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ON THE AUTOGENOUS SHRINKAGE OF CEMENT PASTES

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Abstract

Introduction: This study focuses on autogenous shrinkage in cement pastes and presents a novel calculation method considering variations in internal relative humidity (IRH). IRH significantly influences autogenous shrinkage, and its evolution is modeled based on decline curves. The proposed method accurately evaluates autogenous shrinkage and aligns well with experimental data. Additionally, we calculate capillary depression and meniscus radius using the Laplace–Kelvin equation. **Methods:** To address early autogenous shrinkage in construction materials, we developed our calculation method, emphasizing IRH variation. We analyzed decline curves to model IRH and validated our model using literature-based experimental data. **Results:** Our validated model for predicting IRH and autogenous shrinkage in Portland cement pastes, based on cement paste hydration degree, water-to-cement ratio (w/c), and the critical degree of hydration (α_{cr}), closely aligns with experimental data and existing models.

Keywords: cement paste, autogenous shrinkage, internal relative humidity, prediction, modeling, decline curves.

Introduction

Autogenous shrinkage refers to the chemical shrinkage due to Le Chatelier contraction derived by the difference in density between hydration products and reactants (Davis, 1940); generally, it is a phenomenon caused by three parameters: depression capillary, superficial tension, and disjoining pressure (Mounanga, 2004).

A decrease in w/c ratio leads to an increase in autogenous shrinkage; this is due to the spacing between the particles. A low value of the radius of the meniscus generates a difficult circulation of water (capillary depression) which creates stresses in the matrix leading to its contraction.

Due to its complex composition, there are difficulties with modeling the shrinkage of cement paste. The researchers work on two types of models: macro (or phenomenological) and micromechanical models. Both have limitations because they do not take into consideration that cement paste is a porous medium, and fail to take into account time-dependent properties. In addition, identifying the model parameters for most of these models is rather complicated as well (Mounanga, 2004).

Internal relative humidity (IRH) plays a role in the development of capillary depression. During the progression of hydration, the reduction of IRH generates capillary tension in the interstitial water, then a modification of the radius of the capillaries to balance compressive stress in the solid skeleton; compressive stresses are accompanied by deformations (van Breugel, 2001).

IRH is a key parameter influencing the microstructure and durability of cement-based materials. Understanding the relationship between IRH and autogenous shrinkage is important for the design and performance evaluation of these materials. The evolution of degree of hydration, which is influenced by factors such as cement composition, curing conditions, and environmental exposure, affects both IRH and autogenous shrinkage. In this context, several models (Bažant and Prasannan, 1989; Bentz et al., 1994; Eguchi and Teranishi, 2005; Haecker et al., 2005; Hua et al., 1995, 1997; Koenders and van Breugel, 1997; Lura et al., 2003; Mabrouk et al., 2004; Neubauer et al., 1996; Paulini, 1994; Shimomura and Maekawa, 1997; Ulm et al., 2004; Xi and Jennings, 1997) have been proposed to predict the variation of autogenous shrinkage over time. However, the majority of these models do not account for the evolution of hydration degree, which limits their accuracy and applicability. Additionally, there are models predicting other properties such as IRH, Young's modulus, and temperature, all of which are in direct relationship with the progression of hydration degree.

The main goal of this study was to develop a comprehensive calculation method for early autogenous shrinkage in construction materials. To achieve this goal, we took into account the variation of IRH as a crucial factor in the shrinkage process. In a second step, we analyzed decline curves to model IRH. Furthermore, we validated the proposed model using experimental data from the literature

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and compared its performance with that of other models. The paper is organized as follows. Section 2 describes the method used to calculate autogenous shrinkage. Section 3 presents the proposed model for predicting IRH. Section 4 presents the validation results of the proposed model and calculation method and compares their performance with that of other models. Section 5 presents a discussion. Finally, Section 6 summarizes the conclusions of the study.

