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# Mix Procedure of Ultra-High Performance Concrete Containing Nano-Silica; Advantages And Challenges To Achieve Required Characteristics

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**Keywords:**
Ultra-High Performance Concrete (UHPC)
Nano Silica
Mix procedure
Mechanical properties
Physical properties

**ABSTRACT**

The paper describes the mix method and the results of the influence of nanoparticles of nano silica (NS) (d ≈ 65 nm) on the Ultra-High performance Concrete (UHPC) matrix properties. Five different contents of NS particles were applied: 0, 0.5, 1, 1.5, and 2 wt.% by weight of cement. The studies regarding different physical and mechanical parameters of cementitious composite matrix specimens such as water absorption, porosity, compressive strength and flexural strength. Structural properties have been carried out and the results of these studies are presented and discussed. It was shown that the nanoparticles in the amounts of 1-2 wt.% can influence the cement hydration process and thereby enhance the compressive strength of cementitious composite. The higher enhancement was obtained during the first three days of hardening due to so called filling effect. In the following stage of hydration, the strengthening effect diminished. This phenomenon was convergent with the XRD analysis, which proved that the nanoparticles react with the cement paste components.

# أسلوب خلط و إنتاج الخرسانة الفائقة الآداء المحتوية على النانو سيليكا، الميزات والتحديات لتحقيق الخصائص المطلوبة

مفتاح محمد سريح

قسم الهندسة المدنية، كلية الهندسة، جامعة المرقب، ليبيا

**الكلمات المفتاحية:**
الخرسانة فائقة الآداء
النانو سيليكا
طريقة الخلط والإنتاج
الخواص الميكانيكية
الخواص الفيزيائيـة

**الملخص**

هذه الورقة البحثية تدرس وتشرح طريقة خلط وإنتاج الخرسانة فائقة الآداء وتبحث في مدى تأثير إضافة النانو سيليكا بحجم حبيبات متوسط يصل إلى 65 نانو متر على خواصها. في هذه الدراسة تم إعداد خمس خلطات من الخرسانة الفائقة الآداء المحتوية على نسب مختلفة من النانو سيليكا، حيث تم استخدام النسب 0.0، 0.5، 1.0، 1.5 و2.0 من وزن الاسمنت المستخدم في الخلطة. وللتحقق من خواص الخرسانة المنتجة شملت الدراسة عدد من الاختبارات التي تبحث في الخواص الفيزيائية كنسبة الامتصاص والمسامية، والتي تبحث أيضاً في الخواص الميكانيكية كمقاومة الضغط ومقاومة الإنحناء. وبشكل عام أظهرت النتائج أن إضافة النانو سيليكا بنسبة من 1-2 % من وزن الإسمنت في الخلطة له تأثير واضح على عملية الإماهة والذي ينعكس في التحسن الملحوظ بمقاومة الضغط للخلطة المنتجة. كما لوحظ أن أفضل تحسن كان في الثلاثة أيام الأولى من الخلط الأمر الذي يؤكد دور النانو سيليكا في عملها كمادة بوزلانية ومادة مالئة تعمل بكفاءة داخل المزيج المكون. نتائج اختبار XRD يؤكد دور النانو سيليكا في تحسين عملية الإماهة وبالتالي تحسن الخواص الميكانيكية.

## 1 Introduction

The development of more durable and sustainable concrete in order to decrease life cycle cost of structures is an important trend in modern civil engineering. In this context, nanotechnology has attracted much attention over the past decade. It is opened a new world in the field of construction and building materials [1-5]. Much of nanomaterials such as nano silica, nano clays, calcium carbonate nanoparticles, nano titanium oxide, nano alumina and carbon Nano tube have been investigated [2, 6, 7]. Applying nanotechnology theory in concrete has started in the last period in line with the increasing demand for Ultra-High Performance Concrete (UHPC). Classical mix formulations of UHPC with the inclusion of silica fumes provide better durability and strength. However, the limited

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availability in some countries and also the high cost of silica fume makes UHPC technology declining and less demanding compared to HSC. Therefore, emerging technology in nano production has developed an alternative to silica fume. By applying nano production concept, a common nanomaterial having the similar action of silica fume is designed [8].

