higher than those that are normally recorded in in-situ construction of structural elements. The temperature rise also occurs much earlier than concrete cast in-situ. Nonetheless they offer a way of calibrating the accuracy of strength predictions made with previously determined activation energies.

The neat PC lightweight concrete mixture, *i.e.* LW-PC Control, reaches a peak temperature of around 80 °C (Figure 4). This mixture has the highest cement content from the concretes that are not self-compacting concretes. Use of PFA (LW-PFA) significantly reduces the peak temperature of the concrete by more than 20 °C. It is interesting to note the effect of the activator (LW-PFA Activated), *i.e.* it accelerates the reaction so that the peak temperature occurs earlier but does not increase the peak temperature. Use of GGBS (LW-GGBS) reduces the peak temperature less than PFA, *i.e.* by only 10 °C. It is worth noting that the use of the activator with this binder (LW-GGBS Activated) appears to on one hand accelerate the reaction but on the other hand it reduces the peak temperature.

The self-compacting concrete with 100% PC (NWSCC-PC Control) has a 10 °C temperature difference from the vibrated one (LW-PC Control). A possible explanation for this difference

1 could be the different aggregate used. The self-compacting concrete with LSP (LWSCC-
2 LSP) has approximately the same peak rise as the vibrated concrete with neat PC (LW-PC
3 Control) despite having lower cement content and lower water-cement ratio. The self-
4 compacting concrete with GGBS (LWSCC-GGBS) has a total binder content of
5 approximately 600 kg/m³ and it therefore reaches a peak temperature of 92 °C.
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14 Figure 5 depicts the adiabatic compressive strength measured on the 100 mm size concrete
15 specimens. The strength development obtained at 20 °C, *i.e.* normal curing, the adiabatic
16 temperature history and the activation energies previously obtained were used to estimate the
17 strength development of adiabatically cured concrete mixtures and shown as “Predicted”
18 curve in Figure 5. The equivalent age, t_e , at time, t , was first calculated using Equation 5
19 which requires the value of activation energy $E = Q/R$ for the specific concrete (Table 4).
20 The specified reference temperature, T_s , of 293 K (20 °C) was used in Equation 5. The value
21 of equivalent age obtained, t_e , was then substituted for t in Equation 4 with constants S_u , k_T
22 and t_0 as previously determined for the strength data obtained for similar concretes cured at
23 20 °C (see Table 3). The strengths obtained, *i.e.* predicted, were then plotted versus t as
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43 The acceleration of the strength development resulting from higher early age curing
44 temperatures is predicted well thus increasing confidence in the values of activation energies
45 previously determined. The cross-over effect (*i.e.* at the same value of low maturity, a high
46 curing temperature results in greater strength than a low curing temperature, and conversely
47 at later maturities, result in lower strength^[26]) appears from an early age for some of the
48 mixtures tested, especially the ones with GGBS. The cross-over effect results in an over-
49 estimate of the ultimate strengths as it does not consider the detrimental effect of early age
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1 curing temperatures. On the other hand, the 28-day strength prediction for the lightweight
2 vibrated PFA concrete (LW-PFA) and the activated one (LW-PFA Activated) is
3 underestimated. The reason for this is that PFA contributes to the strength at later ages and
4 testing ages of 56 and 91 days are needed to determine accurately the strength versus age
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11 **4 Conclusions**

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18 There does not seem to have been any other study that has evaluated the use of maturity
19 functions for lightweight aggregate concretes. Conclusions from this work are:
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25 • The early age strength development of lightweight concretes can be predicted
26 relatively accurately from the activation energies determined in this research project.
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32 • Activation energies for lightweight aggregate concretes appear to be similar to those
33 of normal aggregate concrete with similar binders.
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40 • Equivalent ages can be determined based on the strength requirements of precast
41 concrete factories. These can then be used to determine the curing temperature
42 required, as well as its duration, for these strengths to be achieved.
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50 • Measurement of temperature history can be used together with maturity functions to
51 monitor the strength development of factory cast specimens if these are to be air
52 cured, *i.e.* variable temperature history.
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- Measurement of temperature history, together with maturity functions, can also be used for the estimation of in-place strength based on strength development data obtained under standard laboratory conditions

Improved early age strengths resulting from high early age curing temperatures can also be exploited in fast track construction. However, to achieve this then (a) the expected in-situ temperature history will have to be modelled using finite element analysis software – heat of hydration values, which can be obtained from adiabatic tests, will be needed as inputs, and, (b) the in-situ strength development will have to be predicted using maturity functions.