Calculation of Autogenous Shrinkage

Let us consider a small piece of material located in the full mass of the paste of cement subjected to compressive stress generated by the depression of the water in the capillaries (Fig. 1). The interstitial fluid in depression exerts a pressure on the element in question so that the element is subjected to a uniform triaxial stress (Fig. 2).

We can write the volume change ε as:

$$\varepsilon = \frac{\Delta V}{V} = \varepsilon_x + \varepsilon_y + \varepsilon_z. \tag{1}$$

By applying this equation to a differential element of volume and then integrating it, we can obtain the change in volume of a body even when the normal strains vary throughout the body.

By Hooke's law in mechanics of materials, the element is subjected to triaxial stress (beer et al, 2012):

$$\varepsilon = \frac{1-2\nu}{E}\left(\sigma_x + \sigma_y + \sigma_z\right). \tag{2}$$

In the case of uniaxial stress (prismatic specimen in compression), Eq. 2 is simplified to:

$$\varepsilon = \frac{\Delta V}{V} = \frac{(1-2\nu)}{E}\sigma, \tag{3}$$

where ν is Poisson's ratio and E is the elastic modulus of cement paste.

The determination of the deformation (ε) requires the knowledge of stress (σ), which is the value of the constraint generated by capillary depression. At younger ages, deformation mechanism by capillary depression is dominant due to high internal relative humidity; stress σ in this case is given by the Laplace–Kelvin equation:

$$\sigma = \frac{2\gamma}{r} = -\frac{RT}{V_m}\mathrm{Ln}(\mathrm{IRH}), \tag{4}$$

where σ is the capillary depression (Pa), R is the universal gas constant [8.314 J/(mol K)], T is the internal temperature in (K), V_m is the molar volume of water [18.02 10^{-6} m^3/mol], and IRH is the internal relative humidity (with values between 0 and 1), γ is the superficial tension (N/m) and r is the meniscus radius (m).

By bringing Eq. 4 into Eq. 3, we can deduce the equation of autogenous shrinkage:

$$\varepsilon = -\frac{(1-2\nu)}{E}\cdot\frac{Ln(IRH)RT}{V_m}. \tag{5}$$

The unknowns in this equation are IRH, E, ν and T. There are several models proposed in literature to calculate these variables. For example, L. Stefan et al. (2010) proposed an equation to predict the evolution of E as a function of hydration degree (Eq. 6); other authors considered the relationship between adiabatic temperature and degree of hydration (Cervera et al., 1999) (Eq. 7):

$$E(\alpha) = E(\alpha = \alpha_\infty)\left[\left\langle\frac{\alpha - \alpha_0}{\alpha_\infty - \alpha_0}\right\rangle^{+}\right]^{\beta}, \tag{6}$$

where β = w/c is a material parameter, α_∞ = 0.75 (Cervera et al., 1999) and α_0 = 0.2.

$$\frac{\alpha}{\alpha_\infty} = \frac{T}{T_\infty^{ad} - T_0}, \tag{7}$$

where T_∞^{ad} = 80°C and T_0 = 20°C.

In the upcoming section, we will present a model that predicts the IRH variable, which is the primary cause of autogenous deformations in Portland cement pastes. This model is a function of the degree of hydration developed and validated using the