Nano silica is a breakthrough in nanomaterials that has been applied in UHPC. In general, nano silica was produced from micro based silica. The positive reactions created by nano silica in UHPC are similar to silica fume or micro silica in terms of performance strength and durability enhancement [9-11]. In addition, nano silica is similar to silica fume in a way that it increases the packing density, particularly interface between the pastes and aggregate. Using 1% nano silica has an effect of about 10% of micro silica at 90 days. As well as, with more addition of nano silica than 2%, e.g., 3%, the results still higher than those of the control concrete (without nano silica). It was observed that the binary use of nano and micro silica has better performance on the characteristics of UHPCs when compared to its individual use [12].

Study conducted by Qing and Zenan [11] showed that concrete modified with nano silica gained early strength compared to that of silica fume, as well as improving its workability. Workability improvement is referred to the round shape of nano silica which provides a ball bearing maneuver in cement particles [4]. Furthermore, size of nano silica which in nanoparticles acts as ultra-filler in concrete. Micro voids in concrete will be densified and refined to provide a neat concrete microstructure [13, 14]. Quercia and Hüsken [15] revealed that addition of nano silica at certain dosage not only improves strength of HSC, but also acts as a cement replacement material, about 20%–30% of cement content can be reduced by nano silica. Although, the above advantages of nano silica, it's technology is limited in some country, in the present time, which in turn increases its production costs [9]. Camiletti, et al. [16] investigated the effects of nano and micro limestone on early age properties of UHPC and found that addition of nano and micro limestone reduced its setting time. Moreover, addition of 2.5% and 5% nano limestone could lead to 32% and 75% improvement in 24 h compressive strength compared to control mix. Rong, et al. [17] found that nano silica accelerated the hardening and enhanced mechanical properties of UHPC when incorporated with 3% nano silica, by mass of cementations materials. Ghafari, et al. [9] reported that nano silica reduced the workability of UHPC and increased the compressive strength, especially at early age. Although both nano CaCO3 and nano SiO2 could improve mechanical properties, their hydration mechanisms, hardening processes, and age dependencies are different. This leads to different hydration products and thereby change in mechanical properties [18]. Furthermore, mechanical stirring and ultrasonic dispersion techniques are often adopted to avoid agglomeration of nanomaterials [19]. However, the dispersion time and speed would contribute to the experimental results, which are often neglected. If nanomaterials can be efficiently dispersed under normal mixing procedure, this would not only facilitate their applications in cement-based materials but also reduce energy consumption. Franke, L., et al. [20]. investigated the pore size distribution of some different concretes such as UHPC, High Performance Concrete (HPC) and ordinary concrete (OC) tested by mercury intrusion method in the University of Kassel [21]. Test results showed that the ordinary concrete own a total porosity of 15% while it is 8.2% and 6.0% for HPC and UHPC, respectively. Results also showed that, UHPC has a capillary pore content of 1.5%, while in ordinary concrete and HPC it is about 8.3% and 5.2% respectively. However, studies attempting to investigate the UHPC incorporating nano materials still need more research, although, it's importance due to its widely applications. An experimental investigation was done by this study about this subject.

### 1.1 Scope of work

The interest in using nano silica in UHPC production has been projected in this present research.

As a comparison with previous researches, this study aimed to evaluate the effects of nano silica on absorption, porosity of UHPC. The mechanical properties of the investigated mixtures are also discussed. The content of the nanomaterials is increased from 0 to 2% by mass of cement content. The gained results of the present study provide insight of nano silica as a key component of UHPC.