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Table 1: Activation Energy Values found in the literature

Concrete Mixture Identifier	Source	w/b	Activation Energy (kJ/mol)
CEM I (C25/30*)	Hatzitheodorou ^[27]	0.66	22.851 and 37.382
CEM I (C40/50*)	Hatzitheodorou ^[27]	0.46	18.063 and 29.698
Type I Cement	Carino & Tank ^[20]	0.60	48.000
Type I Cement	Carino & Tank ^[20]	0.45	61.100
General for Type I Cement (without admixtures)	ASTM C1074-98 ^[21]	–	40.000 – 45.000
CEM I + 30% PFA (C25/30*)	Hatzitheodorou ^[27]	0.53	19.440 and 22.539
CEM I + 30% PFA (C40/50*)	Hatzitheodorou ^[27]	0.35	27.309 and 34.506
Type I Cement + 20% PFA	Carino & Tank ^[20]	0.60	36.600
Type I Cement + 20% PFA	Carino & Tank ^[20]	0.45	33.100
CEM I + 30% GGBS (C25/30*)	Hatzitheodorou ^[27]	0.65	53.265 and 59.600
CEM I + 30% GGBS (C40/50*)	Hatzitheodorou ^[27]	0.46	41.296 and 41.606
Type I Cement + 50% GGBS	Carino & Tank ^[20]	0.60	51.300
Type I Cement + 50% GGBS	Carino & Tank ^[20]	0.45	42.700
CEM I + 10% Microsilica (C70/85*)	Hatzitheodorou ^[27]	0.25	38.999 and 50.997

* – Concrete compressive strength class according to BS EN 206-1:2000^[28]

Table 2: Concrete Mixture Proportions

Concrete Mixture Identifier	LW-PC Control	LW-PFA	LW-PFA-Activated	LW-GGBS	LW-GGBS Activated	NWSCC-PC Control	LWSCC-GGBS	LWSCC-LSP
<i>Mixture Constituents</i>	<i>Quantity kg/m³</i>							
CEM I	450	225	225	225	225	460	424	419
PFA	-	154	154	-	-	-	-	-
GGBS	-	-	-	211	211	-	181.5	-
LSP	-	-	-	-	-	-	-	180
Lyttag 4-14 mm	561	561	561	561	561	-	351	351
Sand	787	787	787	787	787	818	818	818
Granite	-	-	-	-	-	896	-	-
Na ₂ SO ₄	-	-	15.15	-	17.43	-	-	-
SP1	2.25	1.89	1.89	2.18	2.18	-	-	-
SP2	-	-	-	-	-	1.611	3.3	3.3
Free water	189	159	159	183	183	208	210	208
Free w/b	0.42	0.42	0.42	0.42	0.42	0.45	0.35	0.35

Table 3: Regression Parameters obtained from Equation 4

Concrete Mixture Identifier	Curing Temp. (°C)	Regression Parameters			
		S_u (MPa)	k_T (1/day)	t_0 (day)	R^2
LW-PC Control	20	45.1342	1.0747	0.2200	0.9978
	30	46.5641	1.4850	0.1820	0.9973
	40	43.6659	2.3291	0.1143	0.9919
	50	42.8027	1.9324	0.0618	0.9748
LW-PFA	20	36.0680	0.3633	0.2757	0.9932
	30	37.2000	0.4821	0.1419	0.9895
	40	42.6950	0.4868	0.0303	0.9875
	50	40.2881	1.0374	0.0823	0.9904
LW-PFA Activated	20	32.8476	0.6652	0.1940	0.9756
	30	37.9515	0.6812	0.0206	0.9585
	40	34.7283	1.2395	0.0690	0.9865
	50	32.0804	1.6305	0.0000	0.9649
LW-GGBS	20	47.9345	0.2419	0.3620	0.9954
	30	50.2172	0.4127	0.2245	0.9990
	40	48.0835	0.7337	0.1707	0.9910
	50	43.2951	1.1346	0.1199	0.9959
LW-GGBS Activated	20	40.0872	0.5518	0.2608	0.9969
	30	40.5744	0.8712	0.1983	0.9977
	40	39.9613	1.0074	0.0202	0.9895
	50	33.3558	2.1255	0.0000	0.9162
NWSCC-PC Control	20	63.3790	0.5950	0.3980	0.9963
	30	60.0823	0.8715	0.1982	0.9936
	40	57.9857	1.0004	0.0463	0.9938
	50	54.8870	1.4751	0.0646	0.9785
LWSCC-GGBS	20	56.3194	0.6146	0.3590	0.9968
	30	54.8325	1.0666	0.2331	0.9990
	40	52.5678	1.6869	0.1954	0.9971
	50	51.6407	2.5280	0.1170	0.9980
LWSCC-LSP	20	45.5570	0.8381	0.3491	0.9947
	30	42.4575	1.4086	0.1832	0.9952
	40	40.6134	1.6288	0.1033	0.9783
	50	38.6104	1.8124	0.0385	0.9828

Table 4: Activation Energy Values obtained using the ASTM Method^[21]

Concrete Mixture Identifier	Activation Energy (kJ/mol)
LW-PC Control	17.636
LW-PFA	24.591
LW-PFA Activated	25.724
LW-GGBS	41.032
LW-GGBS Activated	32.811
NWSCC-PC Control	22.501
LWSCC-GGBS	37.041
LWSCC-LSP	19.549

