



Effectiveness of Limestone Powder in Controlling the Shrinkage Behavior of Cement Based System: a Review

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Abstract

Limestone (LS) powder is commonly used in concrete from the last two decades as it is widely available and having low cost. Since, LS powder is mostly used as a replacement of cement for avoiding the CO₂ emission; however its incorporation in concrete may influence the shrinkage behavior of the cementitious system mainly by filler and nucleation effect. Therefore, in this paper, the effect of LS powder is reviewed in the light of literature studies to quantify its influence on the shrinkage behavior of cementitious system. The factors that influence the shrinkage behavior of cementitious system are the fineness (particle size), dosage, chemical-mineralogical effects of LS powder and w/b ratio of the mix. In general, high dosage of nano LS powder replacing cement content leads to reduction in the shrinkage behavior; however this high dosage causes nano LS particles to agglomerate which further reduces the inhibitory effect of LS powder on the shrinkage reduction.

Keywords Limestone · Shrinkage · Nano calcium carbonate · Filler · Particle size · Cementitious system

1 Introduction

From the last two decades limestone (LS) has been used to replace a part of the cement up to 5% by mass and has successfully implemented it as a part of the cement industry. Many researchers used LS powder in concrete as it is widely available and have low cost. The idea of using LS powder in cement came in from the oil shortage problems in 1970 to 1980. This led Canadian standards to allow 5% addition of LS in the cement since 1983 which was then adopted by the Brazilian standards (NBR-5732) in 1988. The motivation to control

greenhouse gases in cement production by means of LS powder started in 1990's. In 2000, European standards accepted the proposal of adopting LS in cement production followed by AASHTO M85 and ASTM C150 in 2007 and 2004, respectively [1]. Up till now, a large number of research results depict the use of LS less than 35% in cement production by Canada [2], Europe [3], United States [4] and china [5].

Shrinkage of cement based system occurs either due to the change in internal relative humidity or due to the chemical reactions. It cannot be avoided because of the chemical reaction of water with the cement due to which the resulting hydrates occupies less volume than the reactants. If there is excessive shrinkage then cracking will occur thus compromising durability of concrete. Factors contributing to the cracking potential of concrete structures are free shrinkage, reinforcement, size of the specimen and edge restraints [6]. Concrete mixes are designed to satisfy the requirement of strength and durability properties. These properties can be compromised if concrete is susceptible to cracking which is responsible for shrinkage to occur. To overcome the cracking phenomenon, many researchers adopted various effective strategies. Among these strategies, the use of LS powder has started to control the excessive shrinkage of cement mix. Since LS is a cheaply available mineral, it is used by many researchers with the aim of decreasing cement content and achieving enhanced

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engineering performance. In cement pastes, mortar and concrete, LS powder shows physical and chemical influences whereas physical influences involve filler effect, nucleation effect and dilution effect. The influence of LS powder is affected by its fineness, percentage of addition, rate of dissolution, polymorphs of LS powder and mineralogical composition of cement and additional supplementary cementitious materials (SCMs) [7–10]. The shrinkage of the cementitious system is influenced by the quality of LS powder incorporated as a part of the cement or as a fine aggregate [11]. However, when cement and concrete mixes contain high content of LS powder, shrinkage behavior is significantly influenced by the amount and chemical mineralogical characteristics of LS powder [12].

Up till now, many standards have been established on using LS powder in cementitious system and thus their corresponding research studies have been conducted. But still many of them are controversial regarding the shrinkage behavior of LS containing mixtures. Therefore, in order to use LS powder in a scientific manner, this review study is carried out to discuss the literature results based on the influence of LS powder on the shrinkage behavior of the cementitious system.

2 CaCO₃ as the Main Constituent of Limestone

LS can be formed from various minerals such as calcite, aragonite, vaterite and amorphous CaCO₃ [13]. CaCO₃ can also be artificially produced in the form of calcite, vaterite and aragonite by varying temperature and pressure while combining calcium and carbon dioxide [14]. Among these, calcite is mostly common and stable mineral which can be used in the formation of natural LS [15]. LS contains CaCO₃ as a major part with other clay minerals and alkali oxides as an impurities (a few percent) [16]. Therefore, this review study also discusses the effects of CaCO₃ in the form of nano CaCO₃ (NC) and micro CaCO₃ (MC) on shrinkage of the cementitious system.

LS is used in the cement based system at a macro scale (>1 mm) in the form of fine LS aggregate [17] or coarse LS aggregate [18, 19]. Besides, it is used at a micro scale (1 μm–1 mm) and at nano scale (<1 μm) as well. Incorporation of LS at different scales influences the shrinkage characteristics of cement based materials through physical and chemical effects. Mechanism of the former is by filling, dilution and nucleation effect. For example, using LS at macro scale is considered mainly to act as an inert filler in skeleton building of hardening cementitious composites, helping in the strength development. Micro LS powder and LS dust such as MC are considered to act as filler i.e. by filling the voids within the cement particles. Micro LS also influences the properties of cement based system through dilution, nucleation and chemical

effects. At nano scale, incorporation of LS in the form of NC powder acts more effectively than at micro scale [13]. Morphological structure of NC is in the form of white powder and scanning electron microscopy of NC shows its cubic crystalline shape [64].

3 Shrinkage of Concrete Containing LS Powder

The shrinkage of concrete is basically the variation in volume of a certain hydration product caused by evaporation of water, hydration of cement and also by carbonation [20]. However, it is a complex phenomenon influenced by many factors, including the constituents, temperature and relative humidity of the environment, the age when concrete is subjected to the drying environment and the size and shape of the structure or member [21]. The main action mechanisms of LS that influence the total shrinkage behavior of cementitious system in early ages are filler effect, dilution effect and nucleation effect as shown in the Table 1. These action mechanisms are evident in case of micro and nano LS, however, macro LS are commonly considered as inert filler [13]. It is also proved that these effects not only influence the shrinkage behavior but also influence the overall performance of cementitious system, as listed below.