### 1.2 Materials

The cement used throughout this investigation was Ordinary Portland Cement (OPC) CIM I 52.5N which complies with ESS 4756-1/2013 and had a 28-day mortar compressive strength of 57.5 MPa. The initial and final setting times were 150 min and 230 min respectively. The used silica fume (SF) was commercially called Sika fume from Sika (Egypt) Limited which conformed with ASTM C1240-15 [22]. The used silica fume was very fine with particle size of 0.1-1 µm, which existed in grey powder form and including 93.5% reactive silicon dioxide and no containing chloride or other potentially corrosive elements. The very low water to binder ratio (0.17) was considered with the use of polycarboxylate based Superplasticizer (SP) for obtaining its workability (diameter of flow table 320-360 mm). In this investigation, the 3rd generation of SP called MasterGlenium® RMC 315 from BASF (Egypt) limited with the solid content of 40% was used. It was an extremely high range water reducing agent that was compatible with the requirements for SPs according to ASTM C494-15 [23]. Descriptions of used SP are shown in Table 1. nano silica (NS) was used. The morphology and XRD analysis of NS are shown in Figs. 1 and 2, which indicate the amorphous nature of NS particles. The Dynamic Light Scattering test (DLS) was used to detect the particle size distribution of NS as shown in Fig. 3. From this figure it is observed that the NS particle sizes ranged from 40 nm to 90 nm. The chemical composition and physical properties of OPC, SF and NS are presented in Table 2, 3. The used sand in this investigation was Fine natural siliceous Sand (FS) with particle size ranges from 300 to 600 µm and absorption ratio 0.55%. In this study, all UHPC mixes contain 1.0% micro steel fibers (STF) by volume of concrete. The steel fibers was used namely Dramix with brass coated, straight and smooth fibers with a diameter of 0.2 mm and a length of 13 mm. The tensile strength of the fiber was 2600 MPa. The used steel fibers are according to ASTM C1116-15.

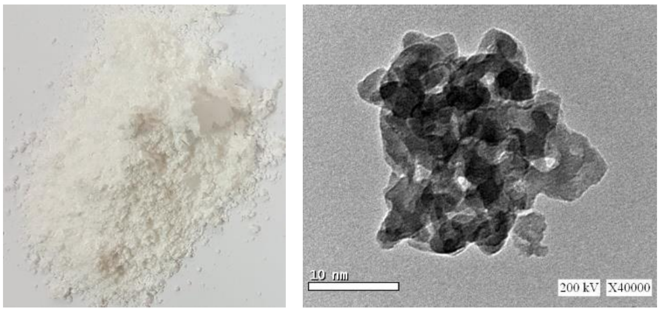


**Fig. 1:** Morphology of NS. (a) NS powder,
(b) TEM image of NS.

Finally, potable water with around 7 pH value was used in the study according to ASTM C1602-04 specification.

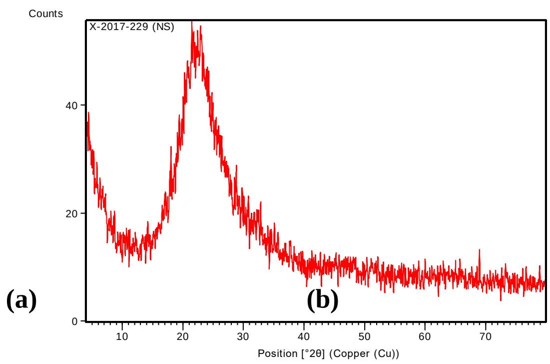


**Fig. 2:** XRD analysis of NS.

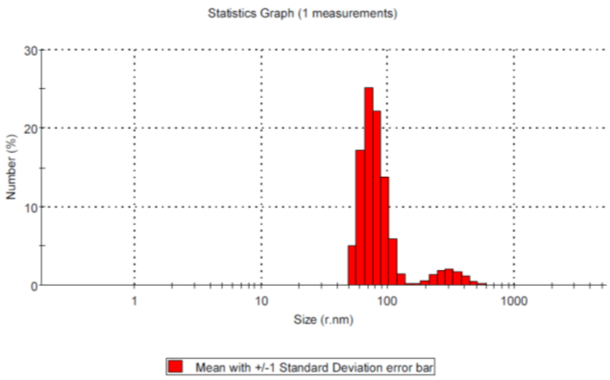


**Fig. 3:** Particle size distribution of NS by DLS test.

**Table 1: Descriptions of SP.**

| | |
|---|---|
| Type of SP | MasterGlenium® RMC 315 |
| Basis | Based on modified polycarboxylic ether |
| Appearance | Off white opaque liquid |
| Specific gravity @ 20°C | 1.10 ± 0.03 g/cm³ |
| pH value | 6.5 ± 1 |
| Recommended dosage | 0.2-3% of cementitious weight (binder) |

