混凝土外加剂及其应用技术论坛2019年会

2019

聚羧酸系高性能减水剂及其应用技术新进展—2019

Recent Advances in Polycarboxylate Superplasticizer and Application Technology-2019

混凝土外加剂及其应用技术论坛　编

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前　言

聚羧酸系高性能减水剂是一种新型、绿色环保的高性能减水剂，与传统减水剂相比，其具有分散性强、减水率高、混凝土的坍落度经时间损失小等显著的综合技术优势，对提高混凝土性能、保证工程质量发挥了十分重要的作用，成为国家重点、重大工程中首选外加剂，其使用量持续快速增长。随着聚羧酸系高性能减水剂及其应用技术的不断发展，相继出现了早强型、缓凝型、缓释型、防冻型和减缩型等功能性聚羧酸系高性能减水剂，满足了混凝土施工及工程应用的需要。

为了更好地总结聚羧酸系高性能减水剂的国内外研究成果与其工程应用经验，促进聚羧酸系高性能减水剂研究的技术创新和技术发展，进一步推动聚羧酸系高性能减水剂及其应用技术的可持续性发展，2019 年 4 月 24—26 日在重庆举办“第七届聚羧酸系高性能减水剂及其应用技术交流会”暨“混凝土外加剂及其应用技术论坛 2019 年会”。

本次会议征集到学术论文 100 余篇，经会议学术委员会专家审核，择录了 97 篇论文，经编辑正式出版名为《聚羧酸系高性能减水剂及其应用技术新进展—2019》的论文集。本论文集内容涉及聚羧酸系高性能减水剂国内外研究进展与发展趋势、理论研究、最新制备技术、在各类水泥混凝土制品与工程中应用技术新进展及其案例分析、相关检测及标准；聚羧酸系高性能减水剂用功能性材料的理论研究与应用技术；其他混凝土外加剂的应用技术及机理研究等。论文集学术水平较高、内容较丰富、涉及面较广，具有一定学术参考价值，为读者提供了大量的技术资料。

由于时间与水平有限，论文集中难免有不妥之处，谨请读者予以指正。

混凝土外加剂及其应用技术论坛

2019 年 3 月

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综述与基础理论

Non – Classical Nucleation Mechanism of C – S – H

Prof. Johann Plank
(Chair for Construction Chemistry, Technische Universität München)

ABSTRACT: Calcium silicate hydrate (C – S – H) presents the main component in hardened cement and is responsible for its principle properties such as compressive strength, brittleness etc. Currently, its formation is thought to start with nanofoils which then grow into the larger, well – known needles. Here, the early nucleation and crystallization of C – S – H precipitated from aqueous solutions of $Ca(NO_3)_2$ and Na_2SiO_3 was investigated. It was found that in the absence of PCE, globular nanoparticles of C – S – H with a diameter of ~ 50 nm are observed. Thereafter, within an hour the globules completely convert to the well – known nanofoils of C – S – H with ~ 150 nm length following a non – classical nucleation mechanism. In the presence of a PCE copolymer, the initial globules show a core – shell morphology, presumably with PCE polymer as shell (thickness 4 – 8 nm) which delays the conversion to the nanofoils for several hours. Apparently, the PCE layer effectively shields the C – S – H droplets and strongly delays transformation from the C – S – H globules to the nanofoils. ^{29}Si MAS NMR spectroscopy revealed that in the C – S – H globules, the silicate chains are slightly branched (Q^1, Q^2, Q^3 species) whereas in the C – S – H nanofoils only chains of silicate (Q^1, Q^2 species) occur.

1 INTRODUCTION

Calcium silicate hydrate or C – S – H presents the main hydration product of ordinary Portland cement. C – S – H is generated from the hydration of the tricalcium silicate (C_3S) and dicalcium silicate (C_2S) phases via a dissolution – precipitation mechanism. It presents the binding phase which is responsible for the strength properties and durability in hardened cement. Generally, C – S – H exhibits low crystallinity and in hardened cement typically exhibits a Ca/Si molar ratio of 1.6 – 1.8. The layered structure of C – S – H consists of linear silicate chains which are aligned in "dreierketten" sequences and share oxygen atoms with calcium ions in plane.

