

## Effect of Activated Pozzolan with $\text{Ca(OH)}_2$ and nano- $\text{SiO}_2$ on Microstructure and Hydration of High-Volume Natural Pozzolan Paste

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Received 06 July 2018; Accepted 05 October 2018

### Abstract

The main aim of this study is to investigate the effect of activated pozzolan on hydration and microstructure of high-volume natural pozzolan paste. Thermal activation of natural pozzolan with  $\text{Ca(OH)}_2$  has been applied with nano-silica (NS) as activator at three different temperatures. X-ray diffraction (XRD), thermogravimetric analysis (TGA), laser particle analysis, scanning electron microscopy (SEM) with energy dispersive spectroscopy were employed. In order to analyze the test results, notions of "pre C-S-H" based on XRD and TGA results of activated pozzolan powders has been used. SEM images indicated microstructural improvements of the pastes with activated pozzolans compared to paste with natural pozzolan and natural pozzolan incorporating NS, showing the pore-filling effect of activated pozzolans. The microstructural improvements were proportion to the amount of pre C-S-H formed during the activation of pozzolan.

**Keywords:** Activated Pozzolan;  $\text{Ca(OH)}_2$ ; Nano-Sio2 (NS); Pozzolanic Power; Pre C-S-H.

### 1. Introduction

Iran has a large number of natural pozzolan sources including Shahindej, Sahand, Sirjan, Rafsanjan, Taftan and etc. [1]. Natural pozzolans have volcanic origin [2] and are used in cement industry for production of pozzolana cement to reduce the effect of sulphate attack and hydration heat for giant constructions like dam construction. Natural pozzolans through a chemical reaction with  $\text{Ca(OH)}_2$ , in the presence of water in cement paste, show cementitious property but their chemical reactivity is too slow. The chemical reactions in hydration of Portland cement are as follows [3]:



Where  $\text{C}_3\text{S}$  is alite,  $\text{C}_2\text{S}$  is belite, CH is calcium hydroxide and C-S-H is calcium silicate hydrate which is responsible for concrete strength but calcium hydroxide reduces the compressive strength of the concrete due to its hexagonal crystalline structure and positioning in the interfacial transition zone (ITZ) which is harmful [4].

Natural pozzolans are low in CaO and high in  $\text{SiO}_2$  and have high ignition loss [2] and due to less amount of CaO in

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<http://dx.doi.org/10.28991/cej-03091171>

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their compositions, low strength is gained in the concrete which natural pozzolan is used in compared to cement-based concrete, but it can gain its strength in long term. The addition of CaO with specified percentage to natural pozzolans and fly ash increases the compressive strength [5, 6]. The pozzolanic reactivity (Ca(OH)<sub>2</sub> consumption) of natural pozzolans is too slow [5]. However, since about 5% of global CO<sub>2</sub> emission is from cement industries [12], the replacement of a percentage of cement in concrete with pozzolanic materials like fly ash or natural pozzolans is a way to reduce the consumption of cement referred to as high-volume fly ash concrete [13]. The use of fly ash or natural pozzolans by a cement plant is dependent on the accessible resources of pozzolanic materials.

Activation of pozzolanic materials like fly ash has improved the mechanical strength, stability, durability, and microstructural improvements. There are different techniques for activation of pozzolanic materials like chemical, mechanical, mechano-chemical and physiochemical methods where most of the chemical activation methods use alkali-based materials [14].

Fan et al. [7] activated fly ash with Ca(OH)<sub>2</sub> and Na<sub>2</sub>SiO<sub>3</sub> at high temperature and used the obtained activated fly ash with cement in concrete in different ratios and improved the compressive and bending strength of the concrete. In another study Vargas et al. [15] tested the compressive strength of activated fly ash with NaOH and Ca(OH)<sub>2</sub> mortar using high curing temperature (80°C) for the first 24 hour to increase the reaction between NaOH and Ca(OH)<sub>2</sub>. The results showed improvement in the compressive strength at the early ages at 7<sup>th</sup> day. However, the compressive strength reduced significantly at the 28<sup>th</sup> day. Dongho Jeon et al. [8] studied the addition of Ca(OH)<sub>2</sub> with a small amount of Na<sub>2</sub>CO<sub>3</sub> to a cementless fly ash system with curing temperature of 60°C and the compressive strength improved even more than addition of NaOH to the same system in the same condition.

Nano materials with Nanometer size and ultra-high specific surface area increase the chemical reactions between cement particles and improve concrete different properties [9, 16]. NS is a pozzolanic material like natural pozzolans, fly ash and silica fume all of which improve concrete properties when used as the replacement for specific percentage of cement. However, the difference between NS and other pozzolanic materials is that it has a high reactivity due to its nano size. Among all nano materials, nano-silica (NS) is used more in concrete technology due to its high pozzolanic reactivity which leads to more formation of C-S-H that fill voids [4, 17]. Also, nano-silica improves the concrete mechanical strength, durability properties, workability, drying shrinkage and thermal stability [4, 17]. To our knowledge, most of the papers investigate the addition of raw nano materials to cementitious materials and different types of concretes. Also, the most important parameter considered is the well-dispersion of nano materials between their particles.

Most of the papers reported activation of fly ash with Ca(OH)<sub>2</sub> and alkali-based materials but less about natural pozzolans. To our knowledge activation of pozzolanic material like fly ash or natural pozzolans, with Ca(OH)<sub>2</sub> has not been applied using NS as activator. Therefore, the objective in this study is to apply the method of thermal activation of pozzolanic materials with Ca(OH)<sub>2</sub> to natural pozzolan with NS as activator instead of an alkali-based activator. Also, some notions such as “pre C-S-H”, “used pozzolanic power” and “remained pozzolanic power” are used in order to analyze the testing results. Thermal activation of raw pozzolan is done at three different temperatures of 55°C, 65°C and 75°C. XRD, TGA, laser diffraction test and scanning electron microscopy (SEM) are performed on raw and activated pozzolans. The hydration and microstructure of high-volume activated pozzolans, raw pozzolan and raw pozzolan incorporating NS pastes are considered through TGA and SEM. It shows that the activated natural pozzolans with Ca(OH)<sub>2</sub> and nano-silica at different temperatures are effective for microstructural improvement of the paste.