- Early age compressive strength of cement mortar is enhanced due to the nucleation effect of the fine LS particles [22].
- Filler effect of LS particles causes a significant increase in the early age hydration process by providing nucleation sites for the hydrates [23, 24].
- 10% replacement of cement by LS particles shows similar or reduced early age cracking risk as compared to plain cement concrete [25].
- Addition of finely ground LS in cement mortars significantly promoted nucleation of hydrates due to which early age hydration and compressive strength of cement mortars increases as compared to control cement mortar having no finely ground LS [26].
- Addition of 10% LS content in concrete showed lower chloride ion permeability due to filler action of LS particles, as compared to concrete without LS [27].
- Initial and final setting times of cement pastes decreases with the increase in the LS amount, this is due to the filling action of fine LS particles (5 μm) in cement pastes [28].
- Interground LS cement mortar exhibited lower linear expansion due to dilution mechanism of LS particles, at any ages in comparison to Portland cement mortar [29].
- Replacement of 40% cement by LS in blended cement paste, causes reduction in the rate of heat dissipation in cement paste due to dilution effect of LS particles [30].

Table 1 Action mechanism of LS powder in cementitious system

Mix type	Shrinkage type investigated	Particle size of LS	Mechanism	Dosage	w/c	Addition pattern	Results
concrete	TS	finer than cement	filler effect	LS powder 10%	0.55	replacing cement	lowered total shrinkage [68]
cement paste	EAS	NC (15–40 nm) finer than cement	filling effect, nucleation effect	NC: 1%, 2% and 3%	0.45	replacing cement	1% NC dominantly decreased shrinkage; however shrinkage increased with the increase in LS content [69]
cement paste	EAS	NC (50 nm) finer than cement	filling effect	NC: 1% and 3%	0.45	addition to cement	shrinkage decreased with increase in NC [59]
normal grade concrete	DS	finer than cement	Nucleation effect, filler effect, dilution effect	4.5% to 12%	0.47 to 0.71	addition to cement during manufacturing	No significant effect on DS of Normal grade concrete using 12% limestone [40]
ultra high performance concrete	DS	MC (3µm) and NC (15–40 nm) finer than cement	Nucleation effect, filler effect, dilution effect	MC: 2.5%, 5%, 10%, 15% NC: 2.5%, 5%	0.28	replacing cement	DS was reduced by either 2.5% NC or 15% MC [41]
concrete	DS	less than 5 mm	mineralogical effect	LS 20,30,40,50,100	0.45, 0.55, 0.65	replacing sand	drying shrinkage decreased with LS content increase [42]
concrete	DS	finer than cement	filler effect	LS 15%, 25%, 35%, 45%	0.45–0.79	replacing cement	increasing the LS content reduced DS magnitude [10]
plain cement concrete	DS	micro LS powder finer than cement	dilution effect	10, 20, 30	0.4	replacing cement	DS decreased with increase in LS content [44]
mortar	DS, AS	finer than cement	filler effect, nucleation effect	LS powder 5%, 15% and 25%	0.47	replacing cement	AS increases at high LS content DS decreases at high LS content [47]
cement paste, concrete	DS, AS, CS	cement 8µm, LS (13.9, 5.3, 24.3, 8.8, 9.2, 6.9, 10.1, 7.3, 12.8, 9.7 µm)	chemical mineralogical effect	LS: 30%, 50%, 70%	0.35	replacing cement	Clay minerals and alkali oxides influence DS of concrete containing high LS content [16]
Self-compacting concrete	DS, AS	11 times finer than cement	filler effect, nucleation effect	Portland Cement 70%, FA 30, 25, 20, 15%, LS 0, 5, 10, 15%	0.4	replacing cement	FA reduced DS while slightly reduced by FA & LS. FA + 5% LS decreased AS significantly [49]
Self-compacting concrete	DS	finer than cement	filler effect, dilution effect	LS 66%	0.4, 0.46, 0.55, 0.6	Addition to cement	shrinkage increases with LS as compared to control mix [50]
Self-compacting concrete	DS, AS	coarser than cement	dilution effect	LS 12.5%, 25%	0.6	addition to cement	DS increased with LS content AS decreased with LS content [52]
cement paste	AS	NC (15–40 nm) finer than cement	Nucleation effect, dilution effect	NC: 1%, 2%, 3%	0.3	addition to cement	2% NC dominantly decreased AS however AS increased with increase in LS content [60]
ultra high performance fiber reinforced concrete	AS	cement 20.5µm fine LS 10.4µm coarse LS 16.6µm.	filling effect	Fine LS: 25%, 50%	0.32, 0.48	replacing cement	AS decreased with increase in LS content however coarse LS was more effective [61]

Table 1 (continued)

Mix type	Shrinkage type investigated	Particle size of LS	Mechanism	Dosage	w/c	Addition pattern	Results
high performance concrete	AS	LS (coarse and fine)	dilution effect	fine: 12%	0.357	replacing cement	Coarse LS dominantly reduced AS as compared to fine LS [22]
ultrahigh-performance concrete	AS	NC 15 to 80 nm finer than cement	filling effect, nucleation effect	NC: 1%, 2%	0.15, 0.18	replacing cement	AS increased with increase in NC content however lower at 0.18 than 0.15 w/c [64]
cement paste and mortar	CS	same as cement	nucleation effect	25%, 67%	0.4	replacing cement	CS increases with LS content [23]
cement paste	CS	cement 11 μm , LS 4 μm	nucleation effect	95%OPC+5% LS 65% OPC + 30% FA + 5% LS	0.5	replacing cement	increase in CS [1]

Where TS, EAS, DS, AS, CS and FA refers to total shrinkage, early age shrinkage, drying shrinkage, autogenous shrinkage chemical shrinkage and fly ash

- Initial porosity of cementitious system is modified by the filler effect, due to which water requirement decreases in maintaining constant workability [31].
- Later age (28 and 90 day) flexural and compressive strength of cement mortar and concrete containing LS filler as partial replacement of cement, decreases due to dilution effect [32].
- Dilution effect of LS stimulate early age hydration of cement [33] whereas filler effect of LS improve durability properties of concrete [34].

It is worth noticeable that various parameters of the system are influenced by these mechanisms because the action of these mechanisms is simultaneous and interrelated. Generally, filler effect and nucleation effect act together in comparison to nucleation effect [35]. Together these mechanisms influence the initial porosity and causes enhancement in the rate of hydration that is why the shrinkage behavior is influenced by the LS addition.