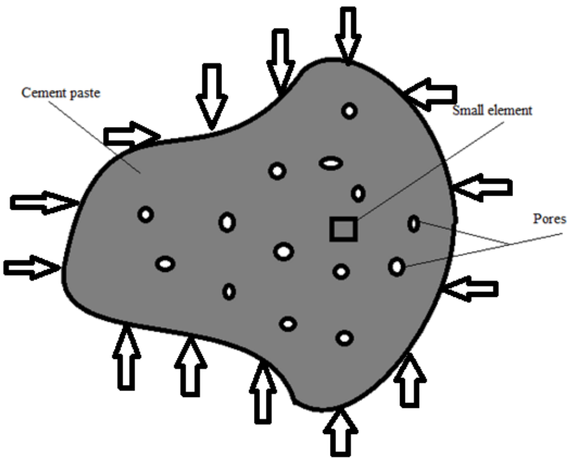

Fig. 1. Cement paste subjected to capillary depression

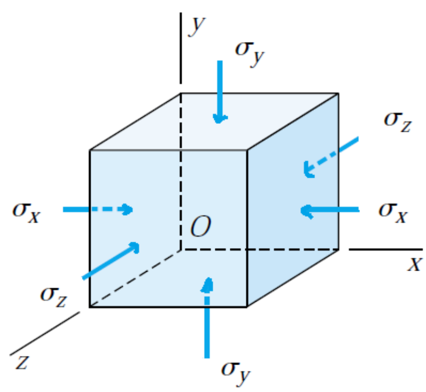

Fig. 2. Element in triaxial stress

data from previous studies. To accomplish this, we connect all variables in Eq. 5 to a single parameter, the degree of hydration (α), which we calculate using the three-parameter model (TPM) (Schindler and Folliard, 2005):

$$\alpha = \alpha_u \exp\left(\left(\frac{\tau}{t_{eq}}\right)^{\beta'}\right), \quad (8)$$

where τ and β' are parameters of the model, α_u is the ultimate degree of hydration and a function of w/c ratio, with the following equations:

$$\alpha_u = \frac{1{,}031\ \text{w/c}}{0{,}194 + \text{w/c}}; \quad (9)$$

$$\tau = 66.78 P_{C_3A}^{-0.154} P_{C_3S}^{-0.401} Blaine^{-0.804} P_{SO_3}^{-0.758}; \quad (10)$$

$$\beta = 181.4 P_{C_3A}^{0.146} P_{C_3S}^{0.227} Blaine^{-0.535} P_{SO_3}^{0.558}, \quad (11)$$

where P is the weight ratio in terms of total cement content.

The equivalent age can be defined as the same level of maturity of cement (mechanical properties and degree of hydration) acquired by specimens of the same composition but under different temperature history. Using the Arrhenius Law, we obtain (Hansen and Pedersen, 1977):

$$t_{eq} = \int_0^t \exp(-\frac{E_a}{R}\left(\frac{1}{T(\tau)} - \frac{1}{T_{ref}}\right))d\tau, \quad (12)$$

where E_a is the activation energy, *T(τ)* is the temperature history, and T_{ref} is the reference temperature generally equal to 20°C, *R* is the universal gas constant; the ratio E_a/R = 4000 for $T \geq$ 20°C for Portland cement (RILEM TC 119-TCE, 1997).

Modeling of Internal Relative Humidity

From the research of Bentz et al.(2004), we can deduce that the fineness of cement has no significant influence on the IRH–α relation. An IRH reduction is less in the systems with higher w/c ratio, due to low values of capillary depression. This is due to the initially larger spacing between cement particles (larger pore radius) (Bentz and Aïtcin, 2008), and is demonstrated mathematically by the Laplace–Kelvin relation.

As shown in Fig. 3, the variation of IRH with the progression of hydration degree is influenced by the w/c ratio. The evolution of IRH with the degree of hydration exhibits a critical point where a decrease begins; we call it the critical degree of hydration (α_{cr}).

The critical degree of hydration (α_{cr}) corresponds to the maximum value of the degree of hydration at which the decrease of IRH begins. It varies linearly between different ratios, as shown in Fig. 4, and can be determined through experimental tests.

In Fig. 5, we observe a simplified representation of the variation of IRH with the ratio *f*, where *f* (abscissa) is divided into two parts – negative and positive. The first part corresponds to the initial stages of hydration when the cement matrix is in a saturated state, while the second part exhibits an almost linear decrease in IRH with the degree of hydration, indicating the consummation of combined water with the progression of hydration reactions. To predict the IRH variation with *f*, we employ the classical analysis of decline curves (Arps, 1945).

