iJRASET
International Journal For Research in
Applied Science and Engineering Technology

INTERNATIONAL JOURNAL FOR RESEARCH
IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 11 **Issue:** VI **Month of publication:** June 2023

DOI: https://doi.org/10.22214/ijraset.2023.49886

International Journal for Research in Applied Science & Engineering Technology (IJRASET)
ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 7.538
Volume 11 Issue VI Jun 2023- Available at www.ijraset.com

Influence of Hydrothermal Curingon Micro Structure and Mechanical Properties of Ultra-High-Performance Concrete

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Abstract: Several studies show that hydrothermal treatment can further improve the excellent properties of UHPC in terms of mechanical strength and durability. The hydrothermal treatment previous studies accredited it mostly to the formation of tobermorite. In the presented study hydrothermal treatment of UHPC samples was systematically varied and thephase formation analysed related to the strength development of a reference sample cured for 28 days in water. For the hydrothermal treatment already heating the specimens up to 185 °C in saturated steam followed by an immediate-cooling leads to a substantial increase in compressive strength. Pre-storage time did not affect the result as far as a minimum of several hours is guaranteed. The improved performance is due to an increase in the pozzolanic and hydraulic reaction. Surprisingly, tobermorite was only found within a very thin layer at the surface of the sample, but not in the bulk. Sulphate and aluminium stemming from the decomposition of the ettringite are bound in the newly formed phases hydroxylellestadite and hydrogarnet.

I. INTRODUCTION

Ultra-high-performance concrete (UHPC) is a new generation of high-performance concrete with extremely high strength, ductility, and durability. This type of concrete is generally considered to be characterized by a compressive strength above 150 MPa, fracture energy inthe range of 1200–40000 J/m2, a Young 's modulus between 50 and 60 MPa, and an ultimatetensile strain of 1%. The term UHPC is commonly used to describe the cement-silica fume mixture with an extremely low water/cement ratio and ultra-fine silica sand (0.15–0.6 mm) as a replacement for ordinary sand. In fact, UHPC should be considered as a type of mortar, rather than 'concrete', because it does not contain coarse aggregates. However, the term 'concrete', rather than 'mortar', has been in wide use due to the use of fibres used to enhance ductility and energy absorption capacity of this type of concrete.

The mechanical properties of UHPC might be improved by manipulating its particulatehomogeneity, porosity, and microstructure that can, in turn, be achieved by removing coarseaggregates and reducing the water/cement ratio to reduce the CaO–SiO2 content of the mixture.

Moreover, the maximum hydration of UHPC ranges from 30 to 55% due to its low w/c ratio. In addition, these materials can not only fill the larger pores among cement particles but are also able to reduce cracking due to hydration heat, thereby, creating a denser concrete matrix. The mechanical properties of cement-based materials depend on their chemical composition, microstructure, aggregate properties, and the bond between the cement matrix and aggregate.

Due to its desirable properties, UHPC has nowadays found wide applications in manufacturingprestressed and prefabricated concrete members as well as building nuclear installations, high-rise buildings, bridges, and certain structural elements of low thickness.

II. LITERATURE REVIEW

1) *Ultra High-Performance Concrete Utilizing Fly Ash As Cement ReplacementUnder Autocl Aving Technique*

Refrence: Mustafa Azeez Bahedh, Mohd Saleh Jaafar, 2018

This study investigated the mechanical and permeability properties of Ultra-high- performance concrete (UHPC) under autoclaving and water curing conditions. The influence of autoclave time and incorporation of five different dosages of fly ash were also examined. Test results revealed a considerable increase in compressive strength of UHPC after water curing. Also, the mechanical performance of UHPC was significantly influenced by the autoclave time and temperature and could be affected negatively beyond the stated value. In addition, the effects of fly ash dosage on the permeability of UHPC were studied. As a result increase in the workability of the concrete mixes as the dosage of fly ash increased from 0%to 40% was reported.

2) Development Of Ultra-High-Performance Concrete Using Glass Powder –Towards Eco-Friendly Concrete

Refrence: N.A. Soliman, A. Tagnit-Hamou, 2016

A green ultra-high-performance glass concrete (UHPGC) with a compressive strength of 220 MPa was prepared and its fresh, mechanical, and microstructural properties were investigated. The test results indicate that the fresh UHPGC properties were improved when the cement and quartz powder were replaced with nonabsorptive glass-powder (GP) particles. The strength improvement can be attributed to the GP's pozzolanic and to its mechanical performance (very high strength and elastic modulus of glass). The microscope investigation revealed the formation of a hydration rim around cement and GP particles. UHPGC provides technological, economical, and environmental advantages compared to traditional ultra-high-performance concrete (UHPC).