The nucleation and crystallization of inorganic minerals is described by two theories. The first, classical nucleation theory is based on the formation and growth of nuclei. The second, non – classical nucleation concept presents that the morphology of the precritical clusters can differ significantly from that of the final bulk crystal. There, an amorphous intermediate (e. g. liquid droplets, amorphous nanoparticles) subsequently crystallizes to form the stable crystalline product.

Polycarboxylate (PCE) superplasticizers are known as high range water reducing admixtures

for concrete. PCEs improve the rheology via an electrosteric dispersing effect. The structure of anionic comb – like PCE copolymers consists of carboxylate anchor groups at the backbone which are negatively charged and responsible for the adsorption onto the positively charged surface sites of cement particles and hydration products like ettringite. While the non – ionic side chains of PCEs are made from polyethylene glycol (PEG) which is accountable for the dispersing ability via a steric hindrance effect.

Synthetic C – S – H – PCE nanocomposites are well – known seeding materials which can enhance the early strength of Portland cement and blended cements. They consist of C – S – H nanofoils which are stabilized by PCE copolymers adsorbed onto the positively charged surfaces of C – S – H. An ultra – small size of the C – S – H seeds is required to achieve a maximum seeding effect on cement hydration and consequently, a much enhanced early strength development of concrete. However, the initial nucleation and crystallization of C – S – H in the presence of PCE is still not well understood.

In this study, the very early nucleation and subsequent crystallization of C – S – H (5 min – 48 h) precipitated from $Ca(NO_3)_2$ and Na_2SiO_3 solutions at a Ca/Si ratio of 1.0 in the absence and presence of low and high concentrations of an IPEG – PCE superplasticizer was investigated by capturing the initial precursors of C – S – H via transmission electron microscopy (TEM). Additionally, their nanostructures were characterized via XRD and ^{29}Si MAS NMR spectroscopy.

2 Materials and Methods

2.1 Raw materials

The starting materials used in the synthesis of C – S – H were $Ca(NO_3)_2 \cdot 4H_2O$ (PanReac AppliChem, Germany) and $Na_2SiO_3 \cdot 5H_2O$ (VWR Prolabo BDH Chemicals, Germany). As PCE superplasticizer, a commercial isoprenyl oxy poly (ethylene glycol) based superplasticizer (IPEG PCE) (Sunrise Co., Ltd., Shanghai, China) was employed in the synthesis and its solid content was 40% by weight. The chemical structure of this PCE polymer is presented in Fig. 1 and its molecular properties and anionic charge amount are listed in Table 1. The pH value of the PCE solution was adjusted by using NaOH (Merck KGaA, Germany).

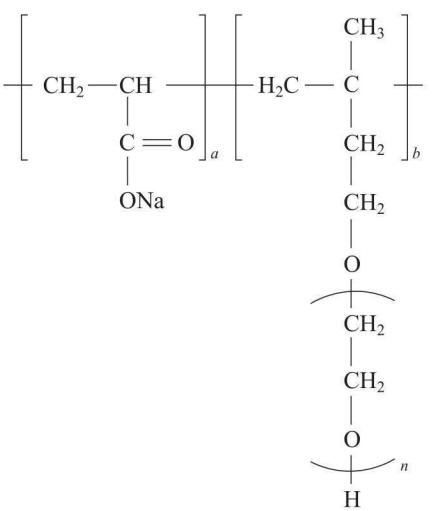

Fig. 1 Chemical structure of the isoprenyl oxy poly (ethylene glycol) (IPEG) based PCE superplasticizer used in the study.