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Figure 01

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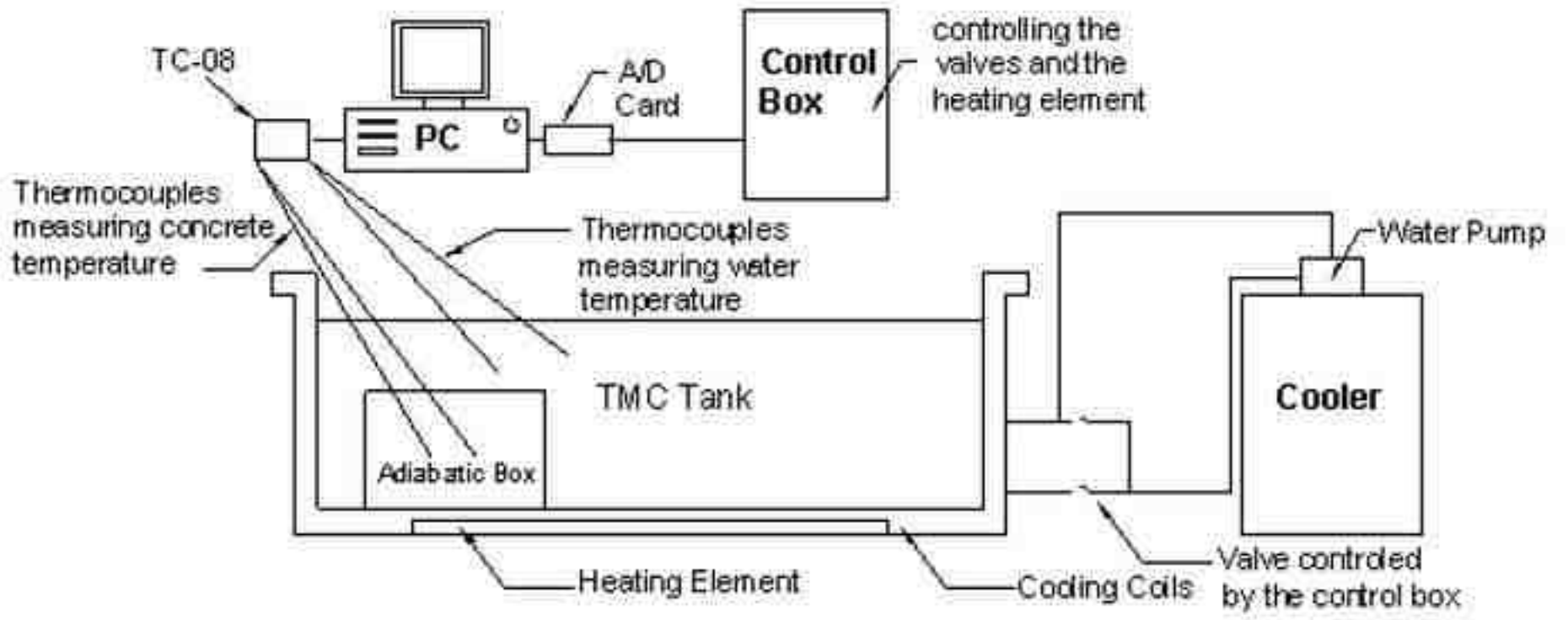


Figure 02a
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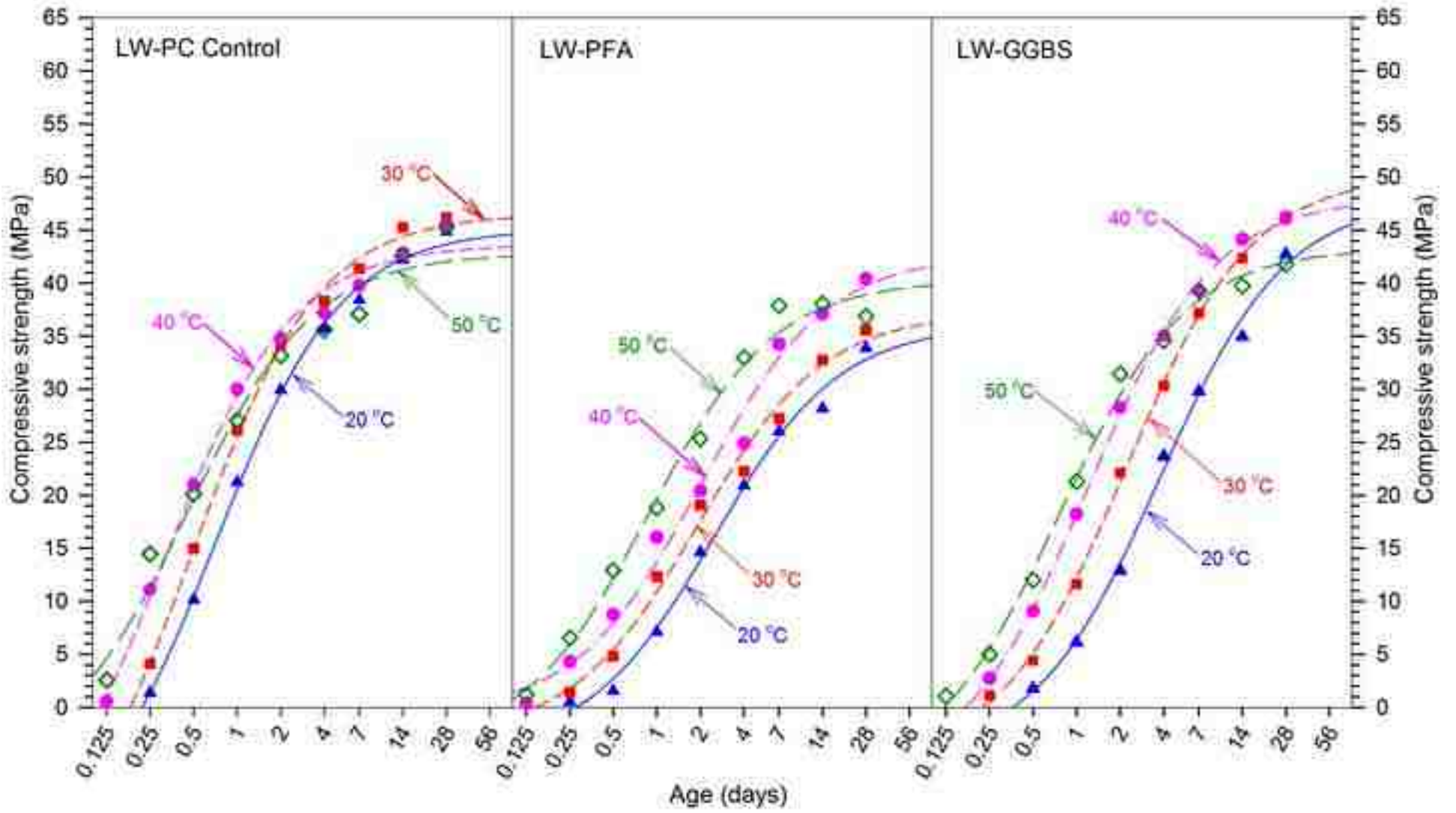