Shrinkage of concrete can occur in two different stages: early and later ages. The first stage (within the first 24 h) is defined as a duration at which the concrete started to set and harden. Second stage, on the other hand, refers to the age beyond 24 h. At early age, concrete is more prone to internal stresses due to lower strain capacity and cracks at this age are in the similar pattern as at later age. However, if cracks at early age are hairline micro cracks, the shrinkage at later age further opens those existing hairline micro cracks and compromising the durability. High risk of cracking potential may occur if the magnitude of free shrinkage at early age exceeds 1 mm/m which is almost 10 times the tensile strain of concrete [36]. This limit is in compliance with the guidelines of American Concrete Institute (ACI) which allows an expected shrinkage of 0.4–1 mm/m [11]. Shrinkage of cement paste or concrete may be classified into autogenous shrinkage, drying shrinkage, chemical shrinkage and carbonation shrinkage [11, 36]. Shrinkage at both stages mainly include autogenous shrinkage, drying shrinkage and thermal shrinkage which have overlapping results with different mechanisms. In long term, the carbonation shrinkage is also added which has an accumulated effect. A quick glance at differences is helpful in elaborating the mechanism and influential factors in various types of shrinkage in concrete containing LS which is discussed in the succeeding sections.

3.1 Drying Shrinkage

Drying shrinkage is caused by internal water evaporation from the capillary pores of the matrix due to the low external environmental humidity of the cement-based materials [37]. During moisture loss from the capillary pores, the surface tension rises due to increase in the curvature of menisci.

Subsequently, a negative pressure created due to the difference in the capillary pore pressure and atmospheric pressure. This negative pressure induces contraction in the volume of the concrete body, thus resulting in drying shrinkage [38, 39]. The development of drying shrinkage of concrete is lengthy relative to the autogenous shrinkage. Accurate measurement of drying shrinkage is a challenge as the autogenous shrinkage deformation of the sealed specimens should be separated from the total measured deformation of the concrete due to their distinct physical behavior. The drying shrinkage value measured by traditional method contains part of autogenous shrinkage; however, it is not a simple superposition, since the drying condition has a serious effect on the hydration of cement.

Drying shrinkage of cement based system depends upon the particle size and dosage of LS powder. For instance, one of the investigations [40] concluded that the incorporation of LS powder up to 12% into general purpose cement (GPC) results in drying shrinkage within 3.6% of the control samples. Besides, it was reported that cement containing 12% LS content has not affected drying shrinkage of normal-grade concrete. Nonetheless, smaller drying shrinkage strains were reported by some researchers with the addition of LS in the cementitious system. In one of the studies [41], a part of cement volume in UHPC was partially replaced with LS (NC and MC) in which 0%, 2.5% and 5% NC was added in combination with 15% MC while 0, 2.5, 5, 10 and 15% MC was added in combination with 2.5% NC. Lowest drying shrinkage was reported in two mixtures incorporating a combination of 15% MC and 0% NC or 2.5% NC and 0% MC, as shown in Fig. 1 (a, b). Likewise, when fine aggregate was replaced with LS, the later age drying shrinkage (at 180 days) of hardened concrete reduces considerably [42]. In another study [43], it was reported that by increasing LS content, the magnitude of long term drying shrinkage decreases. For instance, the ultimate drying shrinkage of cement paste decreases by increasing LC content as shown in Fig. 2. A decrease of 10 to 17% was reported when cement was replaced up to 45% by LS powder [10]. This was further supported by Wang et al., [44] using 10%, 20% and 30% LS powder in concrete mixes, indicated as L₁, L₂ and L₃. It was reported that long term drying shrinkage decreases with the increase in the dosage of LS powder at the end of two years. This decrease is attributed to two reasons: one is due to water to binder ratio [45]. The water to binder ratio of L₂ and L₃ is lower than L₁ and the reference mix having 0% LS content. Another reason is due to increase in the replacement ratio i.e. more cement particles were replaced by the LS powder thus resulting in less hydration products [46]. One of the studies [47] concluded similar results and it was reported that drying shrinkage strains of mortars decreases when LS powder is added up to percentage levels of 5, 15

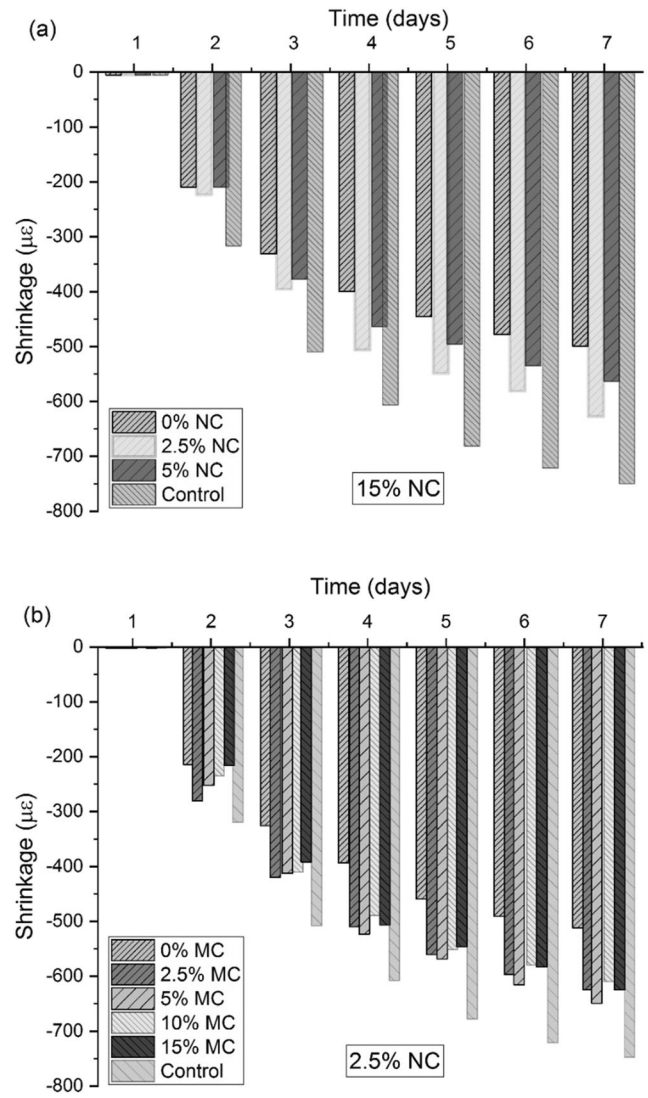


Fig. 1 Drying shrinkage of UHPC mixtures incorporating (a) 15% micro-CaCO₃ (b) 2.5% nano-CaCO₃