The loss ratios are represented by an arithmetic series (Fig. 6), where the difference between successive loss ratios is the hyperbolic exponent *n*, which is approximately constant. Using this information, we can establish the following differential equation:

$$\frac{d\left(\frac{\text{IRH}}{d(\text{IRH})/df}\right)}{df} = -n. \quad (13)$$

Integration of Eq. 13 gives:

$$\frac{\text{IRH}}{d(\text{IRH})/df} = -nf - a_0. \quad (14)$$

The constant loss ratio at *f* = 0 is denoted by a_0. We can simplify the above equation as follows:

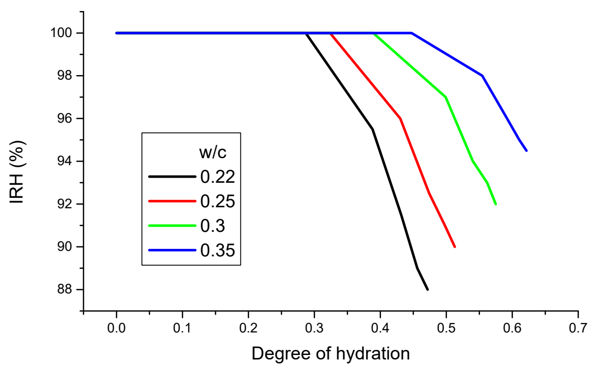

Fig. 3. Relation between IRH and degree of hydration (experimental data from Wyrzykowski and Lura (2013))

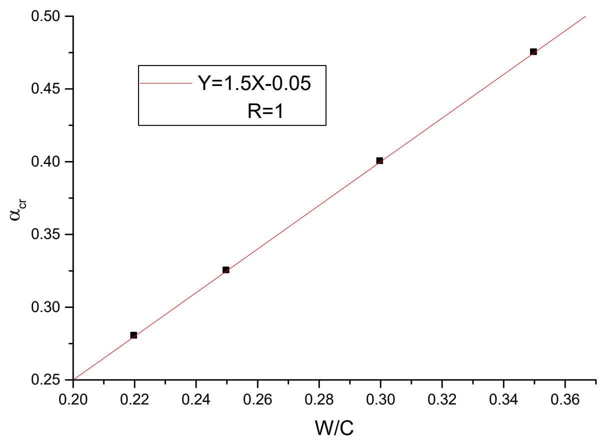

Fig. 4. Variation of the critical degree of hydration with w/c ratio

$$\frac{d(\mathrm{IRH})}{\mathrm{IRH}} = -\frac{df}{a_0 + nf}. \quad (15)$$

To eliminate the constants of integration from the previous second-order differential equation, we assume that IRH is equal to IRH_0, which is 100% for f = 0. This results in the following relationship between IRH and f:

$$\mathrm{IRH} = \mathrm{IRH}_0 \left(1 + \frac{nf}{a_0}\right)^{-1/n}. \quad (16)$$

We pose: $\frac{n}{a_0} = -k.$

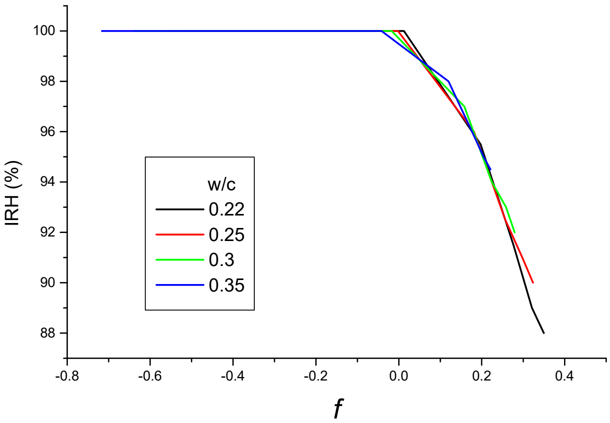

Fig. 5. Evolution of IRH with f ($f = \frac{\alpha - \alpha_{cr}}{\alpha_u}$)