## 2. Materials and Methods

### 2.1. Raw Materials and Their Properties

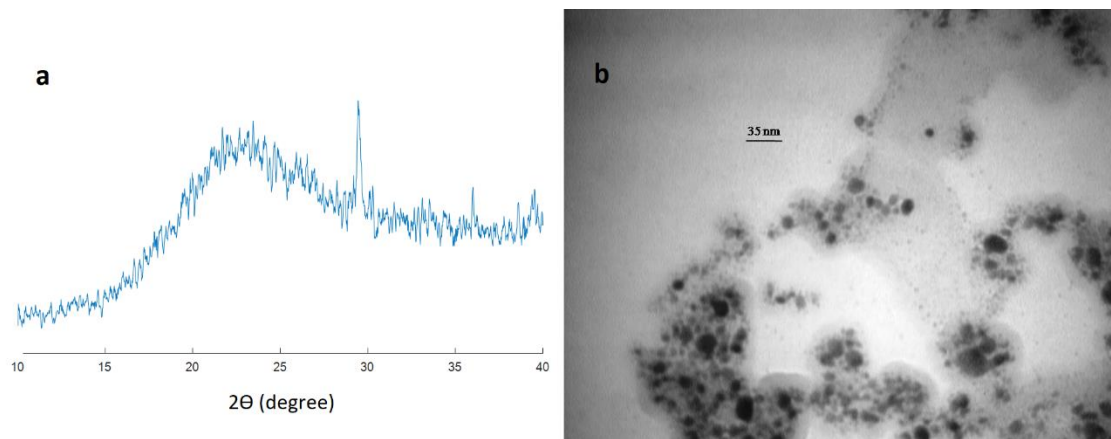
Portland cement type II and natural pozzolan were provided from Kerman Cement Factory in Iran. The pozzolan used in this research is Sirjan pozzolan which is extracted from Sirjan mine in Kerman state in Iran. Table 1 shows the chemical analysis of Portland cement type II and Sirjan pozzolan.

**Table 1. Chemical analysis of Portland cement type II and Sirjan pozzolan**

Chemical compound	Portland cement type II	Sirjan pozzolan
SiO <sub>2</sub>	21.74	63.08
Al <sub>2</sub> O <sub>3</sub>	5.0	18.38
Fe <sub>2</sub> O <sub>3</sub>	4.0	4.16
CaO	63.04	3.33
MgO	2.0	1.59
SO <sub>3</sub>	2.3	0.05
K <sub>2</sub> O	-	3.41

Na <sub>2</sub> O	-	2.14
Loss on ignition	1.3	4.18
C <sub>3</sub> S	45.5	-
C <sub>2</sub> S	28.0	-
C <sub>3</sub> A	6.5	-
C <sub>4</sub> AF	12.2	-
Insoluble res	0.60	-

Sirjan pozzolan was powdered and passed from sieve #200 (74  $\mu\text{m}$ ) for the experiments. Cement and pozzolan specific surface areas are 2900 and 11705  $\text{cm}^2/\text{gr}$  respectively. NS average particle size is 11-13 nm and its specific surface area is 200  $\text{m}^2/\text{gr}$ . Nano-silica particles have separated spherical shape with an amorphous XRD pattern. The transmission electron microscopy (TEM) image of nano-silica and XRD pattern of nano-silica is shown in Figure 1. Figure 2 shows about 2 grams of NS in a watch glass. This image superficially shows the necessity of well-dispersion of NS particles between cementitious materials.



**Figure 1. (a) XRD pattern of NS showing amorphous (non-crystalline) phase (b) TEM image shows the spherical shape of NS**



**Figure 2. two grams of NS in the watch glass**

## 2.2. Preparation of Pastes and Tests

Five different pastes were prepared: (1) cement + raw pozzolan (defined with CP); (2) cement + raw pozzolan + NS (defined with PNS); (3) cement + activated pozzolan at 55°C (defined with APNS55); (4) cement + activated pozzolan at 65°C (defined with APNS65); (5) cement + activated pozzolan at 75°C (defined with APNS75). The weight of raw pozzolan and activated pozzolans was the same as cement meaning that 50% cement and 50% raw or activated pozzolans and NS was added in PNS sample for 2% of cementitious materials. For preparation of pastes, cementitious materials were mixed dry for 2-3 minutes then de-ionized water was added and hand-mixing performed for 2-3 minutes. The w/c ratio was 0.35 for all samples. This process was repeated for PNS but for well dispersion of NS between cement and pozzolan particles, cementitious materials and NS were mixed with pure acetone [10] for 5 minutes, then the mixture was dried in oven at 55°C. The pastes were cast into cylindrical tubes with diameter of 3 cm and well compacted by hand. The molds were removed and the pastes were cured in 22±1°C water (at room temperature) for 7 and 28 days. For thermogravimetric analysis (TGA) and XRD tests, pastes were powdered and kept in pure alcohol for 2 days to stop

hydration then dried in oven at 55°C and passed from sieve #200 (74  $\mu\text{m}$ ). Thermogravimetric analysis was performed on powders in the range of 50°C-750°C with 25°C/min in argon atmosphere. In order to obtain the DTG (differential thermogravimetric analysis) curve, the raw data of TG analysis was extracted and differentiated with respect to temperature using MATLAB software. For SEM test, the pastes cured for 28 days in water, were cut and polished with polishing papers #1000, #1500, #2000 and #3000 in order to obtain a flat surface and a thickness of about 3 mm. SEM was performed using a VEGA\TESCAN-LMU electron microscope. The samples must be dry since they are imaged in vacuum atmosphere. The voltage used for imaging the samples was 15 KV. The surface of paste samples and powders were coated with gold in order to make them conductive. SEM backscattered electron (BSE) images were taken. Also, energy dispersive spectroscopy (EDS) was performed on the pastes in order to obtain the atomic percentage of Ca, Si, Na, K and Al elements. The range of  $2\theta$  for activated pozzolan powders was 10°-50° using copper as the anode for production of X-rays (Cu-K $\alpha$ ).

### 2.3. Activation of Pozzolan

Raw pozzolan, Ca(OH)<sub>2</sub> and NS were mixed dry then dissolved in de-ionized water for 5 minutes. Ca(OH)<sub>2</sub> was added for 25%, NS for 4% of pozzolan weight with water/pozzolan ratio of 3.5. Three different activation temperatures were considered for activation of raw pozzolan with Ca(OH)<sub>2</sub> and NS. The solutions were kept in oven in a sealed cylindrical container at temperatures of 75°C, 65°C and 55°C, then dried in oven at 55°C for 15 hours (Figure 3a). Activated pozzolans, which were like dried soil powdered by hand (Figure 3b) then, passed from sieve #200 (74  $\mu\text{m}$ ). The goal in activation of pozzolan is the complete usage of Ca(OH)<sub>2</sub> which was determined through XRD patterns of activated pozzolans at different temperatures and activation times. The activation time were 20, 14, and 5 hours at 55°C, 65°C, and 75°C respectively. Another image of materials used in during the preparation of the paste in the laboratory is shown in Figure 4.