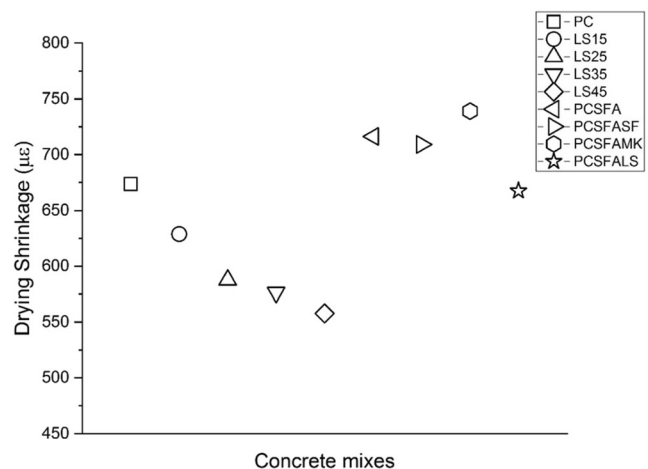


Fig. 2 Drying shrinkage of concretes having various contents of LS and other SCMs

and 25%. The drying shrinkage strains obtained after one year of observation were 367, 384, 305 and 325 μm for mortars containing 0, 5, 15 and 25% LS, respectively.

In many researches, LS powder is used along with other SCMs in which drying shrinkage of cement based materials was affected. In one of the studies [48], it is concluded that drying shrinkage of concrete reduces with the addition of 30% LS powder and 50% slag as compared to concrete mix containing cement only. In another study [49], mortar containing fly ash and 5% LS powder (finer than cement) induced a higher reduction in the drying shrinkage, however, the drying shrinkage was slightly increased when LS content was further increased up to 15%. The slight increase is because of the filling effect of LS powder making the pore structure much finer. Similarly, in case of binary cements, incorporation of LS powder causes the drying shrinkage to reduce, as depicted in Fig. 2 [10]. Such reduction in drying shrinkage is due to the fact that LS powder is less reactive than cement and other SCMs i.e. slag (S), fly ash (FA), silica fume (SF) and metakaolin (M). Therefore, LS is considered as inert filler replacing cement content and increases the relative w/c ratio, thus causing reduction in drying shrinkage.

Drying shrinkage of self-compacting concrete (SCC) at later age shows enhancement when LS content exceeds an average of 15%. This is verified by many researchers, for instance, one of the studies [50] concluded that at constant w/c ratio, drying shrinkage behavior of SCC mixtures shows an increasing trend with the increase in the content of LS powder. In another study [51], similar results were concluded with the increase in drying shrinkage when the LS powder content exceeds 10 to 15%. Similarly, in another study [52], fine aggregate was replaced with LS aggregates and reported enhancement in the drying shrinkage by increasing the LS content up to 25%. It was observed that the drying shrinkage of 25% LS concrete mixture was higher than 12.5% and 0% LS concrete mixtures by an amount of 18.1% and 50%, respectively. The enhancement in drying shrinkage with increasing LS content is due to the fact that LS particles tends to filling up the pores by making the pore structure more refined due to which capillary pressure develops with high amount of LS powder.

Apart from the particle size and dosage of LS, other factors that are responsible for altering shrinkage behavior are the chemical mineralogical properties of LS. In this regard, an extensive study was carried out by one of the researchers [16] using various LS types from different quarries with varying LS contents and varying fineness. The mixes developed were differentiated by size of the particles, density and chemical mineralogical composition. The results obtained were based on the influence of LS content, type and size of the particle on the drying shrinkage behavior. It was revealed that with increasing LS content, the drying shrinkage reduces. Similarly, drying shrinkage increases when different LS type

from different quarry was used in the cement mix. Besides, it was also concluded that the drying shrinkage of cement pastes were not significantly affected by the particle size of LS. Concerning chemical mineralogical effects of LS on the drying shrinkage behavior it was observed that by decreasing CaCO_3 content of LS, drying shrinkage reduces, which indicates the presence of impurities in LS that initiated the drying shrinkage. Similarly it was further observed that the presence of clay minerals contributes to increase in drying shrinkage. MB value signifies the content of clay minerals in LS and most importantly, shrinking clay minerals contribute to increase in drying shrinkage as high disjoining pressure is exhibited by the shrinking clay minerals. Besides, it is observed that the magnitude of disjoining pressure is mainly influenced by two key factors: one is the density of surface charge and other is the concentration and type of cations in the pore solution. These two factors alter the repulsive hydration components in the cement based system [53]. Besides it was examined in a study [16] that the drying shrinkage is increased by increasing the content of $\text{Na}_2\text{O}_{\text{eq}}$ which indicates that drying shrinkage is affected by alkali oxides and shrinking clay minerals independently. However, the rigorous influence of each parameter on the shrinkage behavior is still limited and needs to be further studied.

3.2 Autogenous Shrinkage

In general, the part of shrinkage which does not include any volume change due to loss or ingress of substances, temperature variation, and application of an external force and restraint can be considered as autogenous shrinkage. Unlike drying shrinkage, this type of shrinkage happens without any transfer of moisture from concrete to the environment. It usually occurs due to self-desiccation caused by the negative pressure within the capillary pores. Therefore, it is also referred to as self-desiccation shrinkage. The driving forces for this type of shrinkage are the pore structure i.e. pore distribution and moisture [54–56]. This type of shrinkage is commonly associated with low w/b ratio, specifically occurs in ultra-high performance concrete (UHPC) and high performance concrete (HPC) [37, 55, 57]. Owing to lower w/b ratio and finer pore structure, concrete is prone to early age cracking and thus compromising durability properties of concrete.