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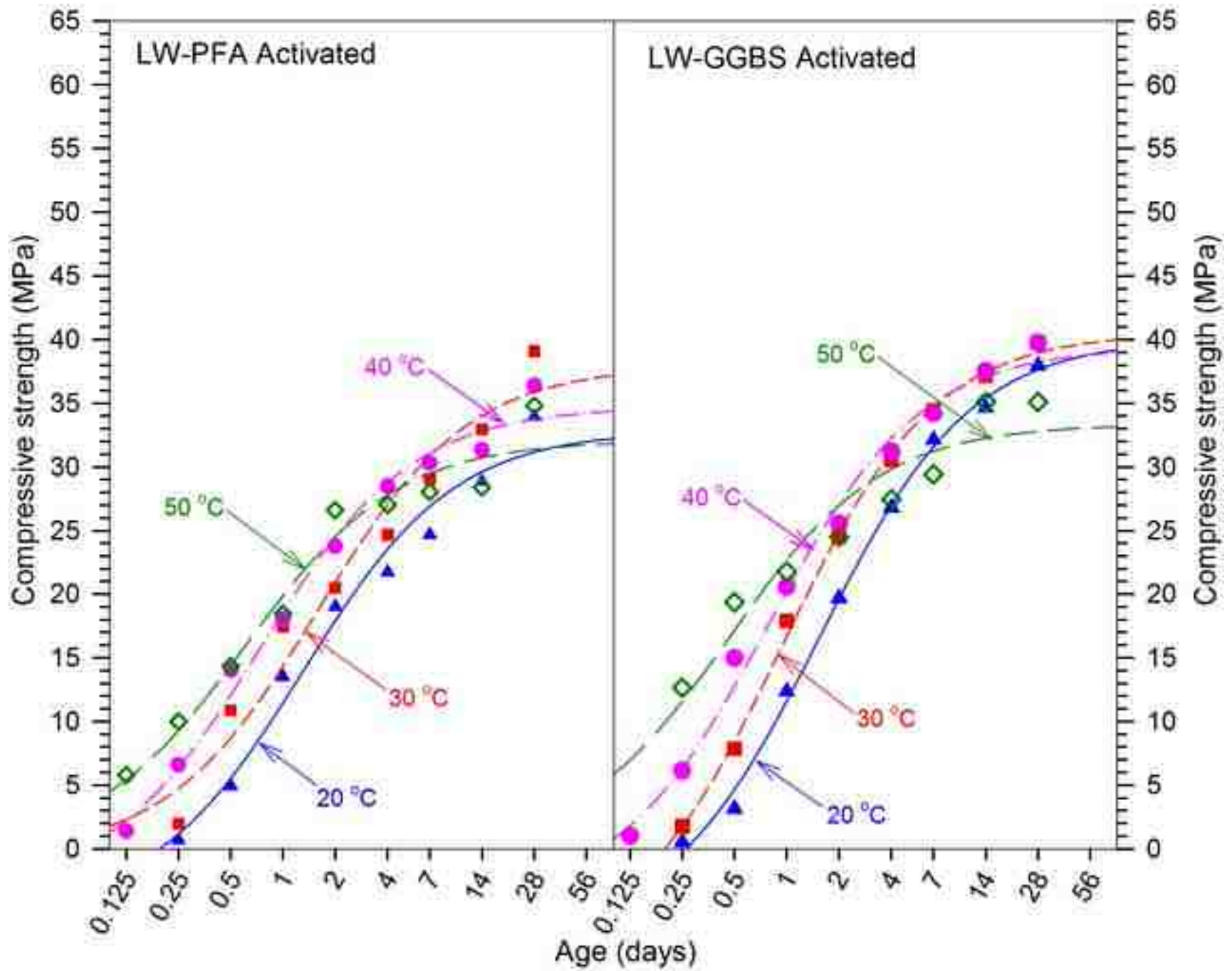