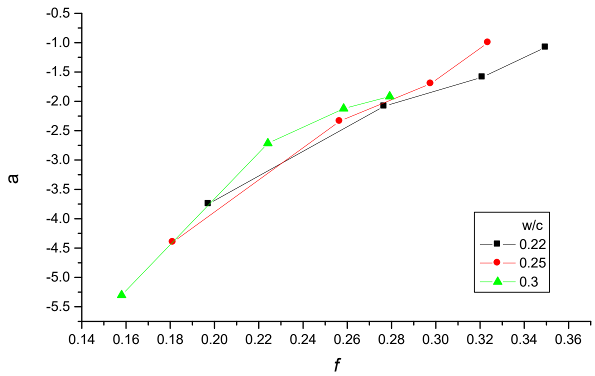

Fig. 6. Variation of the loss ratios $a = \frac{\mathrm{IRH}}{\frac{\Delta(\mathrm{IRH})}{\Delta f}}$ with f

The equation to predict the variation of IRH with α can be expressed as follows:

$$\mathrm{IRH} = \begin{cases} 100\ \% & \text{when } \alpha \le \alpha_{cr} \\ \mathrm{IRH}_0 \left(1 - k\left(\frac{\alpha - \alpha_{cr}}{\alpha_u}\right)\right)^{-n^{-1}} & \text{when } \alpha \ge \alpha_{cr} \end{cases}, \quad (17)$$

where n and k are parameters of the model that can be determined with regression from experiments as a function of w/c ratio and cement composition (C_3S and C_3A contribute most to heat release at early ages).

Validation

The free method for measuring autogenous shrinkage of cement pastes involves the preparation of unrestrained specimens, typically prismatic or cylindrical in shape. These specimens are allowed to undergo natural shrinkage without any external restraints, and their dimensional changes over time are measured. Various techniques such as linear displacement sensors, dilatometers, or image analysis can be employed to accurately monitor the specimen's length or volume. By analyzing the collected data, the autogenous shrinkage behavior of cement paste can be evaluated, providing insights into the material's intrinsic characteristics. The free shrinkage method offers a direct measurement of the unrestrained behavior of cement pastes, allowing for a better understanding of their volume changes and potential cracking risks.

To validate the proposed IRH model, we used the calculated degree of hydration values (obtained using Eq. 8) of the cements reported in literature (Huangand Ye, 2016; Kumarappa et al., 2018; Lu et al., 2020; Wei et al., 2015; Wyrzykowski and Lura, 2013). Subsequently, we used the last proposed method to calculate autogenous shrinkage for the experiments conducted in the work of Song et al. (2020). Table 1 presents the parameters of the proposed IRH model for various cement pastes.

Fig. 7 depicts the predicted values of the proposed IRH model with an overlay on experimental data from

Table 1. **Values of the IRH model parameters**

References	w/c	α_{cr}	α_u	–K	–n
Wyrzykowski and Lura, 2013	0.22	0.28	0.5479	1.997	8.938
	0.25	0.325	0.5805	2.13	10.65
	0.3	0.4	0.626	2.696	16.85
	0.35	0.475	0.663	6.14	18.49
Huang and Ye, 2016	0.25	0.31	0.5805	2.41	9.44
Lu et al., 2020	0.3	0.38	0.626	2.47	10.7
	0.4	0.52	0.69427	1.95	9.92
Huang and Ye, 2016	0.25	0.1	0.5805	1.19	12.63
Wei et al., 2015	0.3	0.2	0.626	1.495	15.8
Wyrzykowski and Lura, 2013	0.35	0.3	0.663	1.986	34.35
Kumarappa et al., 2018	0.4	0.4	0.694	2.68	63.395

literature (Huang and Ye, 2016; Lu et al., 2020). The results show a strong agreement between the model predictions and the experimental tests, indicating the effectiveness of the proposed model in predicting the variation of IRH with α. This validates the use of the model for further analysis and predictions in similar experiments.