3) Evolution Of Phases And Micro Structure In Hydrothermally Cured Ultra-High-Performance Concrete (UHPC)

Refrence: C. Lehmann, P. Fontana & U. Mülle

Thermal curing of Ultra-High-Performance Concrete (UHPC) has a strong influence on its mechanical properties. By applying a water vapour saturation pressure additional to increased temperature the curing conditions are strongly enhanced and leads to improvement of the degree of hydration of the cement paste. Increasing temperature accelerates the formation of crystalline calcium silicate hydrates by dehydrating the cement paste and ends in the formation of gyrolite, truscottite and xonotlite at 200 °C and 15 bars. Therefore the micro structure undergoes change. Cement paste consists of close networked crystal fibres with dimensions up to 1µm. By filling cracks and small pores with crystalline C- S-H phases, flaws in the matrix are healed and generate a more homogeneous micro structure.

Autoclaving encourages dissolution processes at quartz grains, which produces a better cohesion between fillers and the fine crystalline cement paste. As a result, autoclavingenhances compressive and flexural strength significantly but, compared to simple heat treatment at 1 bar, with very low scatter of the test results. Keywords (Flexural Strength, Silica Fume, Calcium Silicate Hydrate, Water Cement Ratio, heat treated sample).

4) Hydration Kinetics And Microstructure Development Of Ultra-High-Performance Concrete (Uhpc) Subjected To Microwave Pre-Curing

Refrence: Jingjing Zhang, Rui Yu, Zhonghe Shui, Kangning Liu, 2022

This research aims to clarify the intrinsic effect of microwave pre-curing on the hydration kinetics and microstructure development of Ultra-High-Performance Concrete (UHPC). Six hours after casting, the UHPC samples are subjected to microwave pre-curing for 60–240 s. Then, the macro and micro characteristics of the cured UHPC are evaluated. The observed experimental results show that the microwave treatment can create a dense shell around thecured UHPC's periphery, which can resist the lateral deformation of the specimen and guarantee the excellent mechanical properties of the treated sample. Moreover, when steel fibres are included into UHPC, the reflection and shielding effect of the 3D fibres network cansignificantly block the promotion effect of microwave on the strength development of UHPFRC. As a result, due to the high energy of microwave, high polymerization hydration products and advanced pore structure (micro and meso scales) can be observed in the pre-cured UHPC.

5) A New Design Approach Of Steel Fibre Reinforced Ultra-High PerformanceConcrete Composites: Experiments And Modelling

Refrence: D.Q Fan, R Yu, Z.H Shui, C.F Wu, Q.L Song, Z.J Liu, Y Sun, X Gao, Y.J He, 2020

This paper addresses a new design of steel fibre reinforced UHPC composites by implanting the fibres into modified Andreasen and Andersen (MAA) particle packing model. A novel method for determining the equivalent spherical diameter of steel fibres is proposed, which preserves the effect on the wet particle packing of an UHPC system, when there are equal volume fractions of steel fibres or these extra spherical particles. Then, the employed steel fibres are treated as spheroidal particles, and implanted into the MAA model for the design of a new UHPC. To demonstrate that the newly designed UHPC has superior performance, itsmacro and micro properties are analysed in detail. The obtained experimental results reveal that the utilized steel fibre (L = 13 mm, d = 0.2 mm) can be treated as a spheroidal particle with a diameter of 5.65 mm in the MAA model. As a result, based on the method proposed in this study, the negative effect of steel fibres on the packing system of UHPC can be minimized, which can enrich the basic design theory of UHPC composite.

6) Microstructural Investigation Of Calcium Aluminate Cement-Based Ultra-High-Performance Concrete (UHPC) Exposed To High Temperatures

Refrence: N.K. Lee, K.T. Koh, S.H. Park, G.S. Ryu, 2017

This study investigated the microstructural and chemical changes of calcium aluminate cement (CAC)-based UHPC exposed to high temperatures. Upon exposure to 100 °C C_3AH_6 was formed by the dehydration of CAH_{10}. A further increase in the exposure temperature to 450 °C resulted in the formation of a new phase $C_{12}AH_7$, which is attributed to the dehydration of C_3AH_6 and AH_3. The compressive strength of UA50 and UA70 increased significantly due tothe formation of C-A-(S)-H gel resulting from the further hydration of anhydrous CAC and silica fume upon exposure to 450 °C. The hydration reaction of CAC in UHPC led to a significant increase in the micro-pores (< 100 nm), thereby releasing the vapor pressure. After exposureto 800 °C the formation of calcium aluminosilicate (C-A-S) gel was observed as a consequence of the sintering of anhydrous CAC and dehydrated C-A-(S)-H with adjacent micro silica, contributing to the residual strength.