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Table 1 Molecular properties and specific anionic charge density of the IPEG PCE sample.

M_w/ ($g \cdot mol^{-1}$)	M_n/ ($g \cdot mol^{-1}$)	PDI (M_w/M_n)	Specific anionic charge amount at pH 11.7/($\mu eq \cdot g^{-1}$)
35 100	15 700	2.2	2 750

2.2 Preparation and characterization of C – S – H

C – S – H and the C – S – H – PCE nanocomposites were prepared by the co – precipitation method. Aqueous $Ca(NO_3)_2$ and Na_2SiO_3 solutions were combined in water or IPEG – PCE solution to obtain either pure C – S – H or the C – S – H – PCE nanocomposites. The initial molar ratio of CaO/SiO_2 based on the starting materials was 1.0. Two different PCE concentrations, namely 2.7% and 6.7%, were used in the synthesis. First, 0.35 g or 0.90 g of the IPEG – PCE solutions were diluted with 5 mL of water resulting in 2.7% or 6.7% PCE solutions, respectively, which were adjusted to pH = 8.5 ± 0.1 by using aqueous 30% NaOH. Next, solutions of 0.35 g (1.5 mmol) of $Ca(NO_3)_2 \cdot 4H_2O$ dissolved in 5 mL of water and 0.32 g (1.5 mmol) of $Na_2SiO_3 \cdot 5H_2O$ in 5 mL of water were prepared at room temperature. After that, both solutions were added to water or the PCE solution within 5 seconds while stirring at 20 ℃. Morphologies of the resulting C – S – H and C – S – H – PCE respectively were observed over time after 5 min, 15 min, 1 h, 2 h, 4 h, 24 h and 48 h via TEM microscopy.

TEM micrographs of the C – S – H and the C – S – H – PCE samples were collected on a JEOL JEM 2011 instrument (JEOL, Japan) equipped with a LaB_6 cathode. Isopropanol suspensions of the C – S – H samples as prepared were placed on a 300 mesh Cu grid with carbon support films (Quantifoil Micro Tools GmbH, Germany) with a plasma – treated surface.

Powder X – ray diffraction (XRD) patterns were obtained from a BRUKER AXS D8 diffractometer (Karlsruhe, Germany) with *Bragg – Brentano* geometry working at 30 kV and 35 mA with Cu Kα radiation between 4.0° and 60° 2θ.

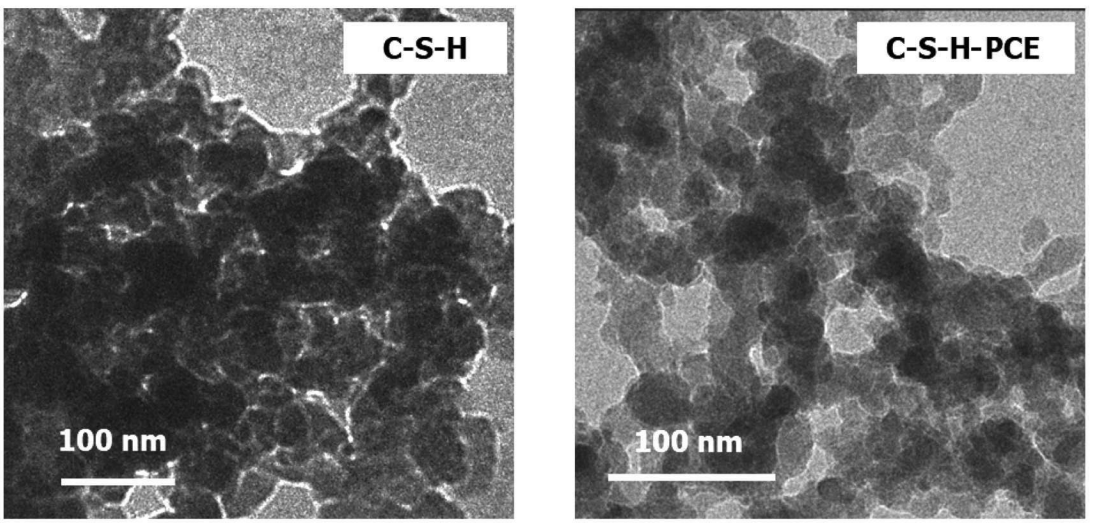
The silicate species present in the synthesized C – S – H and C – S – H – PCE nanocomposites was characterized by ^{29}Si MAS NMR spectroscopy using a Bruker Avance 300 MHz instrument operating at a resonance frequency of 59.595 MHz. The powder samples were filled into a 7 mm zirconia rotor and spun at 5 kHz. All spectra were recorded with a relaxation delay of 45 s, and tetrakis (trimethylsilyl) silane was used as external standard.

