



# Influence of chemical-mineralogical properties of limestone on the shrinkage behaviour of cement paste and concrete made of limestone-rich cements

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## HIGHLIGHTS

- Drying shrinkage of concretes made of limestone-rich cements is affected by chemical-mineralogical properties of limestone.
- Clay minerals and alkali oxides are dominant parameters for the drying shrinkage of concrete with high limestone contents.
- The contribution of autogenous and carbonation shrinkage to the total shrinkage deformation is almost negligible for cement paste and concrete made of limestone-rich cements.

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## ABSTRACT

Shrinkage in concrete structures may lead to cracks which reduce the durability of concrete against aggressive mediums. In prestressed concrete elements shrinkage results in a significant loss of prestressing forces. Limestone-rich cements with limestone contents beyond the values of DIN EN 197-1 were developed in order to reduce the environmental impact of concrete. In this paper, the results of studies on the shrinkage behaviour of cement paste and concrete made of limestone-rich cements up to 70 wt.-% are presented. Results indicated that shrinkage of cement paste and concrete with high limestone contents is strongly dependent on the amount and the chemical-mineralogical properties of limestone. In this context, methylene blue value (MB-value) and alkali oxide content of limestone were identified as the key parameters controlling the drying shrinkage of cement pastes. Depending on the type of limestone, concrete samples made of limestone-rich cements had either higher or lower drying shrinkage than reference samples made of CEM I 52.5 R with the same w/c-ratio. However, lower drying shrinkage deformation was observed in comparison to the reference concrete made of CEM I with a comparable compressive strength. Furthermore, it was ascertained that the contribution of the autogenous shrinkage and carbonation shrinkage to the total shrinkage deformation is almost negligible for cement paste and concrete made of limestone-rich cements.

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## 1. Introduction

### 1.1. Problem definition and motivation

Concrete as the mass building material of the time is responsible for more than five percent of the global anthropogenic CO<sub>2</sub> release [1]. The major environmental impact of concrete comes from the CO<sub>2</sub> emissions during clinker production. It was realised that the reduction of Portland cement clinker in cement can lead to a decrease in the environmental impact of concrete. However, excessive substitution of clinker by limestone in Portland lime-

stone cements (more than 35 wt.-%) with a common water/cement-ratio is reported to be critical [2]. To overcome this problem, limestone-rich cements with high limestone contents up to 65 wt.-% were developed beyond the limits of EN 197-1 [3] based on a modified concrete technology proposed by [4]. This approach suggests a reduction of water content supported by the optimisation of the packing density and the use of high performance polycarboxylate ether superplasticizer. Nevertheless, preliminary studies showed that the long-term deformation of concretes made of limestone-rich cements are strongly dependent on the chemical-mineralogical properties of the used ground limestone as main cement component [3]. Significant change in the drying shrinkage and creep deformations up to 80% was reported when the type of

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limestone was varied. Nevertheless, studies to investigate the influence of chemical-mineralogical properties and high-contents of limestone as main cement component on the shrinkage deformation of hardened cement paste (hcp) and concrete are lacking.

### 1.2. Shrinkage of cementitious materials and influencing factors

Cracking due to the irreversible restrained shrinkage deformation is an undesirable phenomenon which can shorten the service life of the structure by easing the penetration of aggressive mediums. Meanwhile, excessive irreversible shrinkage deformation results in a significant loss of prestressing forces in prestressed concrete members which leads to a reduction in the bearing capacity or even failure of the member. Shrinkage is defined as the isothermal volume change of unloaded hydrated cement paste or concrete due to chemical reactions or changes in internal relative humidity (RH) [5]. Based on the origin and start time, shrinkage can be categorised into plastic shrinkage, chemical shrinkage, carbonation shrinkage, autogenous shrinkage and drying shrinkage [6,7].

Despite extensive conducted investigations, the shrinkage mechanisms are not well understood. According to the literature, three main mechanisms are proposed as the driving forces for the shrinkage of concrete. These include 1) surface free energy [8–11], 2) capillary tension [8,10,12,13] and 3) disjoining pressure [9,12,14]. The capillary tension acts when the capillary and gel pores are almost filled with water or condensed water from vapour (denoting a RH > 80%). It is visible that the capillary tension grows when the curvature of meniscus increases. But the total force due to capillary tension acting on surface of hardened cement paste reduces when acting perimeter decreases by losing moisture (reduction in degree of saturation). Surface tension can be defined as the results of asymmetry of attractive forces on atoms or molecules in the close vicinity of the solid surface. Increase in RH from near zero to 40% results in a rise in the adsorbed water layers on the surface of solid particles, i.e. C–S–H particles.