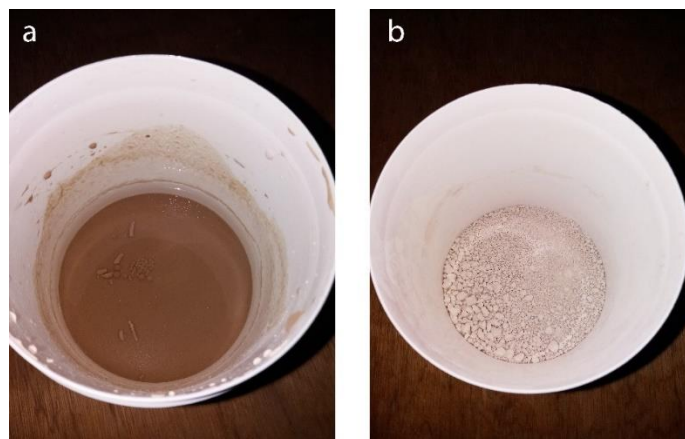


Figure 3. (a) Pozzolan after activation in oven (b) Dried activated pozzolan, agglomeration of activated pozzolan particles are visible in the container



Figure 4. Portland Cement Type II, powdered raw Sirjan natural pozzolan, activated pozzolan with Ca(OH)<sub>2</sub> and NS

## 3. Results and Discussion

### 3.1. XRD Results of Activated Pozzolans

XRD patterns in Figure 5 show that a new and common peak in the  $2\theta$  between 29° and 30° in activated pozzolans has took form. This peak is common between calcium silicate hydrate (C-S-H) and calcite. C-S-H and calcite have their

prominent peak in the 2θ between 29° and 30°. To distinguish between C-S-H and calcite thermogravimetric analysis (TGA) is required. Calcite is formed during carbonation and C-S-H is formed through chemical reactions between SiO<sub>2</sub> and Ca(OH)<sub>2</sub> and CaO in the mixture. Figure 5 shows that the albite and quartz peaks –which are the prominent crystalline structures of raw Sirjan pozzolan - have reduced in activated pozzolans meaning that pozzolan particles have corroded and new particle structures have formed during the activation procedure. The activation temperature affects the crystalline structure of activated pozzolans. The XRD pattern of activated pozzolan at 55°C shows more peaks than XRD patterns of raw pozzolan and activated pozzolans at 65°C and 75°C. Also, there are more peaks in the XRD patterns of activated pozzolan at 65°C compared to activated pozzolan at 75°C.

### 3.2. TG Analysis of Activated Pozzolans

Figure 6 shows the differential thermogravimetric (DTG) curves of activated pozzolans. The DTG curves show the differential of the mass with respect to temperature against the temperature axis. Two main peaks are observed in the curves. The first peak is between 100°C and 200°C which shows the dehydration of C-S-H [2]. De-carbonation zone is also shown on the curves although it does not show all the carbonates. Table 2 shows the mass reduction percentage for dehydration of C-S-H. It shows that more C-S-H is formed at 75°C. Figure 6 and Table 2 show that less C-S-H is formed at 65°C. Also, the curves approve the consumption of calcium hydroxide during the activation process since there is no peak in the temperature between 400°C and 500°C. Through TGA and XRD tests, it was showed that C-S-H has took form during activation. This C-S-H is called “pre C-S-H” here. The nomenclature for pre C-S-H is to distinguish between C-S-H which takes form during activation and the C-S-H which takes form through hydration of the pastes due to the curing of the pastes in water. Raw natural pozzolan has a definite pozzolanic power. When raw pozzolan is activated, some of this power is used in order to consume the added Ca(OH)<sub>2</sub> and produce pre C-S-H. It is worth noting that NS increases the Ca(OH)<sub>2</sub> consumption during activation of pozzolan, nevertheless, most of the Ca(OH)<sub>2</sub> consumption is due to the reaction with raw pozzolan since the amount of NS is low. There is a remained pozzolanic power of raw pozzolan after activation which uses the Ca(OH)<sub>2</sub>, produced during hydration of C<sub>3</sub>S and C<sub>2</sub>S phases of Portland cement, and produces C-S-H in the paste. In order to define the reactivity of added materials including raw pozzolan, raw pozzolan incorporating NS and activated pozzolans, the definitions above will be used in TG analysis of pastes.

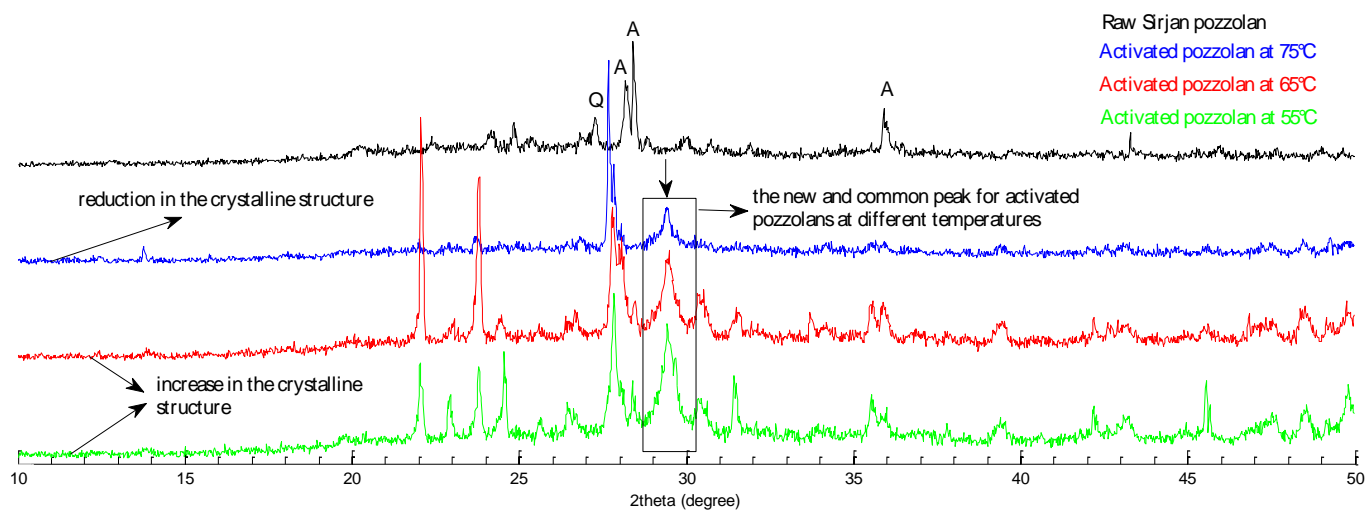


Figure 5. Raw pozzolan and activated pozzolans at different temperatures (A: albite and Q: quartz)

Table 2. Mass reduction (%) in TG analysis

Sample	100°C-200°C
activated pozzolan at 55°C	10.92
activated pozzolan at 65°C	9.25
activated pozzolan at 75°C	13.67

### 3.3. Laser Particle Analysis

Figure 7 shows the frequency and cumulative curves of raw and activated pozzolans particle sizes. Table 3 shows the geometric mean particle size and specific surface area of the materials. The figure shows that bigger particles have formed during the activation even at 65°C which has a geometric mean less than raw pozzolan. The activated pozzolan at 65°C particle size frequency curve, shows that the frequency of particles between 3.58μm and 23.9μm has increased and the frequency between 530nm and 3.58μm has reduced meaning that bigger particles have formed. At 75°C bigger particles compared to others have took form. The frequency curve shows that the frequency of particles smaller than

23.9 $\mu\text{m}$  has reduced meaning that smaller particles have formed structures with bigger sizes. It is expected that there be a direct relation between temperature and geometric mean but at the temperature of 65°C the geometric mean has decreased compared to other activated pozzolans. It is worth note that the same procedure and condition have been performed for activation of pozzolan at this temperature. The reason for this is that although the  $\text{Ca}(\text{OH})_2$  has been used completely there was not enough time for crystallization and formation of bigger particles.