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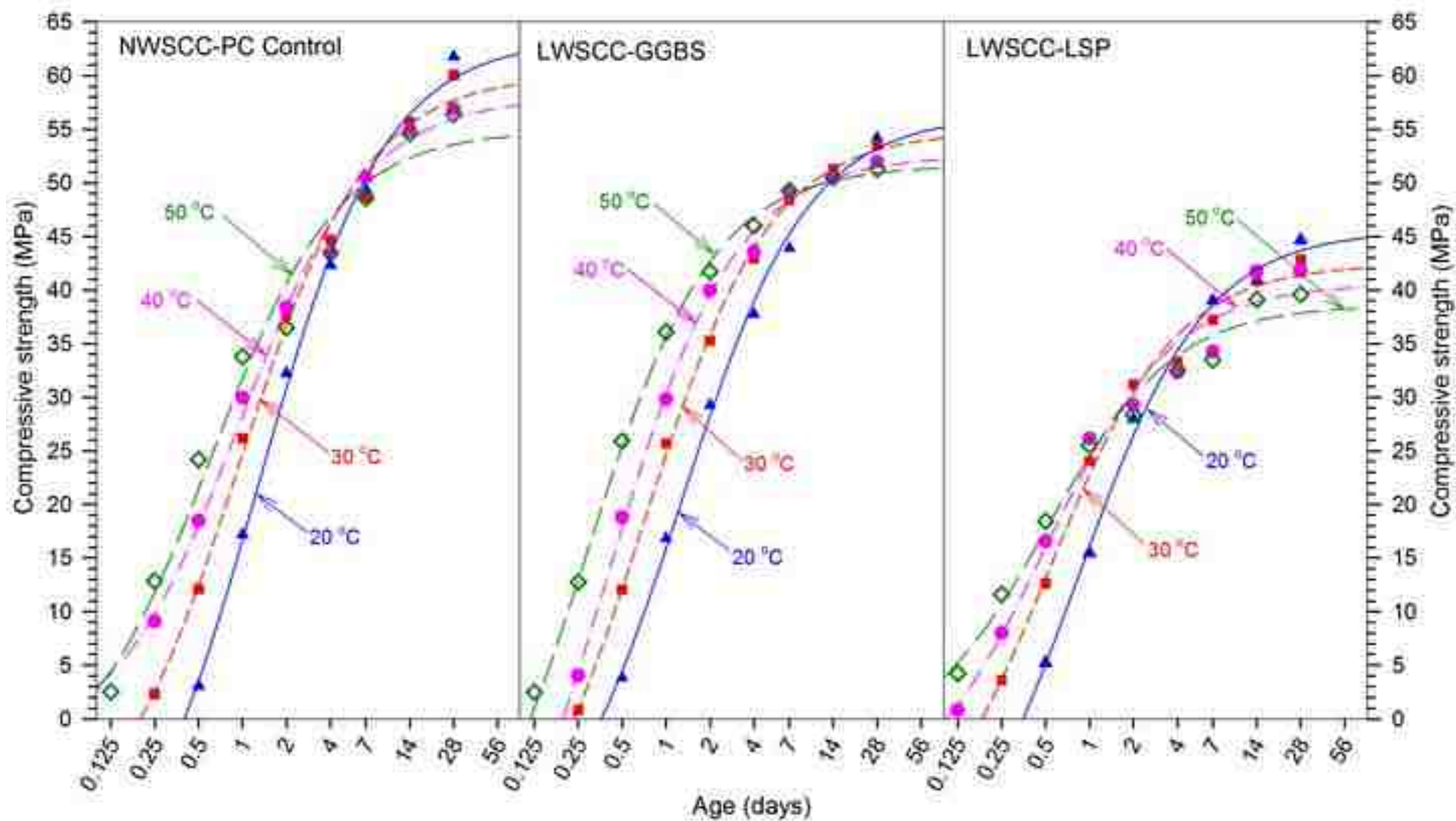