7) A Novel Design Of Low Carbon Footprint Ultra-High-Performance Concrete(UHPC) Based On Full Scale Recycling Of Gold Tailings

Refrence: J.N. Wang, R. Yu, W.Y. Xu, C.Y. Hu, Z.H. Shui, D. Qian, Y. Leng, K.N. Liu, D.S. Hou, X.P. Wang, 2021

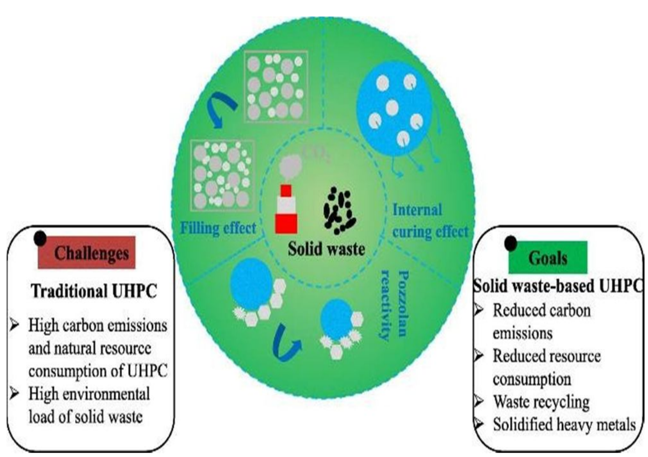
This paper presents a feasible way to design a low carbon footprint Ultra-High PerformanceConcrete (UHPC) with gold tailings based on the idea of full scale recycling. Due to the fact that the particle size distribution of the recycled gold tailings is normally wild, from micrometre to millimetre, it could be utilized to replace powders and aggregates in UHPC simultaneously, which can get rid of sieving process and improve the recycling efficiency.

Therefore, in this study, a particle dense packing model is firstly employed to design the UHPC matrix with gold tailings, based on the full scale recycling point of view. Then, the fresh and hardened properties of the newly developed UHPC are investigated, including flowability, compressive strength, hydration kinetics, durability and microstructure. The tested results indicate that the idea of fully recycling can guarantee a relatively high long- term compressive strength, low D_{RMC}, and low leaching toxicity for UHPC. Additionally, the environmental evaluation further proves that the developed UHPC is a green building material with advanced properties and simple preparation process.

8) Recycling Solid Waste To Produce Eco-Friendly Ultra-High-PerformanceConcrete: A Review Of Durability, Microstructure And Environment Characteristics

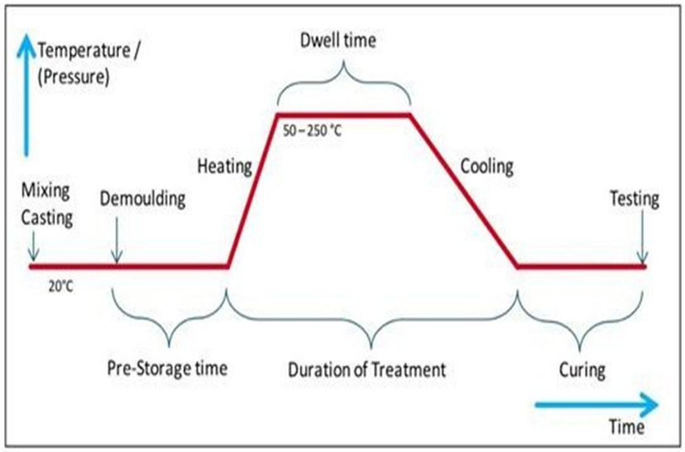
Refrence: Hussein M. Hamada, Jinyan Shi, Farid Abed, Mohammed S. Al Jawahery, Ali Majdi,Salim T. Yousif, 2023

Recycling waste materials (WMs) is a cost-effective method for saving natural resources, protecting the environment, and reducing the use of high-carbon raw materials. This review aims to illustrate the impact of solid waste on the durability and microstructure of ultra-high-performance concrete (UHPC) and to provide guidance for the research of eco-friendly UHPC.The results show that the proper use of solid waste to replace part of the binder or aggregate has a positive effect on the performance development of UHPC, but further enhancement techniques should be developed. When solid waste is prepared as a binder, the durability of waste based UHPC can be effectively improved by grinding and activation. When solid wasteis used as an aggregate, its rough surface, potential reactivity and internal curing effect are also beneficial to the improvement of UHPC performance. Since UHPC has a dense microstructure, it can effectively prevent the leaching of harmful elements (heavy metal ions)in solid waste. As a result, the use of solid waste in UHPC effectively reduces the carbon footprint of the mixture, which is beneficial to the development of cleaner production technologies.