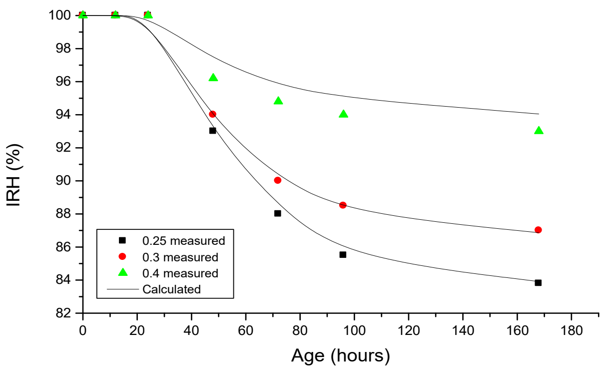

Fig. 7. Measured and calculated IRH (experimental data from Huang and Ye (2016), Lu et al. (2020))

In Figs. 8 to 11, we present the validation results of our proposed model using data from previous studies (Huang and Ye, 2016; Kumarappa et al., 2018; Wei et al., 2015; Wyrzykowski and Lura, 2013). Additionally, we compare the performance of our model with that of two other models in literature, namely the Song model (Song et al., 2020) and the Shen model (Shen et al., 2018).

Upon examining the figures (Figs. 8–11), it is evident that the proposed model exhibits an excellent agreement with the measured data, thus establishing its reliability in predicting the behavior of IRH. Moreover, a comparative analysis with two other models (Song (Song et al., 2020) and Shen (Shen et al., 2018)) demonstrated that the proposed model's performance was comparable to these existing models. This validation process offers robust evidence of the accuracy of the proposed model in predicting IRH.

For the 11 mixtures (Table 1) found in literature, we suggest the following equations of the parameters of the proposed IRH model:

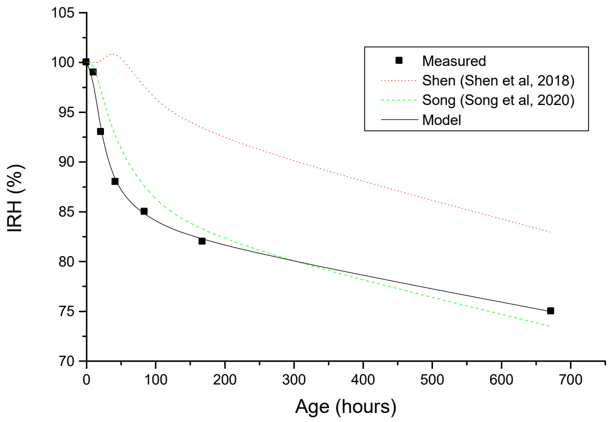

Fig. 8. Measured and calculated IRH for w/c = 0.25 (experimental data from Huang and Ye (2016))

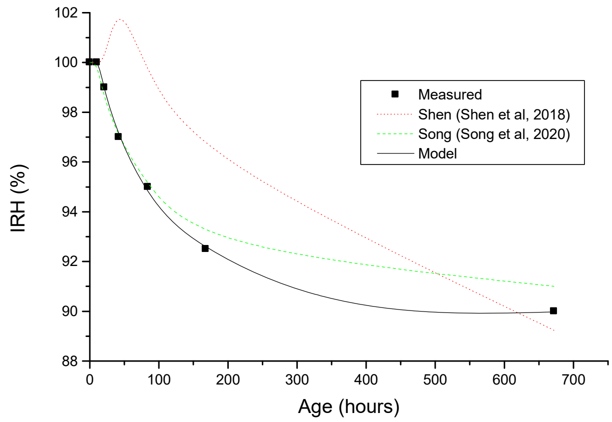

Fig. 10. Measured and calculated IRH for w/c = 0.35 (experimental data fromWyrzykowski and Lura (2013))

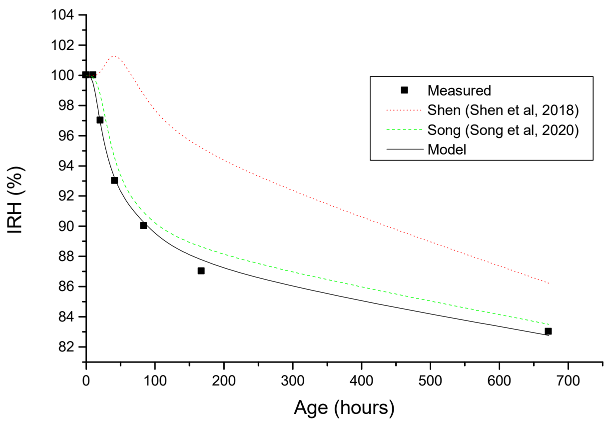

Fig. 9. Measured and calculated IRH for w/c = 0.3 (experimental data from Wei et al. (2015))