Figure 03

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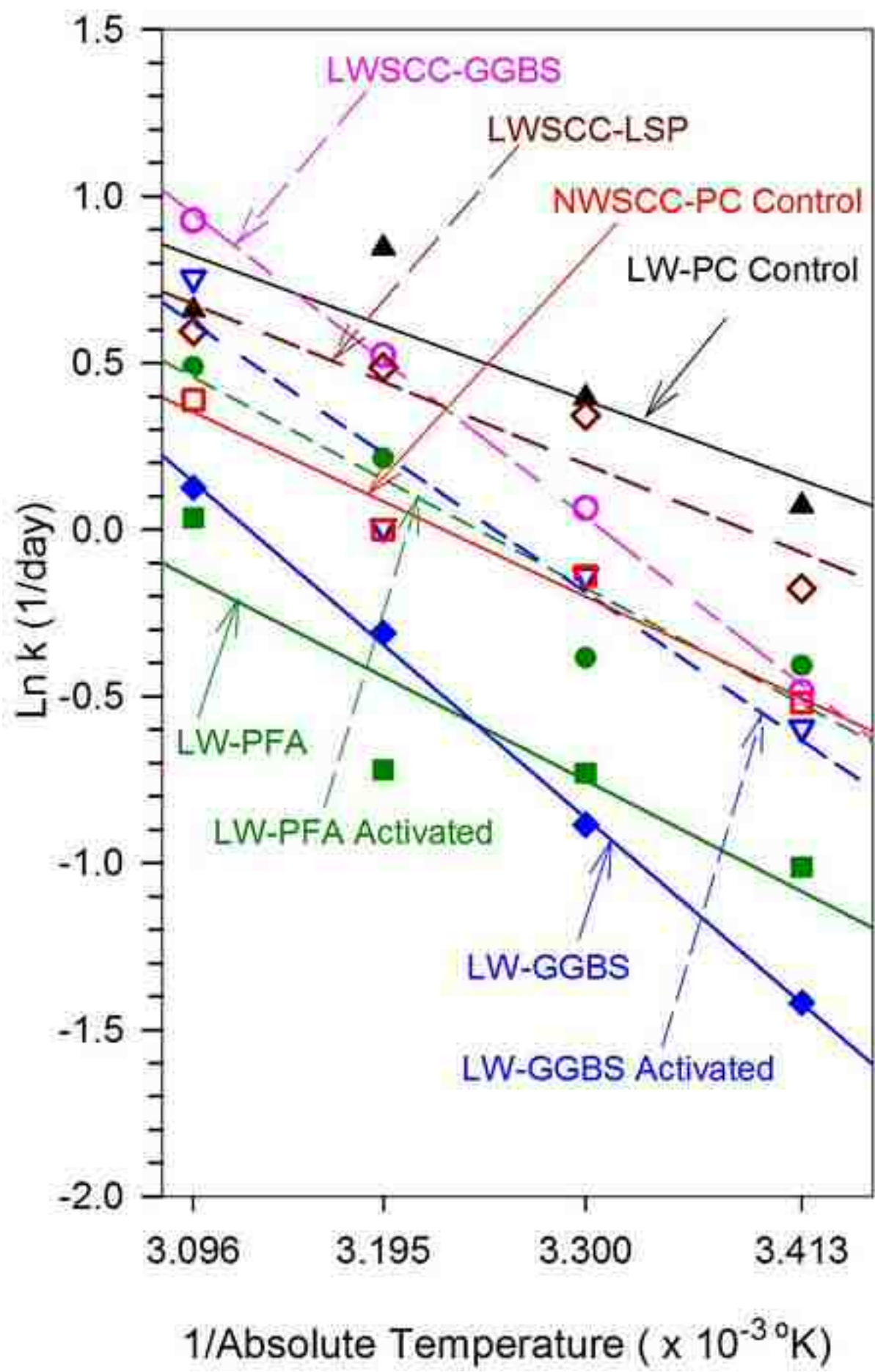