**Table 2: Chemical compositions of raw materials.**

| Oxide % | OPC | SF | NS |
|---|---|---|---|
| $SiO_2$ | 20.8 | 93.5 | 98.98 |
| $Al_2O_3$ | 4.6 | 1.0 | 0.033 |
| $Fe_2O_3$ | 2.8 | 1.5 | 0.015 |
| CaO | 65.4 | 0.5 | 0.130 |
| MgO | 1.9 | 1.0 | < 0.01 |
| $SO_3$ | 2.2 | - | < 0.01 |
| $Na_2O$ | 0.31 | 1.0 | 0.180 |
| $K_2O$ | 0.44 | 1.5 | < 0.01 |
| $TiO_2$ | - | - | 0.025 |
| cl | - | - | 0.270 |
| Lol | - | - | 0.320 |

**Table 3: Physical properties of raw materials.**

| Property | OPC | SF | NS |
|---|---|---|---|
| Specific gravity | 3.15 | 2.23 | 2.3 |
| Average particle size | 1-10 µm | 0.1-1 µm | 65 nm |
| Specific surface area ($m^2$/g) | 0.35 | 20 | 135.5 |
| Density | - | 0.55 (kg/L) | - |

## 1.3 Mix proportions, mixing method and curing

### 1.3.1 Mix proportions

Since there is no standard design mix method for the UHPC, the final mix designed in this study was finalized based on the UHPC mix (M3Q) after going through series of trial mixes. The mix (M3Q) was developed at the University of Kassel and was used as the reference mix in the priority program (sustainable construction with UHPC, SPP1182) of the German research foundation. The decision to select this mix was conducted on the cement content which was the lowest among other studies [24]. In addition, the basis of this modified design mix was used in this study to achieve the targeted compressive strength (at day 28 more than 140 MPa).

Five mixes were studied in this investigation. The detailed of these mixes are summarized in Table 4. Different used dosages of nano silica were 0%, 0.5%, 1%, 1.5% and 2% as an addition by weight of cement content.

**Table 4: Mix proportions of UHPC.**

| | Mix | UH1 | UH2 | UH3 | UH4 | UH5 |
|---|---|---|---|---|---|---|
| (Kg/m³) | W/B | 0.17 | 0.17 | 0.17 | 0.17 | 0.17 |
| | Water | 195 | 195 | 195 | 195 | 195 |
| | OPC | 910 | 910 | 910 | 910 | 910 |
| | SF | 230 | 230 | 230 | 230 | 230 |
| | NS | 0.00 | 4.55 | 9.10 | 13.65 | 18.20 |
| | Fine Sand | 1120 | 1120 | 1120 | 1120 | 1120 |
| | SP (%) | 1.70 | 1.72 | 1.74 | 1.77 | 1.80 |
| | STF | 82 | 82 | 82 | 82 | 82 |

### 1.3.2 Mixing method

Concrete mixing was conducted in a laboratory mixer applying the following steps: i) nano particles' solution was prepared by mixing nano silica thoroughly with part of the mixing water using high-speed stirring machine for one minute [25]. Then the solution was placed in a sonication device for fifteen minutes at temperature of 60℃. The main aim of the sonication process is to disperse the nano silica in the solution; ii) Cement, silica fume and fine sand were dry mixed together for three minutes. During that time, the used SP was mixed with two thirds of the remaining water alongside the previous nanoparticles' solution and stirring was continued for another two minutes [26, 27]; iii) The lastly prepared solution was added to the dry mix gradually within three minutes out of the total mixing time of ten minutes; iv) The mixing process is paused for two minutes. Then the remixing was performed again for twenty minutes, during which the rest of the remaining water was being added; v) Steel fibers were added in the last three minutes of the mixing process. Fig. 4 Explains the steps of the mixing process [28].