The reduction in surface energy leads to a separation of C–S–H particles, which leads to an expansion of the material. Wittmann, Powers and Bažant unanimously ascertained that the Bangham equation cannot approximate the volume change in RH range above 50% [9,12,14]. Nevertheless, they attributed this nonlinearity in swelling (by increasing water vapour pressure) to the disjoining pressure which was established by Derjaguin [15]. In a narrow space smaller than ten water molecules (about 2.6 nm) the inter-layer water is in a hindered desorption condition. The adsorbed water builds up a repulsive disjoining pressure. Conversely, the thickness of adsorbed water reduces and the disjoining pressure will depress during drying and the distance between two layers will be reduced. This results in a sudden increase of overall shrinkage in RH above 40%. In practice, the irreversible part of the concrete is predominant. The irreversibility of the shrinkage deformation arises mainly from the collapse of the small pores at RH > 45% and bridging effect of calcium ion (Si–O–Ca–O–Si) in C–S–H particles [16,17].

Autogenous shrinkage is defined as the isothermal macroscopic load-independent volume change of cement paste or concrete due to self-desiccation without moisture exchange to the ambient atmosphere [18]. In cement pastes with w/c-ratios < 0.40, consumption of water by the hydration results in a reduction of internal RH. As a consequence of the drop in RH, the attractive surface forces between C–S–H particles (mainly capillary and disjoining pressure) grow and the distance of the solid surfaces decreases [19,20]. Carbonation shrinkage is a result of the carbonation reaction in hydrated cement paste [21]. As a result of carbonation, the bulk density increases and the specific surface area (SSA) of hcp decreases [22]. At the same RH, carbonated hcp shows a lower

water content than in non-carbonated state. This reduction of internal RH due to carbonation reactions results in a volumetric carbonation contraction. Water release during carbonation reaction as well as volume reduction due to the dissolution of crystals of calcium hydroxide and deposition of calcium carbonate are known as the main mechanisms of carbonation shrinkage [7].

Shrinkage deformation of concrete can be influenced by the creep effect and stress loss due to relaxation of the hcp [23,24]. Furthermore, non-linear properties of interfacial transition zone (ITZ) as well as stress-induced cracks make the shrinkage behaviour of concrete a complex phenomenon.

### 1.3. Influence of ground limestone on the shrinkage of cement paste and concrete

Different factors regarding the quality of the ground limestone – either as aggregate or binder – influence the magnitude of the drying and autogenous shrinkage of hcp and concrete [7]. In cement paste and concrete containing high limestone contents, the amount and the chemical-mineralogical characteristics of limestone can have significant impacts on the shrinkage behaviour [25]. Espion investigated the drying shrinkage of concrete made of ternary cements. He concluded that the shrinkage of concrete with 30 wt.-% limestone and 50 wt.-% slag is lower than that of concrete with Portland cement [26]. Higher limestone content could result in a larger drying shrinkage deformation for self-compacting concrete with the same water content [27]. Esping showed that the higher BET-surface area of limestone leads to a higher autogenous shrinkage of self-compacting concretes [28]. Wang ascertained a reduction of about 10% in the drying shrinkage of concrete when 30 wt.-% of cement is replaced by limestone [29].

In an early study, Roper et al. investigated the influence of mineralogical characteristics of aggregate on the drying shrinkage of concrete [30]. In concrete made with high limestone contents, the clay-contaminated limestone can influence the shrinkage behaviour of concrete remarkably. Clay minerals can also enter into concrete through clay-contaminated aggregates or fillers which is more studied in the literature. Many fresh and long-term properties of concrete can be influenced by the type of clay mineral [31–33]. However, an increasing effect of clay mineral on the drying shrinkage of cement paste and concrete is reported by many authors [34–36]. Main action mechanism of swelling clay minerals is attributed to their higher disjoining pressure. The disjoining pressure in swelling minerals is affected by the surface charges and their interaction with balancing cations [37]. Norvell et al. observed that at a constant w/c-ratio, the drying shrinkage increases if montmorillonite clay is added to the mixture, whereas the influence of other types of clay mineral (illite and kaolinite) was not very remarkable [34]. Other studies correlated the drying shrinkage of concrete to the presence of clay minerals in the concrete aggregate, denoting a high BET-specific surface area [38,39]. Igarishi et al. studied the influence of the chemical-mineralogical properties of various aggregates (from different Japanese quarries) on the drying shrinkage at RHs between 0 and 98% [40]. They addressed that the shrinkage of aggregate at RHs between 60% and 98% can be attributed to the chlorite mineral, while the contribution of illite and sericite to length change of aggregate is very small.

The influence of alkalis on the drying shrinkage and hydration of hcp and concrete has been widely studied by various researchers [41–43]. Most of these works agree that an increase in the alkalinity of pore solution results in a growth in drying shrinkage, but there is still debate about the governing mechanisms [42]. Change in the vapour pressure and the disjoining pressure are known as the controlling mechanisms for higher shrinkage deformation at higher alkali concentration [37,44]. Furthermore, hydration products from the dissolution of C<sub>2</sub>S have a larger specific surface

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