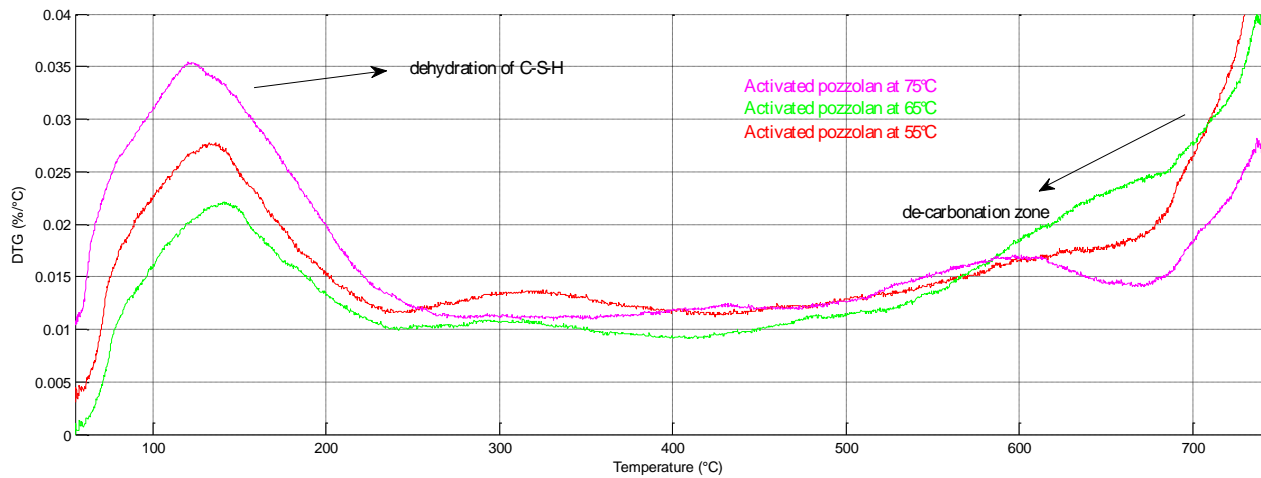


Figure 6. DTG curves of activated pozzolans

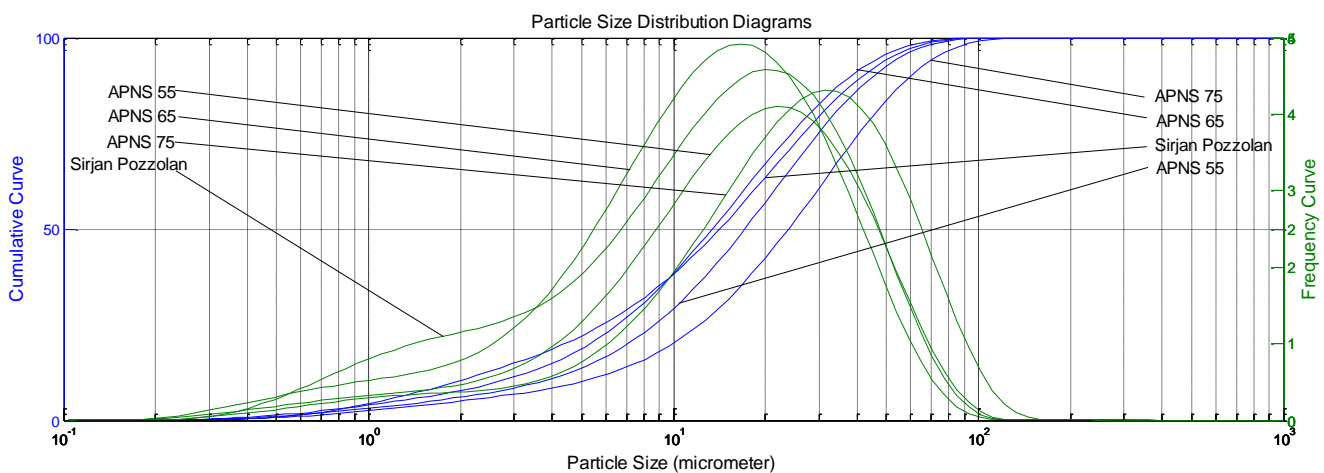


Figure 7. Particle size distribution diagrams of raw and activated pozzolans

Table 3. Geometric mean and specific surface area of raw and activated pozzolans

	Geometric mean ( $\mu\text{m}$ )	Specific surface area( $\text{cm}^2/\text{gr}$ )
Raw Sirjan pozzolan	11.42	11705.94
Activated pozzolan at 55°C	14.50	9273.16
Activated pozzolan at 65°C	11.26	11578.02
Activated pozzolan at 75°C	20.03	7102.3

### 3.4. SEM Images of Raw and Activated Pozzolans

SEM images of raw and activated pozzolans are shown in Figure 8, 9, 10 and 11. Pozzolan particles have asymmetric shape with sharp edges and thin flat surfaces with different particle sizes. The images of activated pozzolans in smaller magnifications show the formation of rough surfaces. The microstructure of activated pozzolans are different showing the effect of temperature in activation of pozzolan. Sheet-like structures and network patterns are formed during the activation of pozzolans. Bigger sheets are formed at 75°C compared to 55°C and 65°C. The microstructure of the activated pozzolan at 75°C and 55°C are similar. At 65°C the formed structure is different from other activated pozzolans and the sheets are smaller. Also, more flat surfaces formed at 75°C and 55°C compared to the surfaces formed at 65°C. Based on the XRD patterns and DTG curves it can be deduced that the formed structures are C-S-H since they show no specific crystalline structure. The images show different structures for C-S-H at different temperatures. Also, sheet-like structure of calcium silicate hydrate has been reported in [11].