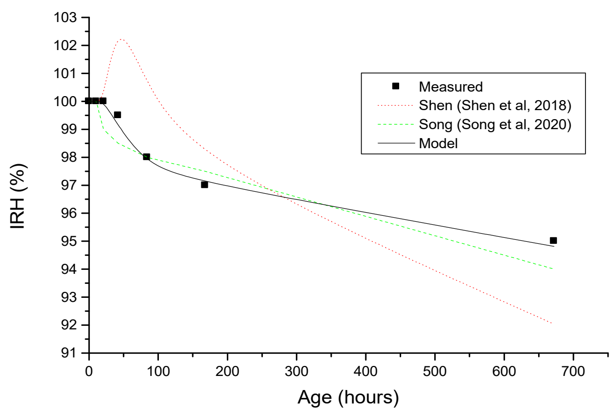

Fig. 11. Measured and calculated IRH for w/c = 0.4 (experimental data fromKumarappa et al. (2018))

$$k = \frac{1.02 - 2.663\left(\frac{E}{C}\right)}{1 - 4.52\left(\frac{E}{C}\right) + 4.94\left(\frac{E}{C}\right)^2}; \quad (18)$$

$$n = 8.05P_{C_3S} - 36.24P_{C_3A} - $$
$$-190.27(\ln\left(\frac{E}{C}\right)) + 5.81\left(P_{C_3S}\right)\ln\left(\frac{E}{C}\right) - $$
$$-25.92(P_{C_3A})\left(\ln\left(\frac{E}{C}\right)\right) - 250.37. \quad (19)$$

The observed small *p*-values (0.01) indicate significant relationships between the independent variables and the IRH parameters. The F-test shows improved prediction accuracy with each additional variable.

The following tables (Tables 2–5) present the calculated values of IRH and autogenous shrinkage along with their corresponding parameters. The results are then compared to the experimental data from Song et al. (2020).

Upon examining the tables (Tables 2–5), we can conclude that the autogenous shrinkage values obtained from the proposed model are in very good agreement with the experimental results from Song et al. (2020). Additionally, the calculated values of capillary depression are consistent with the values reported in literature (Lu et al., 2020; Song et al., 2020).

Discussion

The proposed model for predicting the internal relative humidity (IRH) and autogenous shrinkage of Portland cement pastes was validated using the data from previous studies. The model is based on the degree of hydration of the cement paste and takes into account the water-to-cement ratio (w/c) and the critical degree of hydration (α_{cr}) at which IRH starts to decrease. The validation results show that the proposed model fits very well with the experimental data and is comparable with other models in literature. The calculated values of autogenous shrinkage and capillary depression are also close to the experiments and literature values, respectively.

Table 2. **The case of w/c = 0.25**

Age (h)	α	IRH (calculated)	*E* (GPa)	ν (Stefan et al., 2010)	*T* (K)	σ (MPa) (calculated)	ε (μm/m) (calculated)	ε (μm/m) (measured)
0	0	100	0	0.4	273.15	0	0	0
10.5	0.25	98	19	0.235	293.12	2.73	73	100
21	0.39	93	26	0.225	304.41	10.19	202	200
42	0.48	88	29	0.225	311.73	18.39	330	300
84	0.53	85	30	0.22	315.72	23.67	415	400

Table 3. **The case of w/c = 0.3**

Age (h)	α	IRH (calculated)	*E* (GPa)	ν (Stefan et al., 2010)	*T* (K)	σ (MPa) (calculated)	ε (μm/m) (calculated)	ε (μm/m) (measured)
0	0	100	0	0.4	273.15	0	0	0
10.5	0.2	99.93	13	0.265	289.15	0.13	3	75
21	0.4	96	23	0.235	305.15	5.75	133	150
42	0.51	93	26	0.23	313.95	10.51	217	230
84	0.55	90	27	0.225	317.15	15.42	313	300