The autogenous shrinkage of concrete is a complex phenomenon influenced by many factors including: fineness of cement, cement type, SCMs, aggregate, fiber, water-to-cement ratio, admixtures, and curing. No uniform mechanism has yet explicated the autogenous shrinkage, even though; the capillary pressure was introduced as the main driving force [58]. Many researchers investigated the influence of LS powder on the autogenous shrinkage of cement based system. For instance, some studies [49, 59–61] concluded that the addition of LS into the cementitious system reduces the autogenous

shrinkage at early days and enhances its stability against cracking. In another study [43], 10% LS mortar specimens showed low autogenous shrinkage (187 μ strain) as compared to mortar specimen containing only cement (215 μ strain). In this case the former is cracked slightly longer than the latter. Similar conclusions were reported in [61] using LS powder in ultra-high performance fiber reinforced concrete (UHPC). It was observed that by replacement of cement with 25 to 50% LS powder, autogenous shrinkage was reduced by 9 to 32%. However, in another investigation [60] it was reported that autogenous shrinkage first decreases and then increases with the increase in NC content. For instance, autogenous shrinkage is reduced by 8.6%, 17.1% and 5.8% at the age of 168 h for 1%, 2% and 3% NC addition to the cement paste, respectively, as shown in Fig. 3. It is observed that the rate of reduction in autogenous shrinkage increases in case of 1% and 2% NC cement pastes while it decreases in case of 3% NC cement paste. The same conclusions were obtained in one of the studies [49] using 5% and 15% LS combined with fly ash in cement mortars. Lower autogenous shrinkage was found in cement mortar mixes containing 5% LS as compared to cement mortar mixes containing 15% LS. In the latter two cases [49, 60], the effect of LS content on autogenous shrinkage is not much obvious. This may be due to the fact that LS particles induces nucleation effect and increases calcium silicate hydrate (CSH) content. Besides, LS powder helps in improving the alignment of calcium hydroxide (CH) that causes gradual transitioning of the interface structure from plan to space which leads to the improved interface performance. Autogenous shrinkage reduction is exhibited because of the more space occupied by the non-directional arrangement of CH [62]. However, the incorporation of NC in the cement paste, an expansive compound is formed i.e. $C_3A-CaCO_3-11H_2O$, since this compound is characterized by some expansibility which is also responsible for the autogenous shrinkage. On the contrary, reduction in autogenous shrinkage is

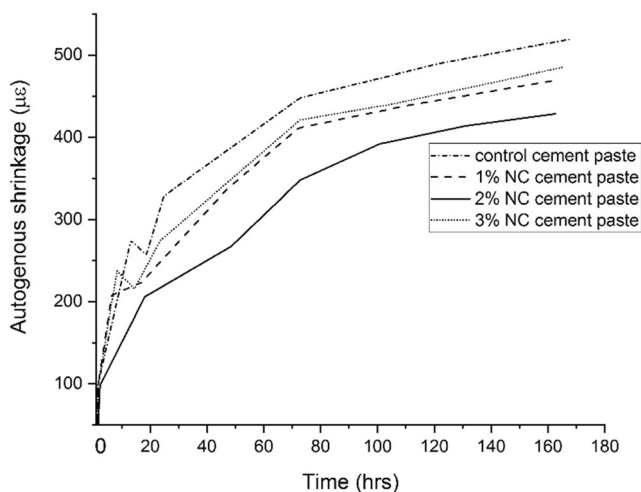


Fig. 3 Autogenous shrinkage cement based materials modified with NC

decreased when the NC content is increased up to 3% (as can be seen from Fig. 3), this is due to the large specific surface area of the NC content. Higher content of NC causes agglomeration of NC particles leading to non-homogeneous dispersion of NC particles in matrix and enhancement of air voids in the paste [63].

Moreover, addition of LS influences autogenous shrinkage of cementitious system based upon its fineness relative to cement. LS induce dilution effect within the cementitious system when the size of the LS particle is greater than or equal to cement particle. In dilution mechanism, LS particles tend to increase the interparticle spacing among the cement particles, releasing the capillary stresses due to which autogenous shrinkage is reduced. Autogenous deformation for mortars containing LS powder of varying fineness was performed by various researchers [22, 25] where the results indicated that the coarser LS mortar shows least autogenous shrinkage deformations than the fine LS mortar and cement mortar as shown in Figs. 4 and 5. Further, coarser LS powder showed longer cracking age and low potential to early age cracking as depicted in Figs. 6 and 7. Similarly, autogenous shrinkage of UHPC mixtures containing coarser LS particles showed 4 to 9% lower autogenous shrinkage deformations than that of mixtures containing finer LS particles [61].

Autogenous shrinkage of UHPC containing LS is also influenced by the content of LS as well as w/b ratio. In one of the studies [47] it was reported that with the increase in LS content, autogenous shrinkage increases at the same w/b ratio. However, others [16, 61, 64] reported increase in autogenous shrinkage with low w/b ratio. For instance, the autogenous shrinkage of concrete specimen containing 2% LS shows high autogenous shrinkage at w/b ratio of 0.15 as compared with the same type of specimen at 0.18 w/b ratio, as depicted in Fig. 8 [64]. Besides, it was concluded that lower w/b ratio is associated with high autogenous shrinkage and vice versa.