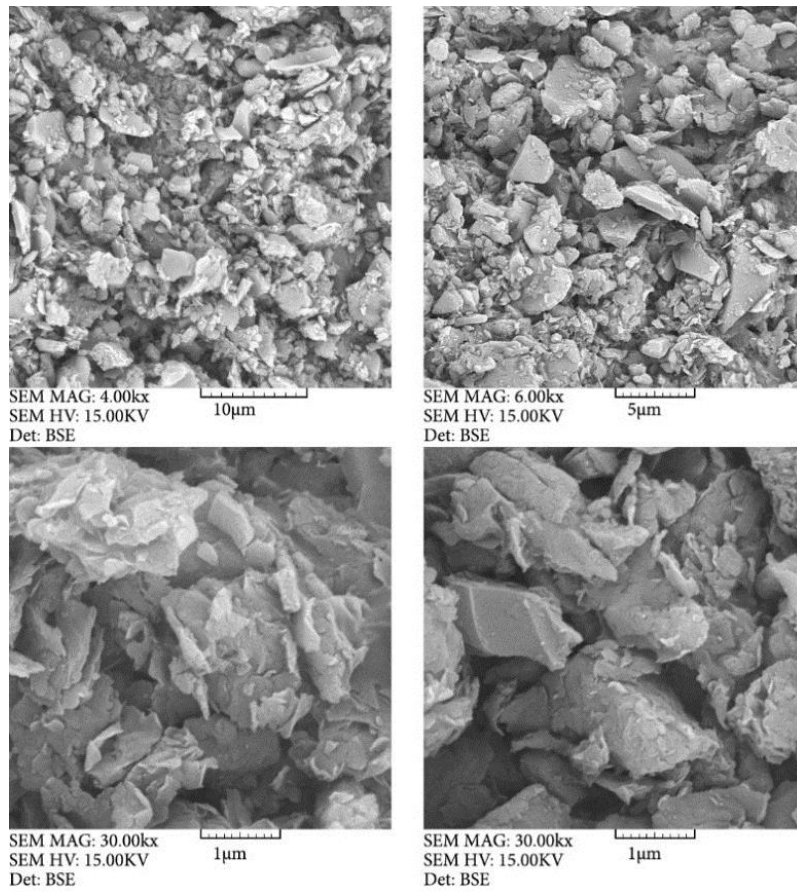


Figure 8. SEM images of raw natural pozzolan powder

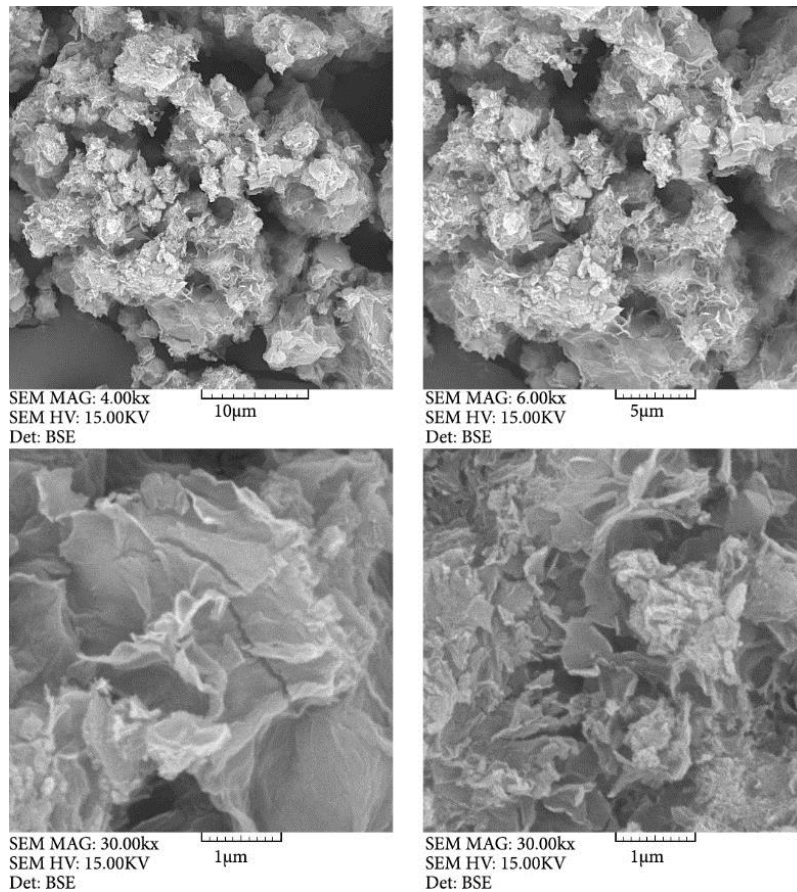


Figure 9. SEM images of activated pozzolan at 55°C

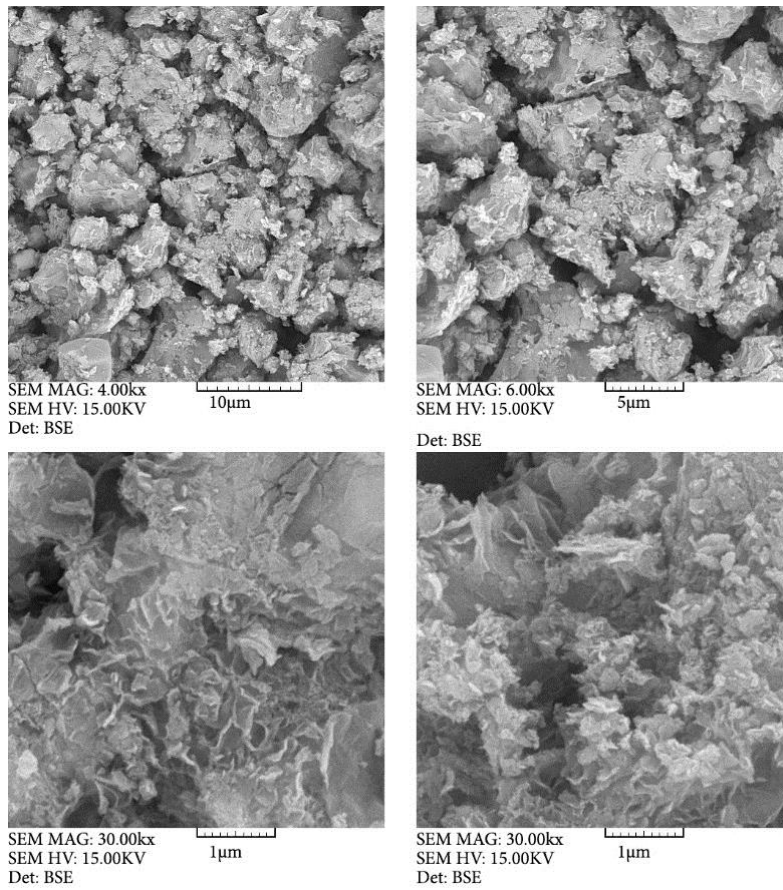


Figure 10. SEM images of activated pozzolan at 65 °C

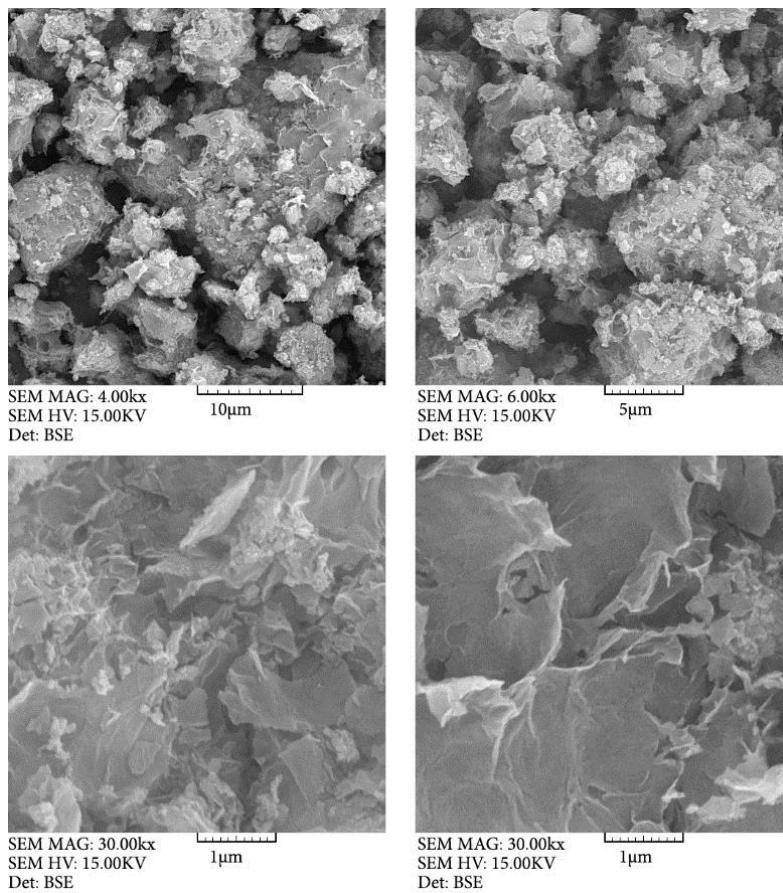


Figure 11. SEM images of activated pozzolan at 75 °C



### 3.5. TG Analysis of the Pastes

The DTG curves of the pastes cured for 7 and 28 days are shown in Figure 12 and 13. The data in Table 4 shows the percentage of mass reduction for the pastes for different curing ages between 100°C and 200°C (dehydration of C-S-H) and between 430°C and 500°C (decomposition of Ca(OH)<sub>2</sub>).