III. HYDROTHERMAL TREATMENT

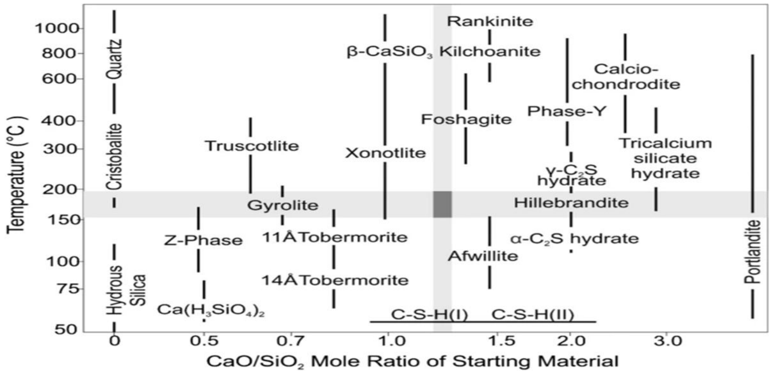
Considering that higher temperatures and longer treatment times consume more energy andare less cost efficient, several authors suggest conditions for an optimal hydrothermal treatment regarding high (compressive) strength. While authors suggest a dwell time of 8h for a hydrothermal treatment at 200 °C and 1.7 MPa, it was proposed that the same dwell time of 8h at a temperature of 150 °C. And analysed the effect of a hydrothermal treatment on Reactive Powder Concrete (RPC) and recommend a slightly longer dwell time of 10h at 180
°C / 1 MPa [29]. All authors agree that longer dwell times or higher pressure do not improve the strength significantly.



There is a technical difference between thermal treatments below 100 °C and treatments above 100 °C. While the former can be done in a heating cabinet or in a water bath under atmospheric pressure, the latter needs a counter pressure in a pressure chamber (autoclave) to prevent excessive drying of the specimens. Hence, it is technically denoted as “autoclaving”, or “hydrothermal treatment” in a more scientific way, because of the permanent water vapour saturation. Thermal and hydrothermal treatment are often referredto as “heat curing” and “steam curing”, respectively.
The heat treatment of precast UHPC elements is following a common procedure: one-day storage in the mould or under humid climate, slowly heating to 90 °C with a dwell time of about 2 days while the elements are protected from drying, and slow cooling to ambient temperature. This method is applied in both scientific field (e.g. [1, 3, 4, 15]) and technical field. However there is no systematic optimisation with regard to dwell time or storage time. Other conditions of thermal treatment (especially temperatures between 150 °C and 250 °C)were studied only scarcely.

IV. PHASE DEVELOPMENT OF UPHC

The effect of limestone on the hydration and microstructural development of ultra-high- performance concrete (UHPC) with different levels of replacement (34%, 54% and 74% by volume) is being investigated. In principle, the general path of phase development in UHPC,
i.e. the formation of hydration products, does not differ from normal concrete. The same phases occur in the same sequence, though the fractions of the individual phases and the kinetics are strongly different. Due to the low w/c ratio especially the amount of non-reactedclinker and supplementary cementitious materials is higher and conversely the degree of hydration is lower than for ordinary concrete.
The amorphous calcium silicate hydrates (C–S–H-phases) found in ordinary concrete as well as in UHPC depend on the chemical composition of the raw materials. Whereas hydration ofpure Portland cement results in C–S–H-phases with a Ca/Si ratio of approx. 1.7 this ratio decreases for increasing replacement with SCMs. The generic term C–S–H describes an indefinite range of semi-crystalline to nearly amorphous phases which in concrete further vary depending on space (inner and outer C–S–H) and time of hydration.
It is generally recognized that thermal treatments at higher temperatures lead to an accelerated formation of C–S–H-phases, which is due to an accelerated reaction of cement clinker and SCMs, such as micro silica. The CaO/SiO_2 mole ratio of the mixture used amounts to 1.2 so that according to the phase diagram the crystalline phases tobermorite, xonotlite and afwillite are likely to be formed during heat treatment.



the impact of the treatment with temperatures above the boiling point of water, or with hydrothermal treatment on the phase composition of UHPC is however less consolidated. Itwas attributed that the high strength of autoclaved reactive powder concrete to the formation of tobermorite . Thermodynamic experiments in the C–S–H system, which also evidenced the occurrence of tobermorite at 200 °C under water vapour saturation.