Figure 04

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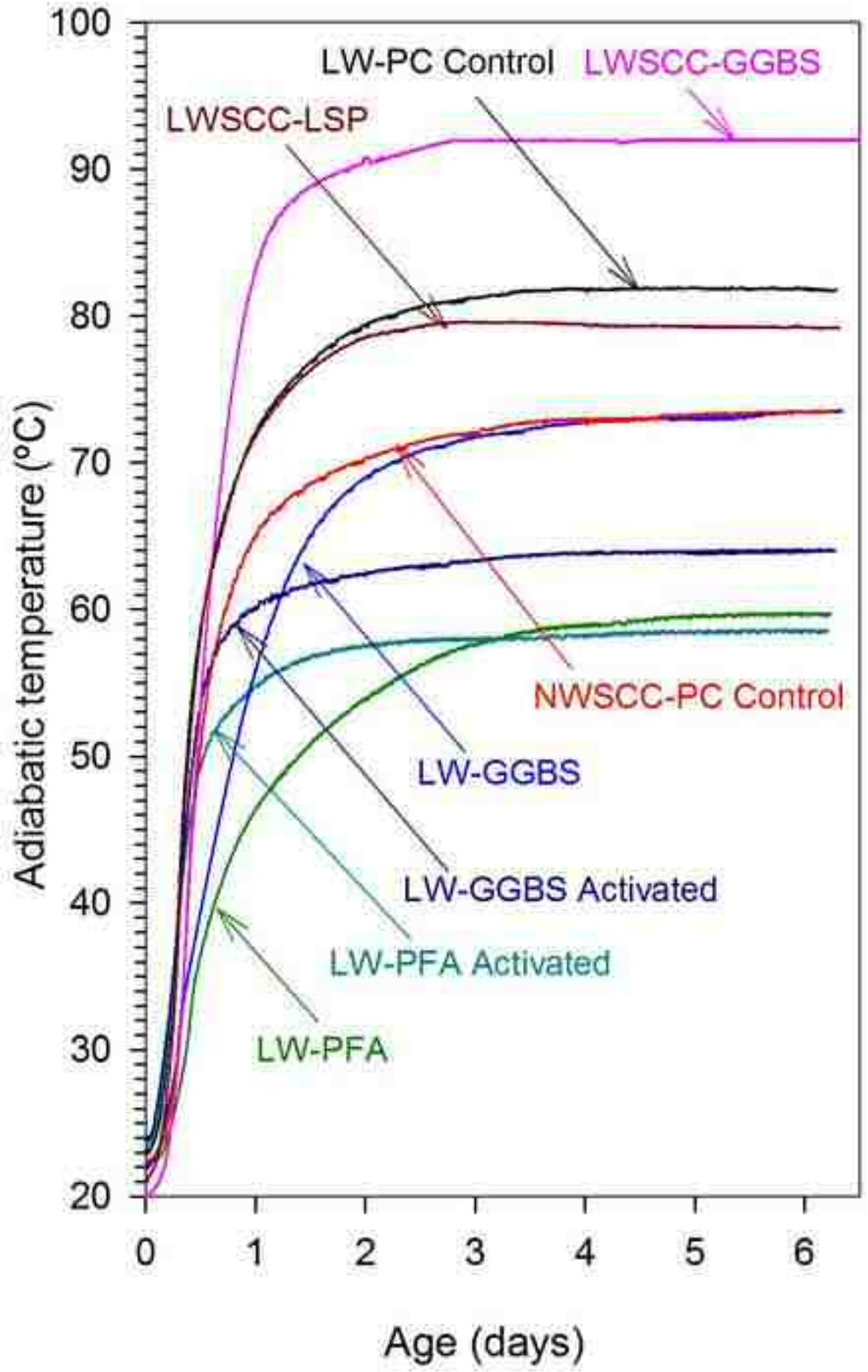


Figure 05a
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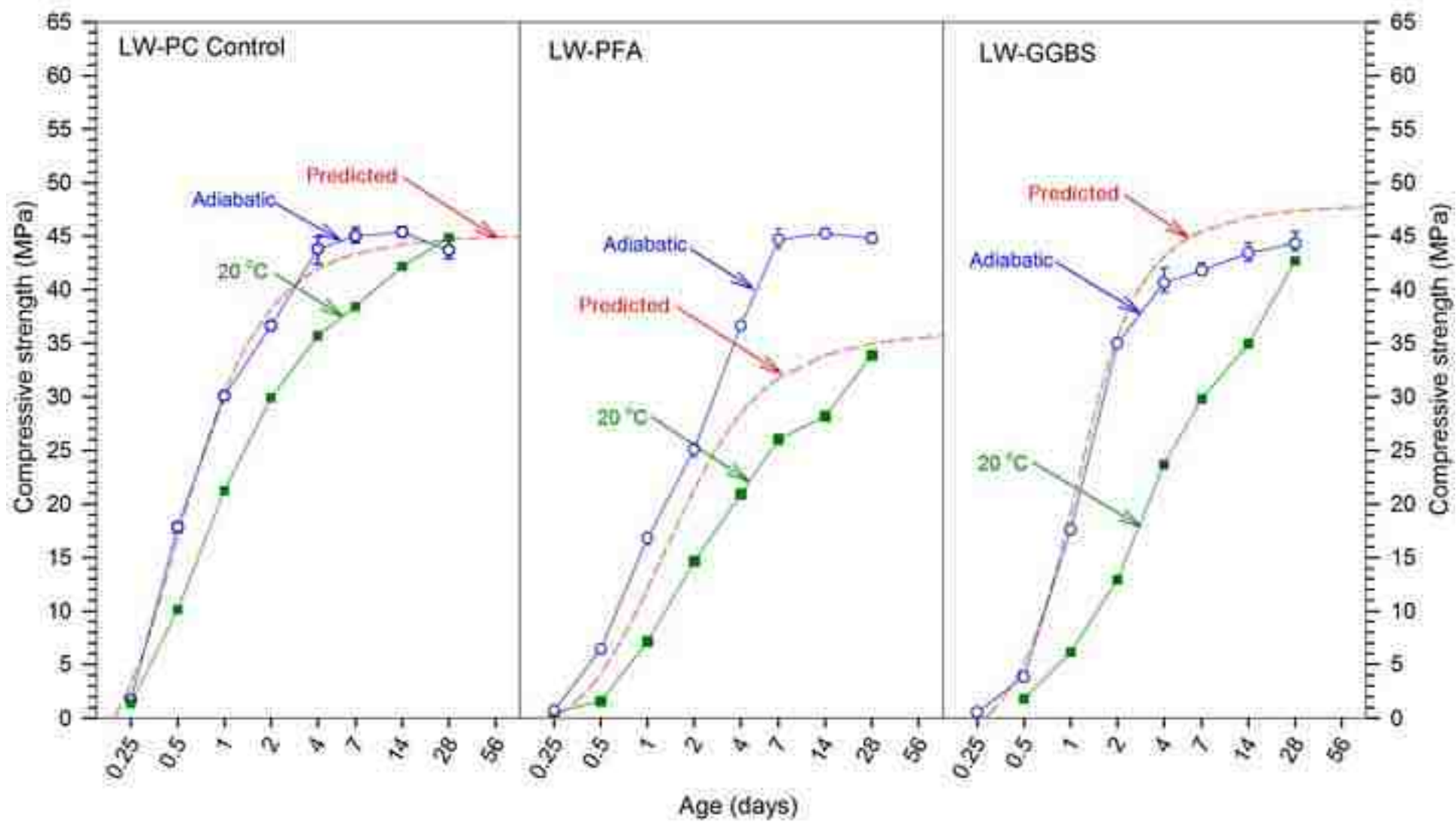