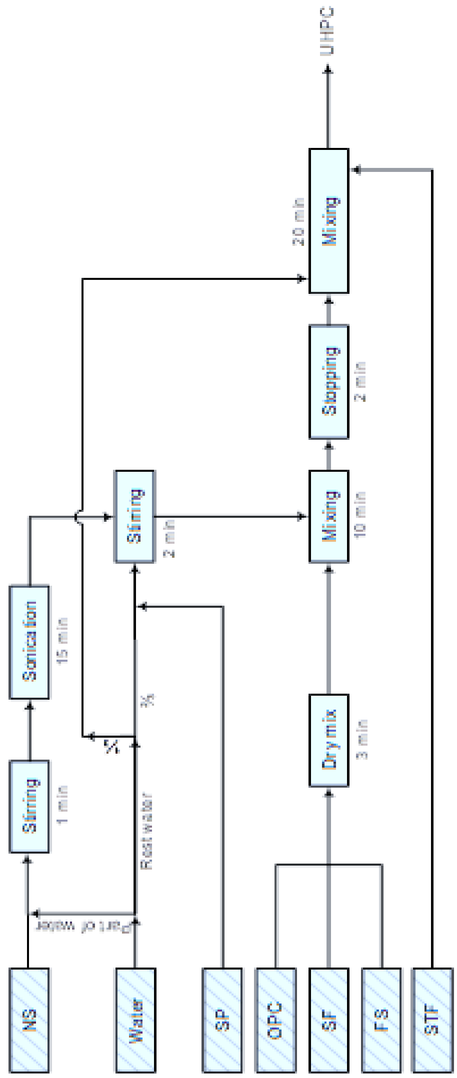


**Fig. 4:** UHPC mixing process.

### 1.3.3 Curing conditions

After the mixing process, the fresh mixtures were cast into molds and compacted by a standard vibrating table. The specimens were covered with plastic sheets after casting and kept for six hours at room temperature (22.0 ± 2℃). After that the specimens were demolded and cured at 80°C under a vapor source for seventy-two hours. Finally, all specimens were placed in a water tank at room temperature and cured for designated ages before testing.

## 1.4 Tests

### 1.4.1 Physical characteristics

Physical characteristics in this study included water absorption, volume of permeable voids (VPV) and bulk density of UHPC. Water absorption and VPV values of UHPC mixes modified with NS were measured after 150 days on 70×70×70 mm3 cubes specimens according to ASTM C642-13 [29]. Subsequently, the average bulk density after 150 days of three cubic specimens with dimensions of 70 mm was determined in oven-dry condition according to ASTM C642-13 [29].

### 1.4.2 Mechanical Tests

Compressive strength of 70 mm cubes was conducted after 1, 3, 7, 28, 90 and 150 days from casting. The test was performed using control compression machine (ELE ADR2000) with a constant loading rate of 0.8 kN/s. The test was carried out on triplicate specimens and average compressive strength values were calculated. Flexural strength test was done on 40×40×160 mm3 concrete prisms for all mixes after 7 and 28 days accordance to ASTM C293-10 [30]. Three specimens per mix were tested, and the average strength was reported.

### 1.4.3 Microstructure investigation (X-ray diffraction)

For X-ray diffraction quantitative analysis, after 28 days the specimens were grind to particles that could pass the 200 µm sieve. Then, the powders were dried in oven at 60℃ for twenty-four hours. Finally, analytically pure $Al_2O_3$ and pre-specimen powders were mixed uniformly under the mass proportion of 1:9 for the qualitative analysis. The target-anode of X-ray diffraction was copper, and the working voltage and electric current were 40 kV and 30 mA, respectively. The step size was 0.02° 2θ and scanning angular range was 5–75°.

## 2 Results and discussion

### 2.1 Permeability indices

The values of water absorption, volume of permeable voids (VPV) and density for the UHPC mixtures incorporating with various dosages of NS (0.0%, 0.5%, 1.0%, 1.5% and 2.0%) are tabulated in Table 5. From the experimental test results, it is obvious that using NS with different dosages has a positive effect on reducing the water absorption percentage and volume of VPV compared with that of control mix, where the water absorption and VPV decrease with the increase of NS dosage. For example, the reduction in VPV of UHPC due to the use of NS is 0.2%, 3.8%, 11.9% and 19.2% for UHPC mixes with 0.5%, 1.0%, 1.5% and 2.0% of NS contents compared with that of control mix, respectively. This is attributed to the discontinuity of capillary pores by formation of more CSH gel due to pozzolanic effect which leads to the dense micro structure with low permeability [8, 31]. The results are in a good acceptance with those obtained by Ghafari, E. et al. [32] and Abbas, S. et al. [33]. The bulk density results as given in table 5 confirm the absorption and VPV experimental results where the use of NS increase the bulk density of different UHPC mixes. The test results of concrete porosity (VPV) confirm the water absorption test results as shown in Table 5.