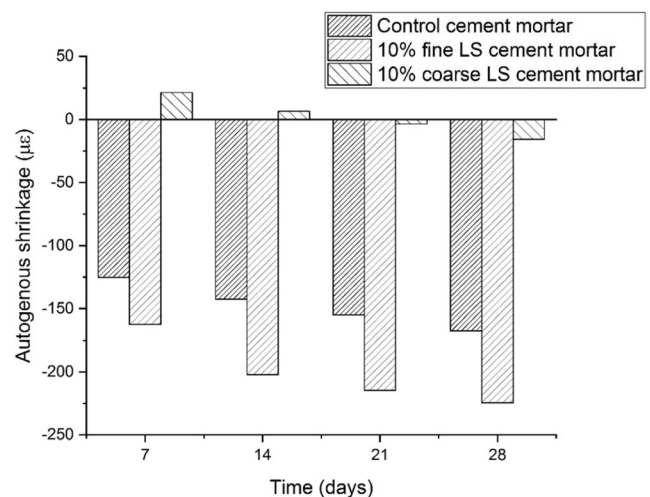


Fig. 4 Autogenous deformation of cement mortars with and without 10% LS content

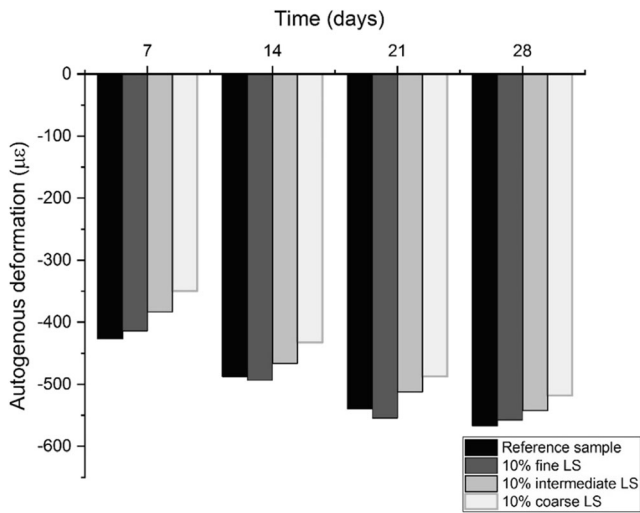


Fig. 5 Autogenous deformation

This statement was supported in one of the studies [61] stating that higher w/c ratio lead to lower autogenous shrinkage of UHPFRC mixtures containing LS content. The reason for increase in autogenous shrinkage at lower w/b ratio is due to the development of finer porosity within the cementitious system. This finer porosity increases curvature of the water meniscus and these menisci further pulls the pore walls with large compressive stresses due to which large autogenous shrinkage occurs. While in other case, the available free water helps in lowering the capillary stresses by increasing the relative humidity, that's why autogenous shrinkage is lower in case of w/b ratio of 0.18 and 0.48 in comparison with w/b ratio of 0.15 and 0.32, as can be seen in Figs. 8 and 9, respectively. Another possible reason is the nucleation effect of LS in cementitious system. LS induce nucleation effect by creating nucleation site

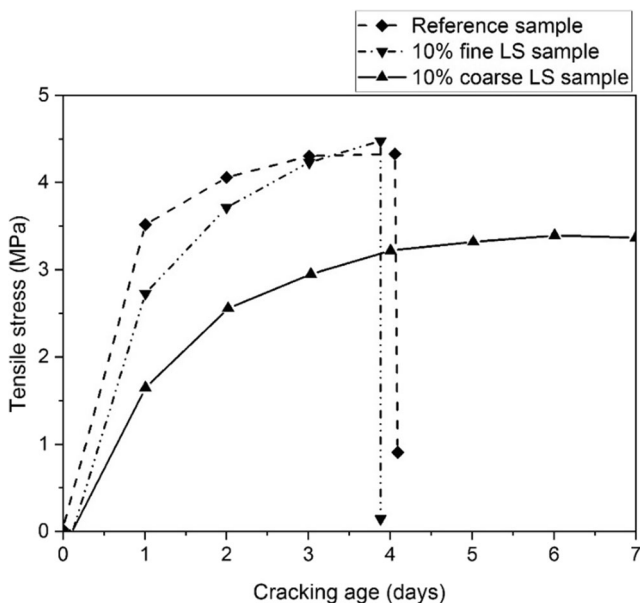


Fig. 6 Stress development

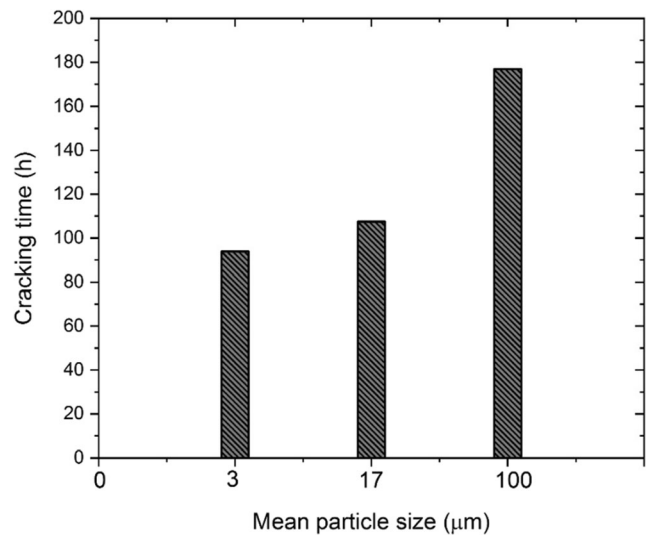


Fig. 7 Time of cracking vs. particle size of limestone cement mortars

for the products of hydration and fasten the hydration reaction. This leads to internal volume reduction and is attributed to internal chemical shrinkage. Therefore, it can be stated that higher internal chemical mechanism is accountable for higher autogenous shrinkage.

3.3 Chemical Shrinkage

Chemical shrinkage is generally the internal change in volume which occurs because of the chemical hydration reaction of cementitious materials. The chemical shrinkage is a result of the reactions resulting between cement and water, which lead to a volume reduction. The basic reactions of cement clinker are well understood and generally defined by four reactions of C₃S, C₂S, C₃A, and C₄AF. Each of these reactions, which require water for reaction, is exothermic and results in a decreased volume of the reaction products. This volume reduction, or chemical shrinkage, begins immediately after mixing