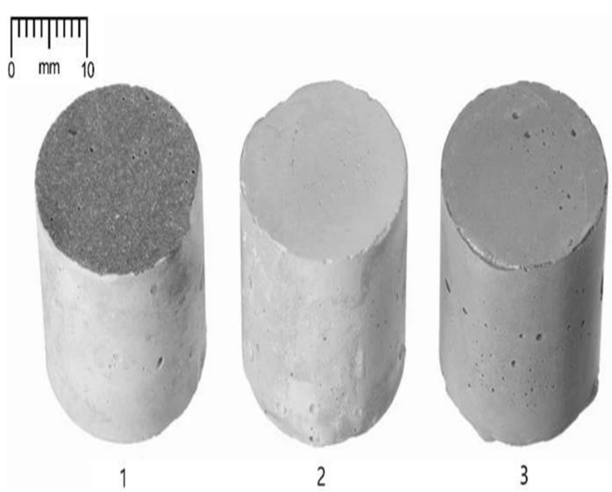
V. UHPC COMPOSITION

Due to its superior granulometry limestone powder is often included as filler into the mixdesign. Also, different SCMs like fly ash and ground granulated blast furnace slag are frequently used as constituents for UHPC.

Influence of binder content, water/binder ratio, ground granulated blast furnace slag (GGBS) content, and limestone powder (LP) replacement on fluidity and compressive strength of concrete were researched, respectively. The test results show that the addition of superplasticizer and fine mineral additives enabled the UHPC to be produced at an extremelylow water/binder ratio of 0.14–0.18, achieving excellent workability with a maximum slump of 268 mm and compressive strengths of 175.8 MPa at 90 d and 182.9 MPa at 365 d.

After mixing in a 10L volume high energy mixer the UHPC was cast in silicone moulds to produce small cylindrical specimens with a diameter and a height of 22.6 mm. The choice of these—even for fine grained concrete—very small dimensions has several reasons. Due to their small geometry, more specimens can be autoclaved at one time and the samples can be heated and cooled without inducing major temperature gradients inside the specimens. Furthermore, regular test equipment for concrete testing can be used for the mechanical testing of the high strength specimens. To compensate for the higher variation resulting fromthe use of small specimens eight samples were produced in each series for averaging.

Cylindrical samples used for strength tests and XRD: (1) after polishing the top surface, (2) after hydrothermal treatment, (3) before hydrothermal treatment.



For better comparability the phase composition and the mechanical strength of all samples were analysed after 28 days, regardless of the pre-storage time before the hydrothermal treatment and the duration of the treatment.

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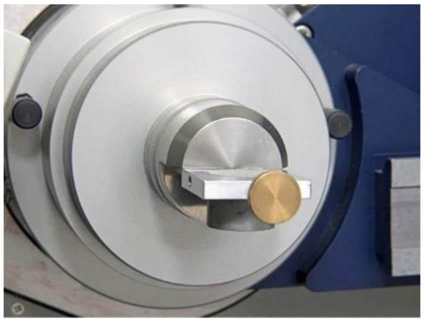
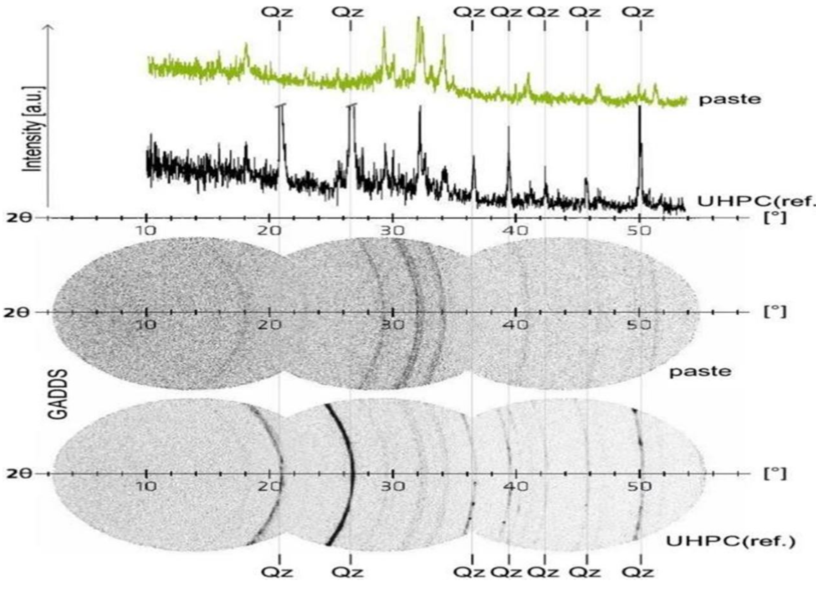
VI. PHASE ANALYSIS