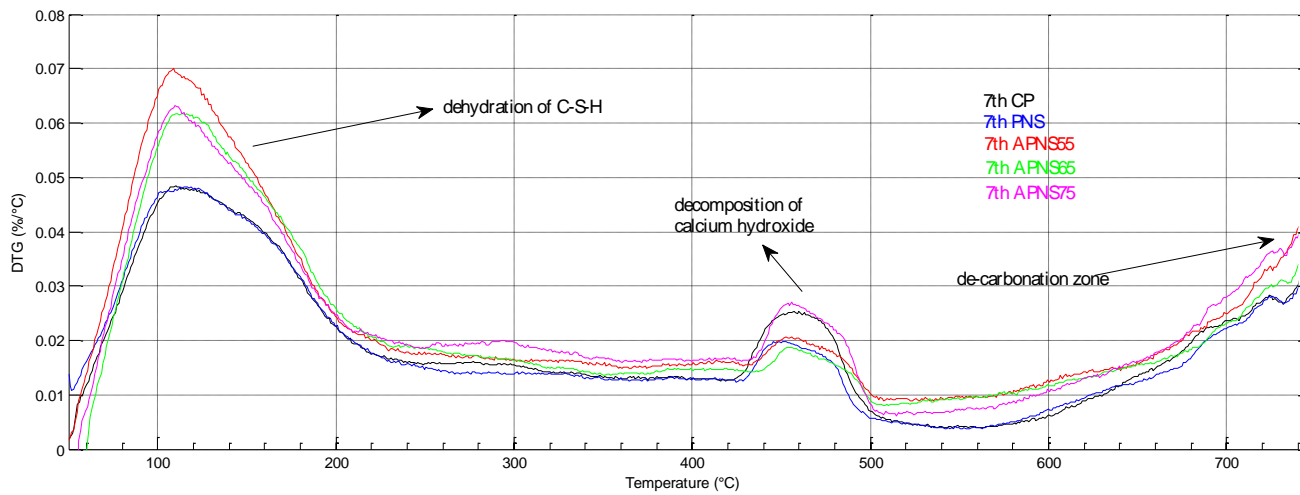


Figure 12. DTG curves of the pastes cured for 7 days in water

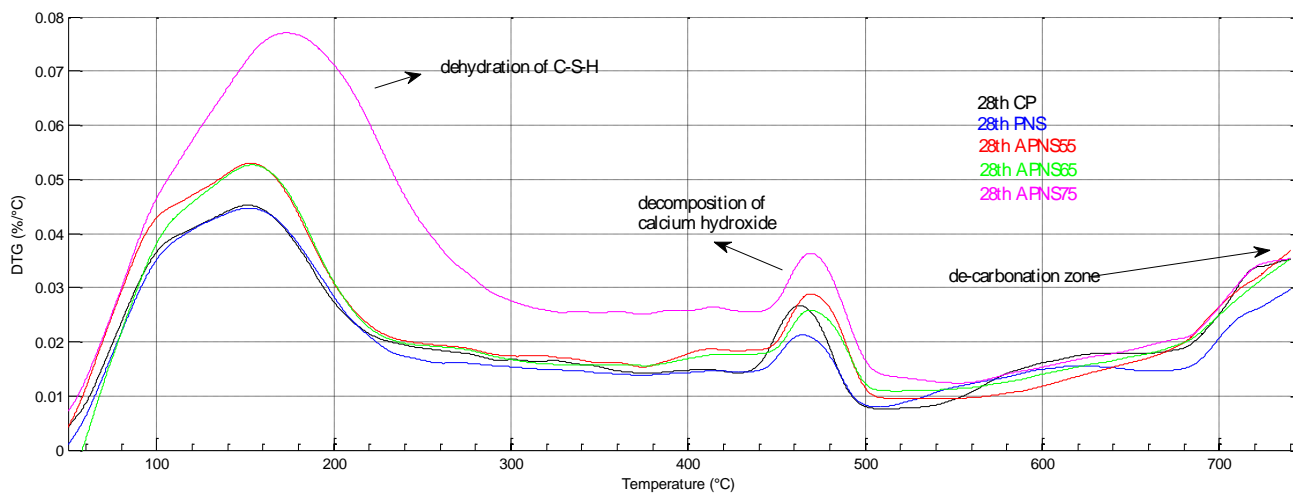


Figure 13. DTG curves of the pastes cured for 28 days in water

Table 4. Mass reduction (%) in TG analysis at 7th and 28th day

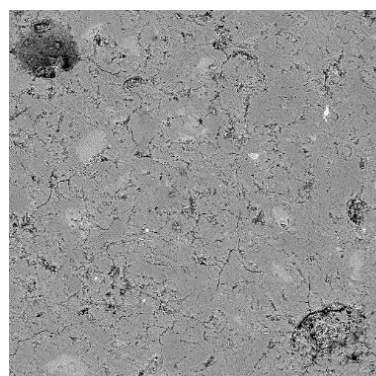
Sample	7 <sup>th</sup> day		28 <sup>th</sup> day	
	100°C-200°C	430°C-500°C	100°C-200°C	430°C-500°C
CP	4.06	1.40	17.79	7.00
PNS	4.06	1.08	17.78	6.11
APNS55	5.16	1.21	20.66	8.32
APNS65	5.00	1.09	20.32	7.70
APNS75	4.74	1.47	30.04	10.77

DTG Curves of the 7th day show that the pozzolanic reactivity of raw pozzolan was slower than other samples except for APNS75. PNS curve shows that Ca(OH)<sub>2</sub> consumption has increased compared to CP showing the addition of NS effect on Ca(OH)<sub>2</sub> consumption. APNS75 has the highest peak of Ca(OH)<sub>2</sub> showing that the used pozzolanic power is a lot more than the remained pozzolanic power. APNS55 and APNS65 curves show that the pozzolanic reactivity has increased at 7th day. Although some of the pozzolanic power has been used during activation at 65°C, its remained pozzolanic power has a similar effect as PNS at 7th day showing the effect of activation on pozzolanic reactivity. C-S-

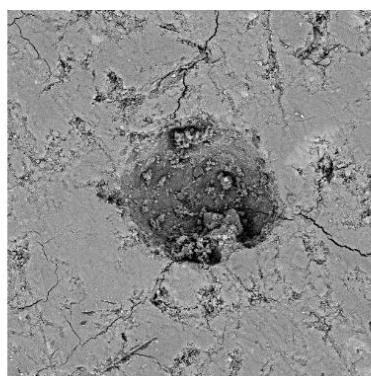
H peak is almost the same for CP and PNS. APNS55 and APNS65 show almost the same peak for C-S-H. The highest peak of C-S-H is for APNS75. The same results are true for the DTG curves of the pastes at 28th day. However, raw pozzolan reactivity was higher at 28th day and the  $\text{Ca}(\text{OH})_2$  peak has reduced showing that the pozzolanic reactivity of raw pozzolan is slow and the reactivity of raw pozzolan occurs in long term. For APNS samples C-S-H peak is due to both pre C-S-H and C-S-H. APNS75 seems to show more pre C-S-H than C-S-H due to its less pozzolanic reactivity than other samples and the explanation above. PNS and CP sample show C-S-H which has took form during hydration of cement. From TG data of activated powders and pastes in Table 3 and Table 4, a relation between the amount of pre C-S-H produced during activation and the amount of  $\text{Ca}(\text{OH})_2$  in the pastes, can be deduced: the more pre C-S-H produced during activation, the more  $\text{Ca}(\text{OH})_2$  existence in the paste. In other words: the more used pozzolanic power during activation, the less remained pozzolanic power and consequently more existence of  $\text{Ca}(\text{OH})_2$  in the paste.

### 3.6. SEM Images and EDS Analysis of the Pastes

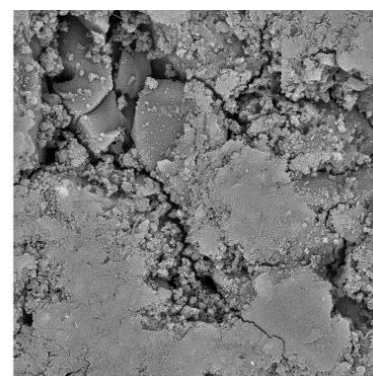
SEM backscattered electron (BSE) images of the pastes cured for 28 days in water are shown in Figure 14 and Figure 15. CP images in all magnifications show a lot of cracks and pores in the microstructure. The unreacted pozzolan particles are present in the CP paste showing that its reactivity is too slow. Addition of NS to the paste has improved the microstructure of the paste. There's less pores and cracks in the paste showing more formation of C-S-H due to the increase in consumption of  $\text{Ca}(\text{OH})_2$  in the paste and more production of C-S-H. However, there is still cracks and small pores in the paste in bigger magnifications. The SEM images of APNS samples are different. APNS55 and APNS75 show considerable improvements in their microstructures. However, activated pozzolan at 65°C did not have similar effects of activated pozzolans at 55°C and 75°C. Pores has been filled the same as in PNS sample but there is still cracks in the microstructure. Nevertheless, the improvement is better than PNS sample. This is related to microstructure of activated powders. Sheet-like structures were formed during the activation, however, bigger and more flat sheets were formed at 75°C and 55°C compared to 65°C. The reason for microstructural improvement in APNS samples is the existence of pre C-S-H in activated pozzolans. This shows the pore-filling effect of activated pozzolans.