3 Results and Discussion

3.1 Initial nucleation of C – S – H

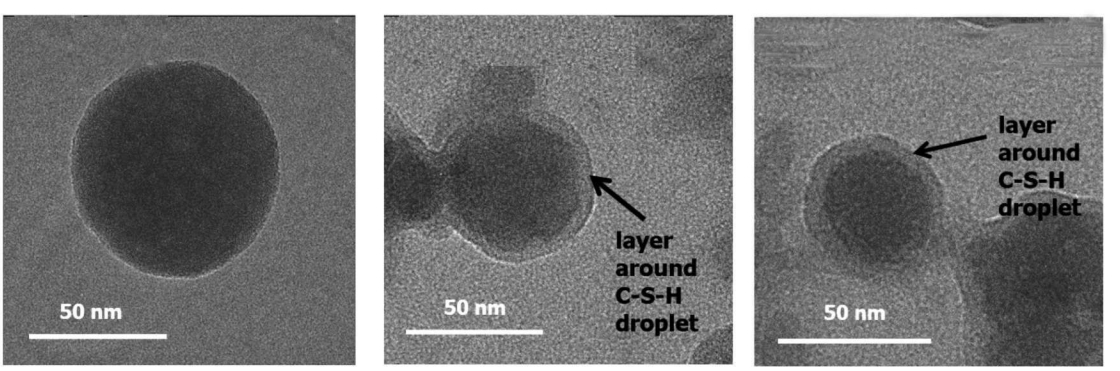
The early nucleation and crystallization of C – S – H synthesized from aqueous solutions of $Ca(NO_3)_2$ and Na_2SiO_3 in the absence and presence of an IPEG – PCE copolymer was observed via TEM imaging. After 5 minutes of reaction (Fig. 2), pure C – S – H as well as C – S – H – PCE

particles exhibit globular morphology, with diameters in the range of 40 – 60 nm. Most interestingly, a thin layer surrounding the C – S – H droplets was observed on the C – S – H – PCE precipitates, resulting in a core – shell structure (Fig. 3). The layer thickness of the C – S – H particles precipitated at low concentration of the IPEG – PCE solution (2.7%) was found at 3 – 4 nm (Fig. 3 (b)) while at high PCE concentration (6.7%), the thickness of the PCE shell was determined at 6 – 8 nm (Fig. 3 (c)).



(a) (b)

Fig. 2 TEM images of C – S – H (a) and C – S – H – PCE (b) precipitates after 5 min of ageing.



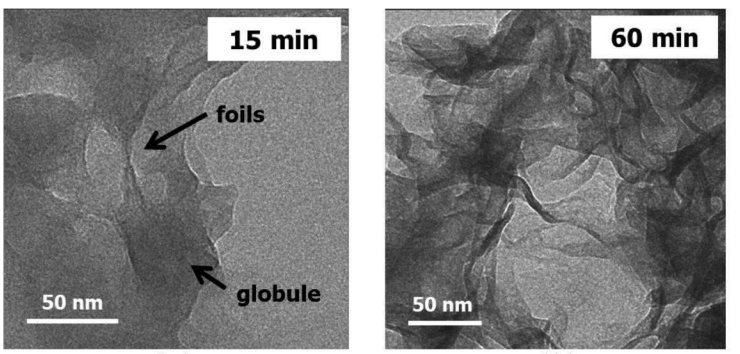
(a) (b) (c)