Table 4. **The case of w/c = 0.35**

Age (h)	α	IRH (calculated)	*E* (GPa)	ν (Stefan et al., 2010)	*T* (K)	σ (MPa) (calculated)	ε (μm/m) (calculated)	ε (μm/m) (measured)
0	0	100	0	0.4	273.15	0	0	0
10.5	0.2	100	7	0.28	289.15	0.09	6	5
21	0.4	99	14	0.25	305.15	1.41	50	75
42	0.51	97	16	0.24	313.95	4.41	140	150
84	0.58	95	18	0.234	319.55	7.56	229	230

Table 5. **The case of w/c = 0.4**

Age (h)	α	IRH (calculated)	*E* (GPa)	ν (Stefan et al., 2010)	*T* (K)	σ (MPa) (calculated)	ε (μm/m) (calculated)	ε (μm/m) (measured)
0	0	100	0	0.4	273.15	0	0	0
10.5	0.27	100	6	0.33	294.37	0.002	11	25
21	0.43	99.5	10	0.275	307.47	0.71	45	60
42	0.55	99	12	0.25	316.80	1.47	88	90
84	0.62	98	13	0.24	322.38	3.01	159	150

Conclusion

In conclusion, this study proposes a model for predicting the variation of internal relative humidity (IRH) with the progression of hydration in cement pastes. The proposed model was validated using the data from previous studies and showed good agreement with the experimental results.

Moreover, the study shows that there is a direct relationship between the decrease in IRH and the increase in autogenous shrinkage. This finding is important for the design of concrete structures, as it highlights the importance of controlling internal humidity in order to avoid excessive autogenous shrinkage.

The proposed IRH model can predict the decrease of IRH at both early and late ages, and it can be used to calculate the capillary depression and the meniscus radius using Laplace–Kelvin equation. Additionally, the proposed method of calculating autogenous shrinkage at early ages gives significant results, and the calculated values are close to the experimental data.

In summary, this study provides a comprehensive understanding of the relationship between IRH and autogenous shrinkage in cement pastes, and offers a practical model for predicting IRH and autogenous shrinkage at early and late ages. These findings are useful for the design and maintenance of concrete structures.

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К ВОПРОСУ ОБ АУТОГЕННОЙ УСАДКЕ ЦЕМЕНТНОГО ТЕСТА

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Аннотация
Введение: Данное исследование посвящено исследованию процессов развития аутогенной усадки цементных паст и представляет новый метод расчета, учитывающий изменения внутренней относительной влажности. Внутренняя относительная влажность существенно влияет на аутогенную усадку, и ее эволюция моделируется на основе кривых снижения. Предложенный метод точно оценивает аутогенную усадку и хорошо согласуется с экспериментальными данными. Кроме того, по уравнению Лапласа-Кельвина были рассчитаны капиллярная депрессия и радиус мениска. **Методы:** Чтобы предотвратить развитие ранней аутогенной усадки строительных материалов, был разработан новый метод расчета, который учитывает изменение внутренней относительной влажности. Проанализированы кривые снижения, использованные для моделирования изменения внутренней относительной влажности, и подтверждена достоверность новой модели на основе анализа эмпирических данных, представленных в других исследованиях. **Результаты:** Новая модель прогнозирования изменения внутренней относительной влажности и аутогенной усадки в портландцементных пастах, основанная на степени гидратации цемента, соотношении воды и цемента (в/ц) и критической степени гидратации (αcr), согласуется с экспериментальными данными и существующими моделями. Это исследование подчеркивает важность контроля внутренней влажности для уменьшения аутогенной усадки в бетонных конструкциях.

Ключевые слова: цементное тесто, аутогенная усадка, внутренняя относительная влажность, прогнозирование, моделирование, кривые снижения.