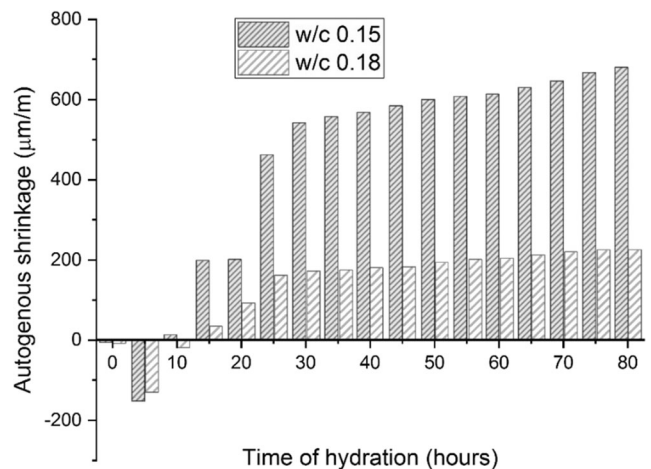


Fig. 8 Effect of w/b on the autogenous shrinkage of UHPC at 2% NC content

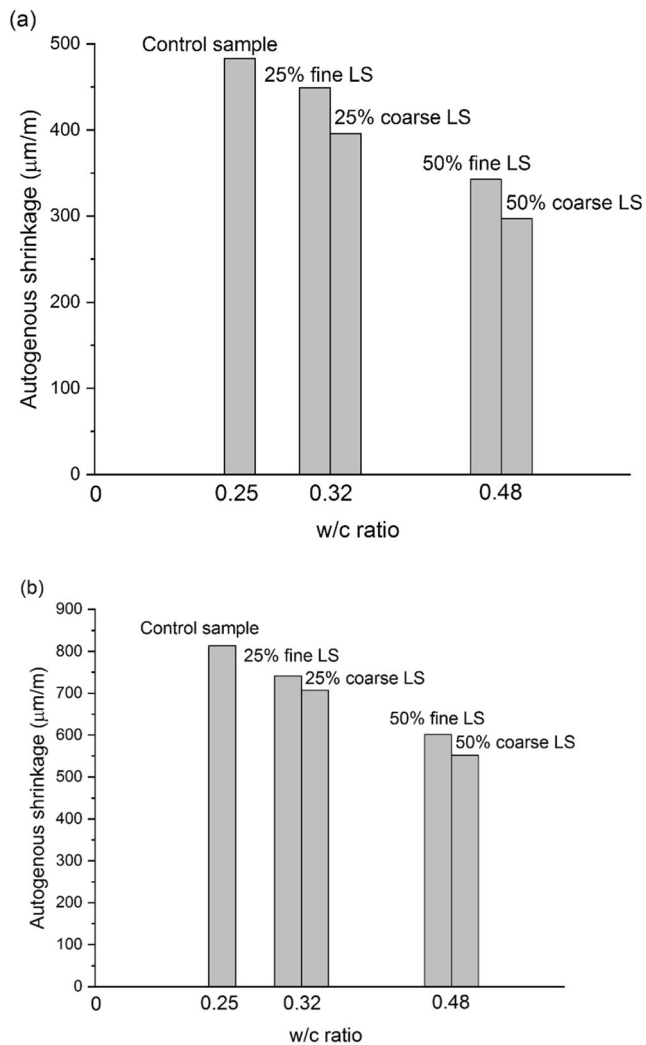


Fig. 9 Autogenous shrinkages at (a) 1 day and (b) 28 days

of water and cement and the rate is greatest during the first hours and days. Many researchers investigated the influence of LS powder on the chemical shrinkage of cement based system. For instance, one of the investigations [23] reported that the presence of LS increases the chemical shrinkage of cement paste in the very early hours of hydration and it induces filler effect and nucleation effect by producing nucleation sites for cement hydrates thus accelerating the chemical shrinkage. Another study [1] reported that by replacing cement with 5% LS content, chemical shrinkage of cement paste increased. It can be observed that chemical shrinkage of LS contained cement paste is slightly higher than OPC; however, it is lower than other SCMs. Further, it is indicated that due to the presence of LS, hydration products formed are more in amount or either different hydration products formed which are having less volume than the hydrates formed in usual cement paste having no LS content. Another study [35] concluded that the chemical shrinkage of Portland LS cements (PLCs) is higher than that of OPCs.

3.4 Carbonation Shrinkage

Carbonation shrinkage usually occur due to the interpenetration of atmospheric CO_2 and its reaction with CH. Due to carbonation reaction specific surface area of the cement paste decreases along with the increase in bulk density of the cement paste [65]. Carbonation shrinkage is activated by the contraction in volume because of the dissolution of crystals of CH, deposition of CaCO_3 and higher w/c ratio [11]. Carbonation and autogenous shrinkage occurs simultaneously, however the former is very limited in mixtures with low w/c ratios [66, 67]. In one of the studies [12], it was concluded that while increasing w/c ratio and LS content, corresponding carbonation shrinkage increases. Cement paste with 70% LS content exhibited highest carbonation shrinkage of 0.26 mm/m in comparison with the other cement pastes having low amount of LS. This amount of carbonation shrinkage contributes very little to the total shrinkage. Therefore, it is stated that such type of shrinkage is rarely found in gross sectional area of concrete members and may occur only in marginal areas which are considered negligible as compared to the gross sectional area. Carbonation shrinkage of LS containing cement pastes and concrete is very limited in the literature and it need to be investigated.

4 Analysis and Discussion

Early age shrinkage can be measured based on changes in volume and length. Regardless of test methods, the measured shrinkage is a combination of autogenous shrinkage, dry shrinkage, and chemical shrinkage. Early age shrinkage of cement based system is affected by the dosage and particle size of LS whether it may be added as an aggregate or as a binder to the mix, as summarized in Table 1. Regarding dosage, it is reported that reduction up to 19% occurs in the early age shrinkage of concrete by replacing cement with 10% LS powder [68]. While regarding particle size of LS, reduction in the early age shrinkage of cement paste occurs when cement is replaced with NC as shown in the Fig. 10 [69]. Results in [69] showed that reduction in the early age shrinkage of cement paste containing 1% and 2% NC, occurs up to one third and twice of cement paste (without NC) having w/c ratio of 0.45, respectively. Similarly, using NC (particle size of 50 nm) up to 1% and 3%, the shrinkage-compensating capabilities were more significant when 3% NC was added into paste in comparison to 1% NC as depicted in Fig. 11 [59]. Therefore, shrinkage of cement paste at early age decreases with the increase in content of NC up to 3%. It is indicated that raising the NC content, shrinkage of the cement paste decreases as NC is considered as an inert filler thus reducing the cement content and delaying the hydration reaction [10]. Besides, NC content induces an enhancement in the effective water to

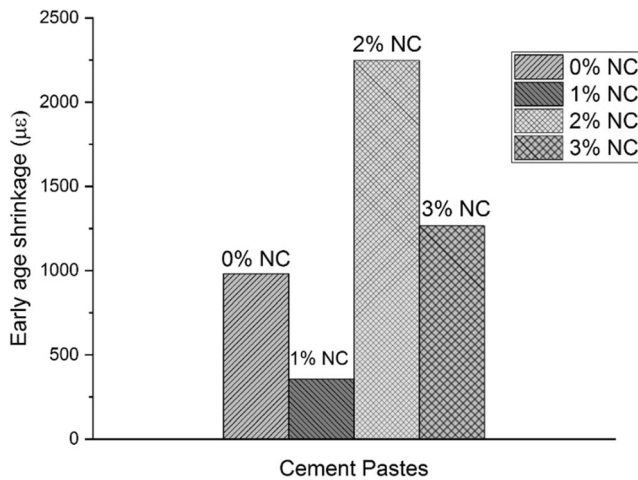


Fig. 10 Effect of NC content of the cement paste on the shrinkage at early age (12 h)

binder ratio and internal relative humidity, thereby reducing the shrinkage characteristic of cement paste [70]. According to one of the studies [59], reduction in shrinkage at early age is due to refining of the pores of cement paste with NC particles, which tends to act as an internal restraint against the shrinkage. In case of 1% NC cement paste, NC played a role of filling rather than promoting the hydration reaction. Whereas, in case of cement pastes containing 2% NC, nucleation is induced due to which hydration rate become accelerated which leads to the enhancement in the content mass of the calcium silicate hydrate (CSH) gel and density of cement paste. These factors lead to higher shrinkage of 2% NC cement paste as shown in Fig. 10. While in case of 3% NC, more cement particles are replaced by the NC content, hence, reducing the early shrinkage [69].