Fig. 3 High resolution TEM images of C – S – H droplets formed in (a) the absence and presence of an IPEG – PCE polymer at (b) 2.7% and (c) 6.7%.
(a) C – S – H; (b) C – S – H – PCE 2.7%; (c) C – S – H – PCE 6.7%

3.2 Conversion of C – S – H globules to nanofoils

Appearance of the initially globular C – S – H and C – S – H – PCE precursors was monitored

over time via TEM imaging. For the pure C – S – H, the transformation from globular to foil – like morphology had already started 15 minutes after the $Ca(NO_3)_2/Na_2SiO_3$ solutions had been combined. After 1 hour, the C – S – H globules had completely disappeared while a network of C – S – H nanofoils with lengths of ~150 nm and a thickness of ~5 nm was found (Fig. 4).



(a) (b)

Fig. 4 TEM images of C – S – H particles after 15 (a) and 60 min (b) of crystallization.

However, TEM imaging of the C – S – H – PCE precipitates revealed a delayed transformation from the initial globules to the nanofoils. At low PCE concentration (2.7%), beginning conversion was evidenced after 2 h by the appearance of first needle – like C – S – H crystallites. Furthermore, after 4 h the globules had completely transformed into nanofoils (Fig. 5 (a)). At high PCE concentration (6.7%), a strongly delayed conversion from the C – S – H globules to the nanofoils was observed (Fig. 5 (b)). Even after 4 h, still only C – S – H – PCE globules were present while after 48 h of ageing a mixture of globules and foils with lengths of 30 – 50 nm (and thus much smaller than for pure C – S – H) were found. This effect presumably is owed to a higher amount of PCE adsorbed which leads to a thicker polymer layer on the C – S – H particles.

The results suggest that, following a non – classical nucleation mechanism, early on C – S – H is formed as a metastable droplet which then transforms to the thermodynamically more stable, foil – like morphology. The IPEG – PCE delays the conversion to the nanofoils significantly.

3.3 Structure of early C – S – H

The XRD patterns of pure C – S – H synthesized at various ageing times are shown in Fig. 6. Immediately after precipitation and nucleation (0 and 5 min), a broad peak indicating a highly disordered structure was detected, suggesting amorphous character. While at 60 min of ageing, the diffraction pattern of semi – crystalline C – S – H (I) constituting an imperfect version of 1.4 nm tobermorite was clearly observed. The main hk0 reflections (100, 110, 200, 020) appear at 16.7°, 29.0°, 31.9° and 49.7° 2θ, respectively. Moreover, the 002 reflection can be found at 7.2° 2θ which signifies a d spacing between the silicate layers of 1.24 nm.