**Table 5: Effect of NS content on water absorption and volume of permeable voids (VPV) of UHPC at 150 days.**

| | UH1 control | UH2 NS 0.5% | UH3 NS 1.0% | UH4 NS 1.5% | UH5 NS 2.0% |
|---|---|---|---|---|---|
| **Absorption** | 2.29 | 2.24 | 2.13 | 1.95 | 1.79 |
| | Compared to control | -2.51% | -7.13% | -15.19% | -22.05% |
| **Volume of permeable voids (VPV)** | 4.95 | 4.94 | 4.76 | 4.36 | 4.00 |
| | Compared to control | -0.23% | -3.84% | -11.86% | -19.18% |
| **Bulk density** | 2.459 | 2.462 | 2.471 | 2.477 | 2.486 |
| | Compared to control | +0.13% | +0.47% | +0.71% | +1.09% |

### 2.2 Strength performance

Figure 5 shows the results of compressive strengths of the UHPC mixes (with and without NS) at different ages. The compressive strength test results clearly show that using different dosages of NS have a positive effect on enhancing the compressive strength compared with that of control mix. For example, the improvement in compressive strength of UHPC mixes modified with NS after 150 days is 2.9%, 6.2%, 12.6% and 13.7% for UHPC mixes with 0.5%, 1.0%, 1.5% and 2.0% NS, respectively, compared with that of control mix (0.0% NS). Also, it is clear that the increase of NS content from 1.5% to 2.0% has slight effect on compressive strength test results specially at later age. The enhancement in compressive strength associated with NS may be due to the filling effect and pozzolanic reaction of nanomaterials [34-37]. Wu, Z., et al. [38] and Mosaberpanah, M. A., et al. [39] studied the effect of NS on compressive strength of UHPC. There results agree with this experimental result. The change in strength properties of investigated UHPC mixtures made with different dosage of NS agreed well with the physical properties.

The modulus of rupture test results of studied UHPC mixes modified with NS are shown in Fig. 6. As can be seen from the figure, it is clear that the addition of NS significantly improves the modulus of rupture. However, the improvement depends on NS dosage. For example, the improvement in modulus of rupture of UHPC mixes modified with NS after 28 days is 2.3%, 11.5%, 13.5% and 19.2% for UHPC mixes with 0.5%, 1.0%, 1.5% and 2.0% NS, respectively, compared with that of control mix (0.0% NS). These results are in a good agreement with the data published by Wu, Z., et al. [38] and Mosaberpanah, M. A., et al. [39]. These results agree well with the compressive strength test results.