Shrinkage behavior of cement paste is also influenced by the chemical mineralogical effect of the LS [16]. For instance, one of the studies [71] indicated that shrinkage of concrete enhances when the incorporated LS has higher BET-surface

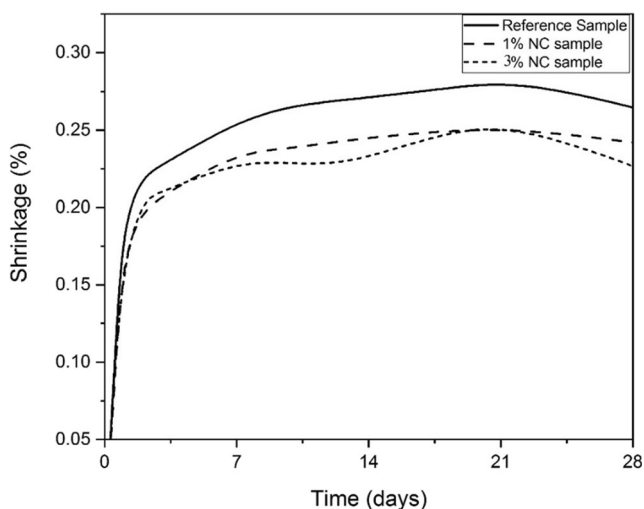


Fig. 11 Influence of NC on the shrinkage of cement paste

area. Moreover, shrinkage of concrete is highly influenced by the clay content of LS when concrete contains high LS content. Drying shrinkage of cement paste is enhanced by raising the clay minerals [53, 72–74]. Besides, different type of clay minerals influences other fresh and long term characteristics of cement based system [75–77]. Similar effect of alkalis on the shrinkage behavior of cement paste is studied by different researchers [16, 78, 79]. To reduce the shrinkage strains due to high alkali contents, disjoining pressure and changing vapor pressure are regarded as the controlled mechanisms [53, 80]. However, most of the authors concluded that higher alkalinity of the pore solution leads to higher drying shrinkage [81], but still further research is required to elaborate their governing mechanism.

In the literature, this is much highlighted that particle size of LS has a significant role in altering the shrinkage behavior of cementitious system. When sizes of the LS particles are finer than the cement particle, the filler, dilution and nucleation effects are more effective as compared to the particle size coarser than the cement particle [19]. Therefore, in order to obtain optimum content of both finer and coarser size LS powder, lesser finer LS powder will be used as compared to coarser LS powder. For example, lowest drying shrinkage is obtained either with 2.5% NC or with 15% MC in UHPC, as shown in Fig. 1 [41]. With the finer LS particles, the interparticle spacing reduces due to which the initial porosity decreases and results in higher autogenous shrinkage. Whereas, with the coarser LS particles, interparticle spacing increases due to which the capillary stresses reduces and results in lower autogenous shrinkage, as can be seen from Figs. 4 and 5 [22, 25, 82]. However, when the particle size of LS is same as that of cement, then LS particles exhibit dilution effect due to which effective w/c is increased. In the latter case, the occurring chemical shrinkage per unit volume is reduced [82]. Moreover, in some studies, finer LS powder as in case of NC causes autogenous expansion in the very early hours of hydration process, as can be seen from Fig. 8. This expansion may be regarded due to thermal expansion of hydration products, since energy is released during cement hydration reaction [64]. Besides, NC reacts with tricalcium aluminate (C_3A) forming calcium carboaluminate which is expansive in nature [60].

5 Conclusions

The above discussion may be concluded as below:

- (i). Influence of artificially made $CaCO_3$ and LS from the quarries on the total shrinkage behavior of cement based materials is slightly different from each other. It is due to the reason that LS contains a few percent of other impurities in the form of alkali oxides and clay minerals.

- (ii). Total shrinkage shows decreasing trend towards the influence of both dilution and filler effect of LS powder whereas, the influence of nucleation effect cause enhancement only in autogenous shrinkage.
- (iii). Apart from action mechanism of LS as a dilution, filler and nucleation effects, associated chemical mineralogical effect of LS also influences the shrinkage behavior of cementitious system. It is revealed that the presence of clay minerals and alkali oxides also contributes to the enhancement in drying shrinkage.
- (iv). Incorporating highly fine LS content in the form of NC to the cement mix, exhibit inhibitory effect on the reduction of shrinkage. This is due to the fact that highly fine LS content possess large specific surface area and cause agglomeration.
- (v). Addition of LS powder when increased beyond an average of 15%, the total shrinkage increases due to finer pore structure and higher capillary stresses.
- (vi). Coarser LS particles significantly reduce the autogenous shrinkage. As finer LS particles relative to cement particles, decrease the initial porosity and leads to higher capillary stresses while coarse LS particles relative to cement particles, causes increase in the initial porosity and reduces the capillary stresses.
- (vii). Addition of LS in the cementitious system causes enhancement in chemical shrinkage in the initial hours due to the filler and nucleation effect of LS particles.
- (viii). Increasing the LS content causes enhancement in carbonation shrinkage, however, this type of shrinkage imparts very little to the total shrinkage.

It is suggested that using high effective w/b ratio, selection of coarser LS powder and proper dosage of LS content can efficiently mitigate the shrinkage of cementitious system.

5.1 Research Needs

- (i). In the literature, effect of LS on long term drying shrinkage was mainly focused while there is limited literature on the early age drying shrinkage and need to be investigated.
- (ii). In view of LS addition into the cementitious system interrelated behavior of each shrinkage type is not discussed in the literature and requires further study to understand the overall mechanism of shrinkage behavior on cementitious system containing LS.
- (iii). Apart from filler, dilution and nucleation effect of LS powder on shrinkage behavior, study of chemical-mineralogical effect of LS on shrinkage behavior is limited. Therefore, in depth investigation is required to quantify the effect of each parameter of LS such as clay minerals and alkali oxides on shrinkage behavior.

- (iv). Besides, there is a limited study on chemical and carbonation shrinkage of cementitious system containing LS and needs to be further elaborated.

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