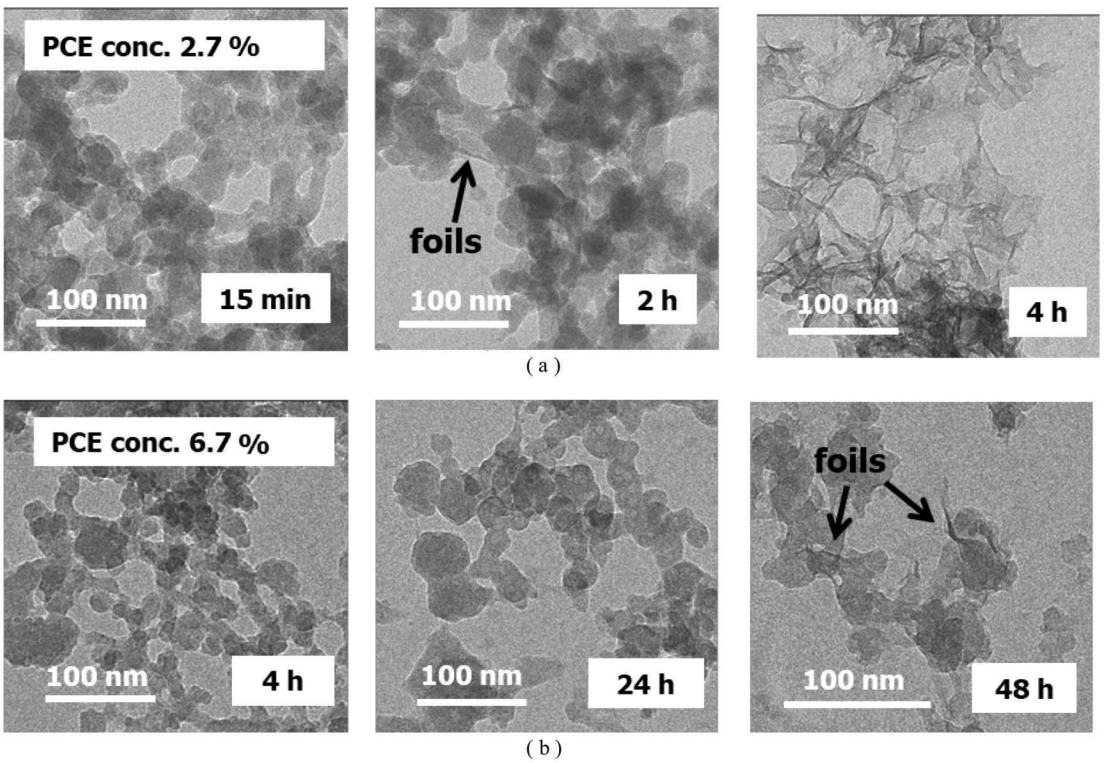


Fig. 5 TEM images of C – S – H precipitated in the presence of an IPEG – PCE polymer at 2.7% (a) and 6.7% (b) concentration at various ageing times.