It is generally difficult to see ettringite in XRD results of UHPC containing silica fume, while the C–S–H and CH phases can be seen. Due to the fact that the heat treatment temperature is very effective in hydration products, as the curing temperature increases, the amount of CH converted to C–S–H increases and a denser cement matrix is formed. The XRD analysis reveal that clinker reacts with water to form C–S–H and Portlandite in specimens subjected to standard curing.

The phase analysis was carried out in Bragg–Brentano-Geometry with a DTex-detector and Cu-Kα radiation. The samples were either milled to powder as a reference or measured as a solid sample. Solid samples were polished with ethanol to obtain a smooth and homogeneoussurface and fixed to a special sample holder. To enhance the statistics the solid samples were randomly twisted and measured for several times.



Sample holder with UHPC cylinder for XRD phase analysis



Comparison of GADDS recordings of a solid sample of the UHPC reference mixture (bottom) and the identical paste mixture without quartz (middle) and X-ray-diffractograms determined by integration of the corresponding GADD recordings (top).

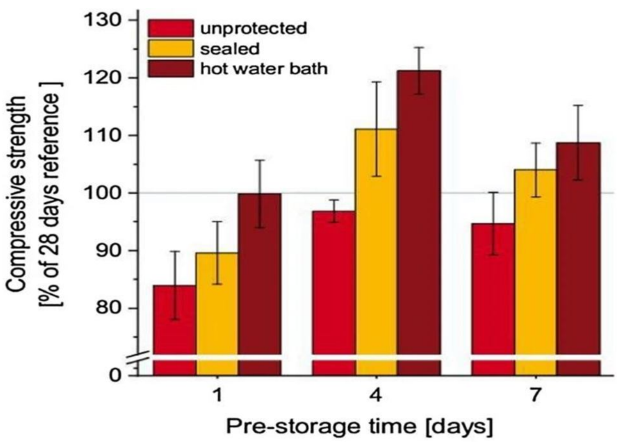
VII. PHASE COMPOSITION AND COMPRESSIVE STRENGTH AFTER TREATMENT

The analysis of thermally treated samples shows that storage for 20 h in hot water of 90 °C leads to a significant decrease of ettringite and portlandite. The advantages of treating prefabricated components made of Ultra-High-Performance Concrete (UCPC) at 90 °C are largely recognized, while hydrothermal treatment at 185 °C and the corresponding saturationpressure of 1.1 MPa.

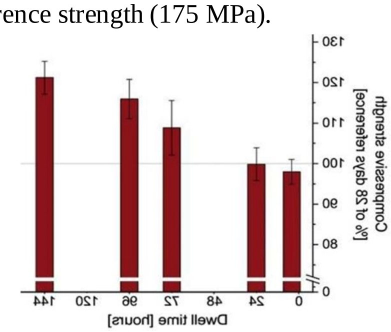
The parameters pre-storage-time before treatment and dwell time in the autoclave were systematically varied. The results illustrate in which way compressive strength increases with the duration of treatment. Already samples which were only heated up and immediately cooled down show an increase in strength compared to the 28-day reference of about 10 %. After 20 h the maximum increase of about 25 % is reached. Interestingly the compressive strength of samples treated very long hardly decreases.

The XRD results indicate that the amount of water, which becomes available due to the decomposition of ettringite at temperatures above 70 °C, is used exclusively for an increased pozzolanic reaction of portlandite with micro silica, forming additional C–S–H-phases. The amount of water appearstoo low to promote a further hydraulic reaction of the cement clinker.

While the compressive strength of the specimens, which were treated in the heating cabinetwithout any protection, is below the value of the reference samples of 175 MPa (representedby the dotted line indicating 100% in Fig. 6), the strength of the samples heat-treated in the water bath is up to 20% higher. The compressive strength of the sealed samples, which are protected from drying but cannot uptake additional water like the samples in the water bath,is still up to 10% higher than the reference value. It can be therefore concluded that for the composition investigated the achievable mechanical strength is not severely sensitive.



Compressive strength of heat-treated cylindrical specimens (144 h at 90 °C) after 28 daysdepending on protection against drying and pre-storage times in percentage of the reference strength (175 MPa).