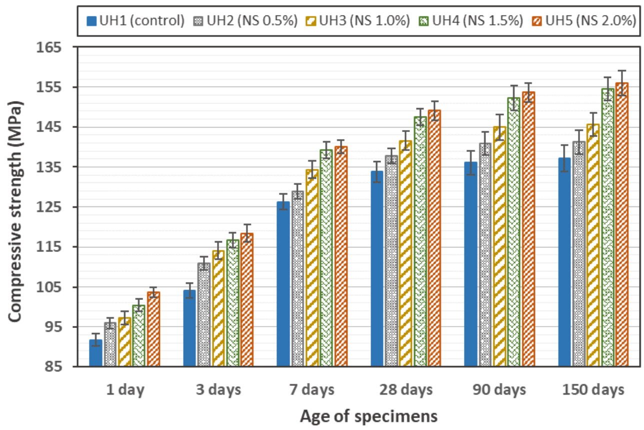


**Fig. 5:** Compressive strength of UHPC specimens modified with NS at different age.

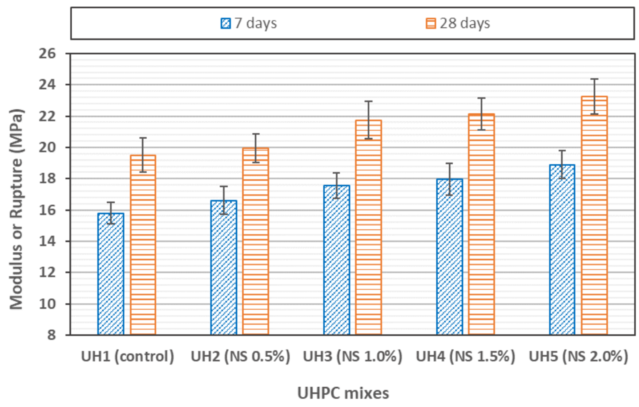


**Fig. 6:** Flexural strength of UHPC specimens modified with NS at different age.

### 2.3 Microstructure properties

#### 2.3.1 X-ray diffraction analysis (XRD)

XRD assessment was performed on the UHPC mixtures incorporation with NS after 28 days. Fig. 7 shows the XRD patterns of UHPC with different content of NS, i.e. 0.0%, 1.0%, and 2.0%. These patterns highlight primarily the quartz which is the constituent of the granular phase, and the anhydrous phases of cement, namely C3S and C2S [40]. As indicated in Fig. 7 the addition of 1.0% and 2.0% of NS to UHPC mixes decreased the peak intensity of all studied component compared to those of reference sample (without NS). This reduction can be related to the reduction of portlandite content in the samples induced by pozzolanic activities of NS, where the portlandite having certainly been transformed into calcium silicate hydrate (CSH) or calcium aluminate silicate hydrate (CASH) [41, 42]. Also, it was noticed, that when amount of NS increases, intensities of anhydrous phases of C3S and C2S tends to decrease. The decrease of anhydrous phases of C3S and C2S peaks are probably related to a better solubility of the clinker phase. Finally, it is observed that, the basic crystalline phases in all samples are quartz and cement minerals. It is evident to state that similar behavior were reported by Ghafari et al [9, 43]. The XRD results confirmed the pozzolanic activity evaluation results from the compressive strength.

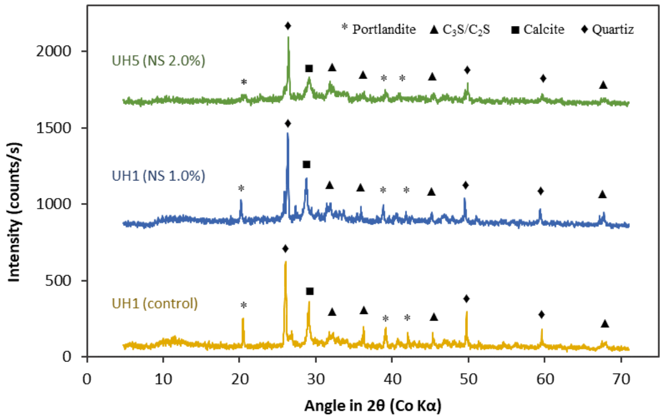


**Fig. 7:** Chemical compounds of UH1, UH3 and UH5 at 28 days, obtained from the XRD technique.

In addition, it was noted that surface cracks appeared on the surface of the samples after casting, this phenomenon was explained in detail in another research that studies the effect of nanoparticles on the shrinkage property of concrete.

## 3 Conclusions

Based on the discussions made in this paper, following conclusions are highlighted in the end:

- Fine mineral admixtures like nanomaterials are very efficient in improving the overall performance of UHPC. The use of nano silica could have a significant effect on the hydration process, pore structure and mechanical strength of UHPC.
- The addition of NS into UHPC can enhance the hydration reaction rate, especially in the early stage of the hydration process because of its pozzolanic reactivity and filling effect.
- Nano silica can help decrease the porosity and injurious pores, increase silicate tetrahedron chain and increase the strength of C-S-H, so improving the mechanical properties and durability remarkably.
- The influence of nano silica on UHPC compressive strength was clearer at the early age (up to 7-day) for all mixes compared with that at 28-day, corresponding to the acceleration of the hydration process due to the ultra-high surface area of nanomaterials.
- It is expected that the hybrid uses nano silica with other materials to improve the performance of UHPC would be the future research trend, which requires in-depth understanding of the effect mechanism of nano silica alone on UHPC.
- Results achieved by XRD analysis confirmed the contribution of the use of nano silica as a partial replacement to cement in enhancing the performance of concrete by improving the pore structure of concrete.

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