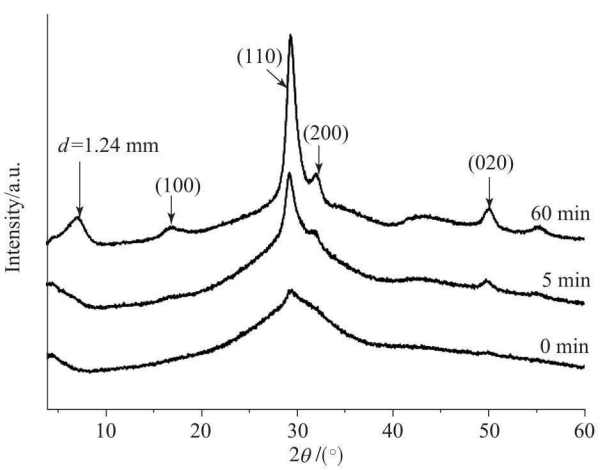
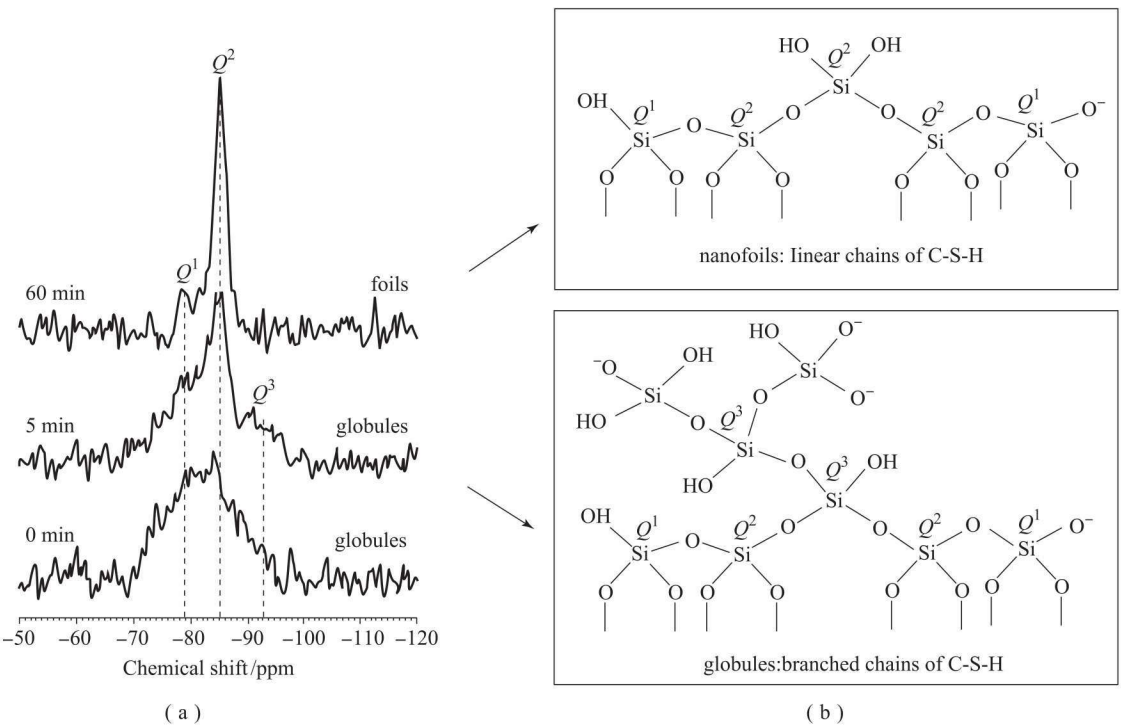


Fig. 6 XRD patterns of synthesized pure C – S – H obtained at 0, 5 and 60 min of crystallization, respectively.

The ^{29}Si MAS NMR spectra of pure C – S – H obtained from the precipitation are shown in Fig. 7. At very early crystallization of 0 and 5 min, the spectra show a broad peak characteristic for end – chain (Q^1, $\delta = -79$ ppm), chain member (Q^2, $\delta = -85$ ppm) and branching site (Q^3, $\delta = -93$ ppm) silica tetrahedra. However, after 60 minutes of ageing, the branching units have

disappeared, suggesting that only linear silicate chains are present in the C – S – H nanofoils. This result supports that the C – S – H globules obtained at very early crystallization exhibit branched silicate chains while the C – S – H nanofoils resulting from the conversion of the globules showed only linear silicate chains.



(a) (b)

Fig. 7 ^{29}Si MAS NMR spectra of pure C – S – H precipitates obtained at 0, 5 and 60 min of ageing (a) and illustration of structural model of linear and branched silicate chains (b) present in the globular C – S – H particles and the C – S – H foils, respectively.

4 CONCLUSION

Theinitial nucleation and crystallization of C – S – H prepared by co – precipitation from $Ca(NO_3)_2$ and Na_2SiO_3 in the absence and presence of an IPEG – PCE superplasticizer was studied. It was found that initially a metastable precursor of C – S – H presenting globular morphology is formed which later converts to the C – S – H nanofoils following a non – classical nucleation mechanism. The presence of the PCE delays the conversion from globular to nanofoil – like C – S – H for several hours because of a layer surrounding the globules which presumably consists of PCE polymer. At high PCE concentration, transformation to the C – S – H nanofoils is strongly delayed for several days due to the thicker layer of adsorbed PCE polymer coating the globular C – S – H particles.

The globular precursor of C – S – H exhibits a highly disordered structure containing branched

silicate chains. Whereas the C – S – H foils formed after the conversion show a layer structure of semi – crystalline C – S – H containing non – branched silicate chains.

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