Figure 05b

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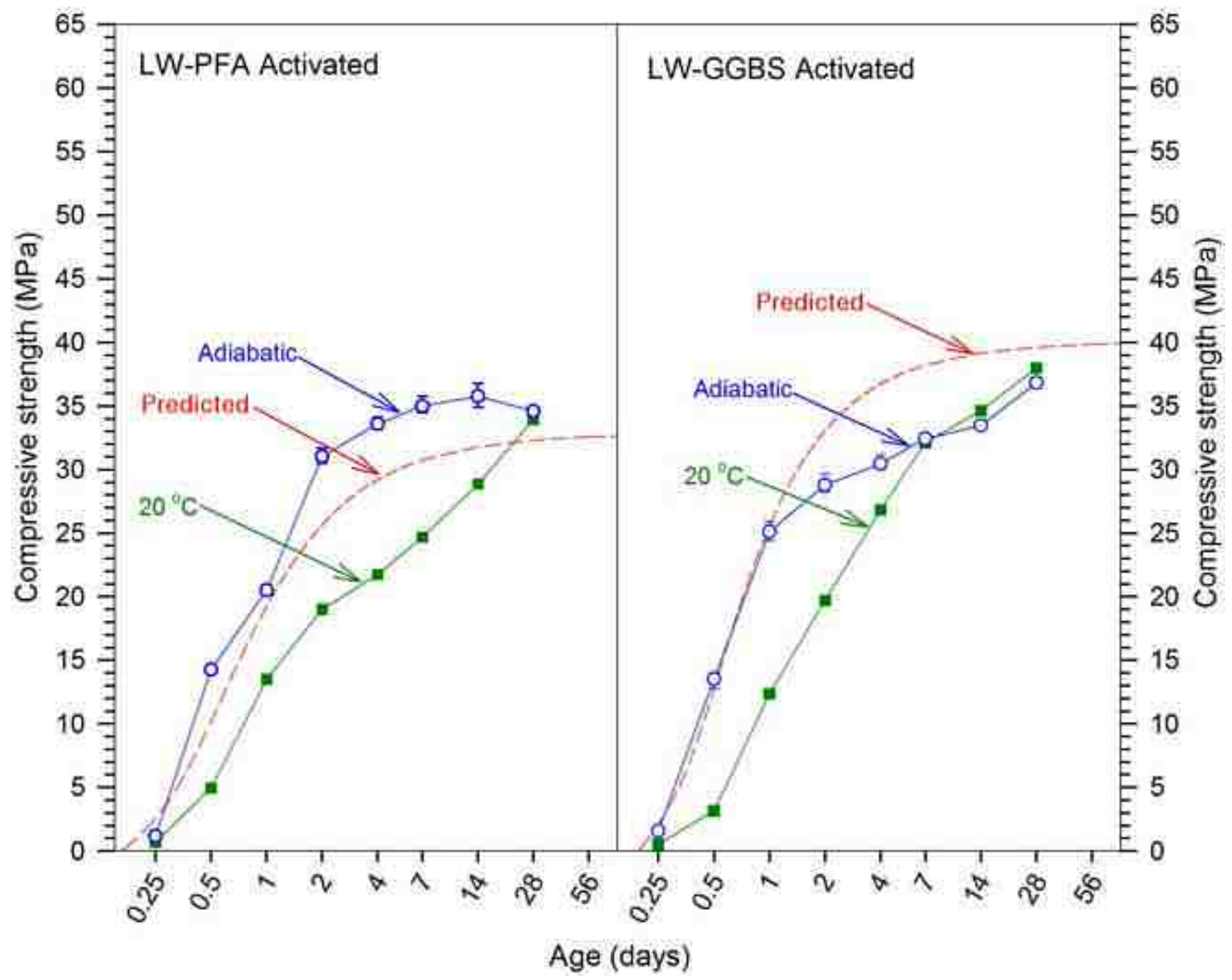


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