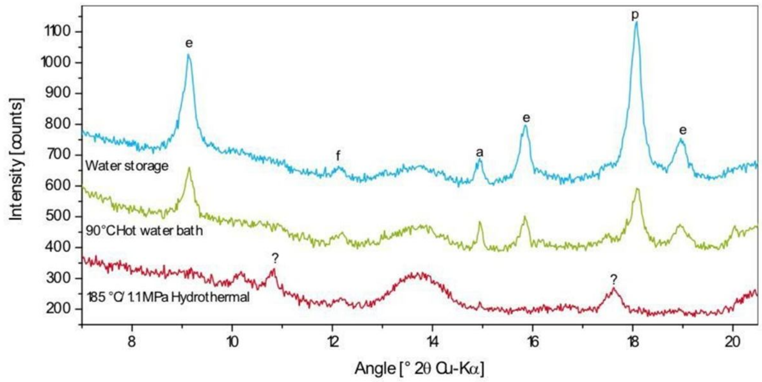
Relative compressive strength of cylindrical specimens with a pre-storage time of 4 ddepending on dwell time in hot water bath.

1) *XRD Measurement Condition:* The results from the powder diffraction and the measurement of the solid samples exhibit no significant differences regarding the presenceand position of peaks. The feasibility of fast qualitative phase analysis using solid samples for the fine grained mixture used is described in detail in a separate publication.

The GADDS recording of the standard UHPC mixture is shown at the bottom of Fig. 4 andexhibits significant spots within the rings, e.g. at 21° 2θ, 36,2° 2θ, 39.8° 2θ and 50.1° 2θ,

which are due to some oversized particles of quartz. The main peak of quartz around 27° 2θ appears as a continuous bold line. Several further homogeneous rings, e.g. at 18° 2θ or around 32° 2θ, are only faintly visible and much more prominent in the paste sample. They can be attributed to cement phases or hydration products. Despite the dominant quartz peaks, all peaks relating to the crystalline hydrates visible in the paste sample can be clearlyand unambiguously determined also in the UHPC sample.

During the hydrothermal treatment at least one new phase was forming, which was not identified yet free of doubt. The XRD peaks are located at d-values 0.820 nm, 0.504 nm, 0,345nm, 0.282 nm and 0.264 nm and might be assigned in parts to hydroxylellestadite or killalaite, a crystalline C-S-H phase. After hydrothermal and 90 °C thermal treatment the surface was covered with a thin layer, which was usually removed by grinding before the measurements. It consisted of calcite and sometimes vaterite. Additionally, the surface layer of the hydrothermally treated samples contained tobermorite.



X-ray diffractograms of hydrothermally treated UHPC samples (185 °C/1.1 MPa) with andwithout surface layer, t-tobermorite, e-ettringite, C-clinker, h-hydroxylellestadite, g- hydrogarnet, Qz-quartz.

The formation of tobermorite is causing the high strength of autoclaved RPC. In fact, tobermorite is a typical phase occurring in other hydrothermally treated calcium-silicate- systems like AAC. However, in this study tobermorite was present only in the surface layer ofthe hydrothermally treated UHPC samples. It was not detected by means of XRD when the surface layer was removed. Therefore, tobermorite cannot be considered responsible for the increased strength of hydrothermally treated UHPC. From the experimental results it is obvious that the reason for the higher strength of thermally treated UHPC samples is simply an intensified puzzolanic reaction. In the case of hydrothermal treatment the strength development is additionally supported by an intensified hydraulic reaction. Hence, more C-S-H is formed that may fill voids, leading to a denser structure and finally to higher strength. At 90 °C these reactions much less pronounced than at 185°C and corresponding water saturation pressure, of course, resulting in lower strength of the former.

As hydroxylellestadite and hydrogarnet are already formed after a short autoclaving time a transition of crystalline phases as cause for the minor decrease in strength with time seems to be unlikely.

Tobermorite, which is known to be the main strength building phase in AAC and CSB was onlyfound within the white deposit at the surface of samples, which had formed during the hydrothermal treatment. Within the bulk of the samples no tobermorite could be identified with XRD.

From the results, it can be concluded that the significant increase in compressive strength observed is due to an increased pozzolanic reaction of the portlandite with the siliceous fillers as well as an increased hydraulic reaction of the remnant clinker.

VIII. CONCLUSION

Thermal and hydrothermal treatment can significantly increase the compressive strength of UHPC depending on temperature and pressure conditions with fundamental variations in strength and phase composition. While for treatment at 90 °C in water the strength dependedin a complex way on pre-storage time and dwell time, it seems to be independent from pre- storage time for hydrothermal treatment at 185 °C reaching a maximum with a dwell time ofabout 20 h. During the the thermal treatment at 90 °C ettringite and portlandite diminished,but they were still present after the treatment. After the hydrothermal treatment both weretotally absent and a new, though not yet identified phase occured. Unexpectedly, tobermorite was not detected in the hydrothermally treated UHPC. Therefore, in contrast toAAC, the formation of tobermorite is not considered as mainly responsible for the increase ofstrength, but for the intensified pozzolanic and hydraulic reactions.

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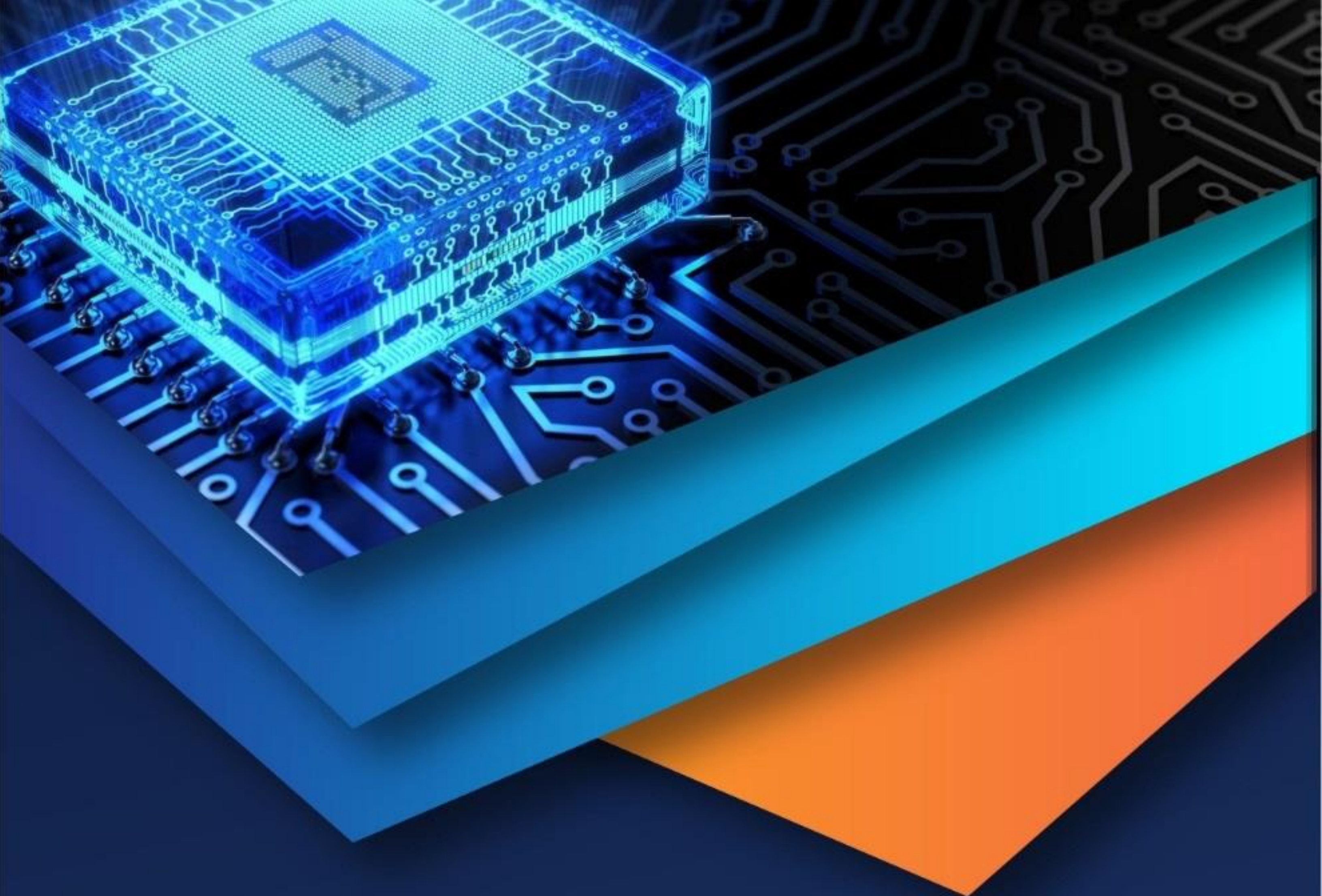

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TOGETHER WE REACH THE GOAL

IMPACT FACTOR: 7.429

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