SEM MAG: 400x  
SEM HV: 15.00KV  
Det = BSE

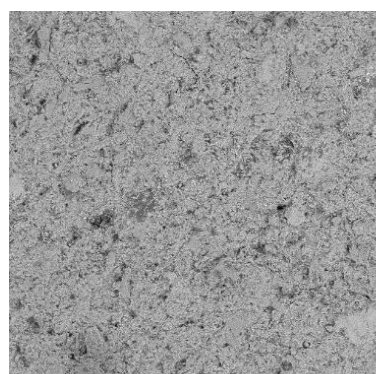


SEM MAG: 1.00kx  
SEM HV: 15.00KV  
Det = BSE

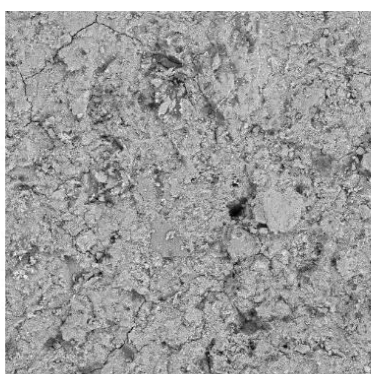


SEM MAG: 6.00kx  
SEM HV: 15.00KV  
Det = BSE

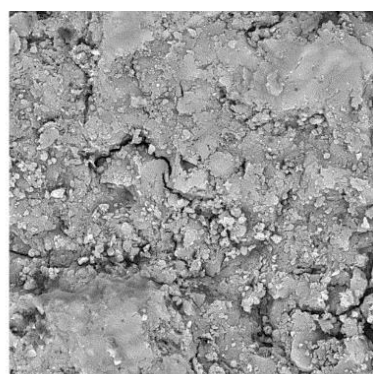
#### SEM images of CP sample



SEM MAG: 400x  
SEM HV: 15.00KV  
Det = BSE



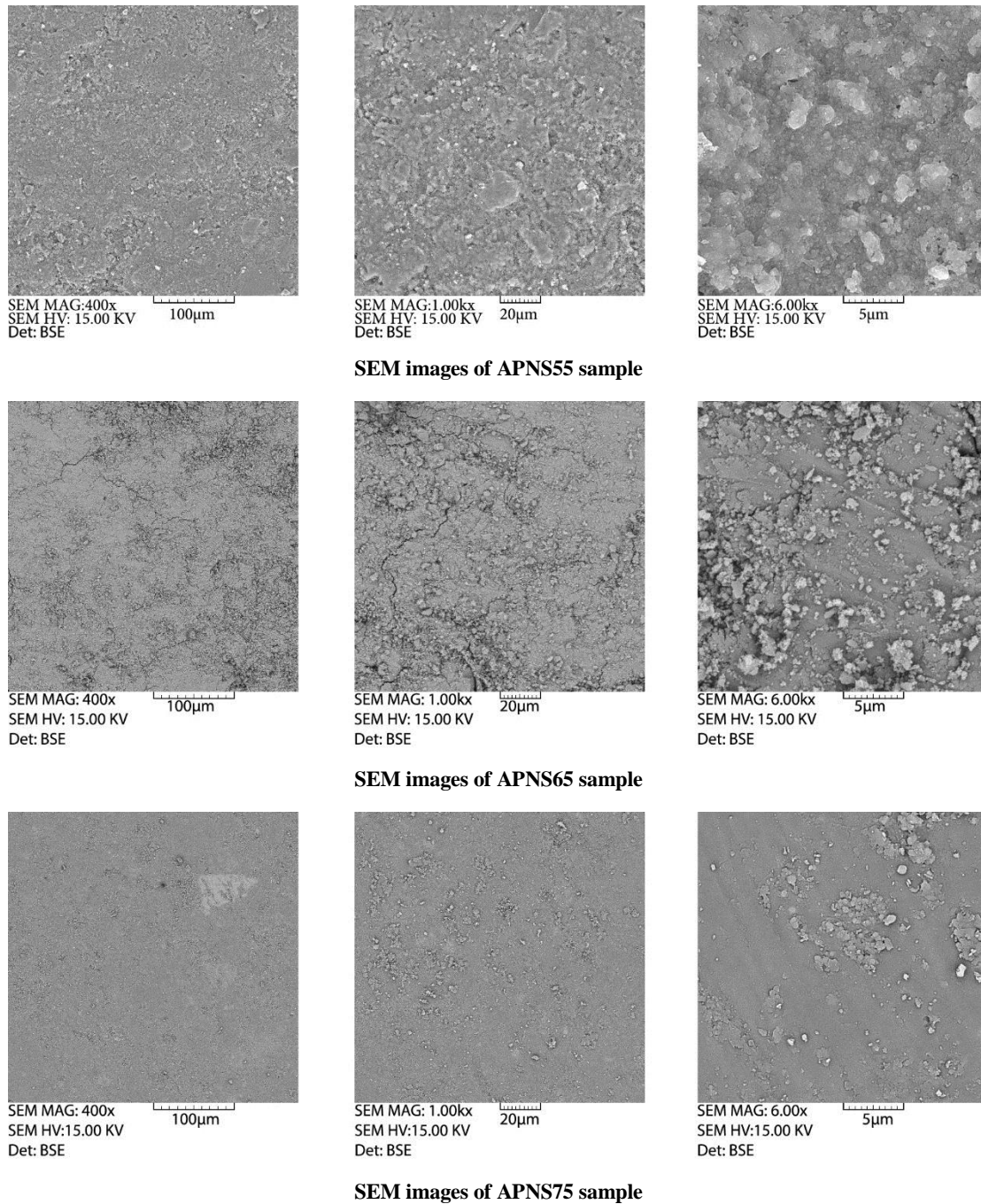
SEM MAG: 1.00kx  
SEM HV: 15.00KV  
Det = BSE



SEM MAG: 6.00kx  
SEM HV: 15.00KV  
Det = BSE

#### SEM images of PNS sample

Figure 14. SEM images of CP and PNS samples cured in water for 28 days



**Figure 15. SEM images of APNS samples cured in water for 28 days**

The results of EDS analysis of the pastes are shown in Table 6 with atomic percentage for each element with Ca/Si and Ca/(Si+Al) ratios. The results show that Ca/Si ratio for APNS55 and APNS75 with a similar improved microstructure, have the same Ca/Si ratio of about 1.43. The CP sample Ca/Si ratio is 0.78. PNS and APNS65 with a similar microstructural improvement have a close Ca/Si ratio of 0.97 and 1.06, respectively. The results are also true for Ca/(Si+Al) ratio.

**Table 6. EDS analysis of the paste samples cured for 28 days in water with atomic percentage (%)**

Sample	Ca	Si	Na	K	Al	Ca/Si	Ca/(Si+Al)
CP	37.22	47.57	1.64	1.05	12.5	0.78	0.61
PNS	43.11	44.05	-	1.85	9.91	0.97	0.79
APNS55	54.78	38.06	0.61	1.18	5.35	1.43	1.26
APNS65	44.48	41.92	-	0.53	10.30	1.06	0.85
APNS75	53.51	37.25	-	1.53	7.68	1.43	1.19

## 4. Conclusions

The main results based on the experiments carried out in this study can be concluded as follows;

- XRD patterns and DTG curves showed that C-S-H has took form during activation of pozzolan with  $\text{Ca}(\text{OH})_2$  and NS which was called “pre C-S-H”. The nomenclature was to distinguish between C-S-H which takes form during activation process and the C-S-H which takes form during hydration of the pastes in the curing ages.
- SEM images of activated pozzolans showed different microstructures for activated pozzolans at different temperatures showing the effect of temperature. The images showed that sheet-like structures were formed in activated pozzolans. At 75°C and 55°C the sheets were bigger and more flat but at 65°C the sheets were smaller.
- Laser particle analysis results showed decomposition of raw pozzolan into smaller particles and formation of bigger particles in all activated pozzolans. However, the frequency of bigger particles in activated pozzolan at 75°C was more than other activated pozzolans. Also, bigger particles compared to raw pozzolan and other activated pozzolans were formed at this temperature.
- TGA data of activated powders and pastes showed that the existing  $\text{Ca}(\text{OH})_2$  in the paste is proportion to the production of pre C-S-H during activation of pozzolans: the more used pozzolanic power during activation (more production of pre C-S-H), the less remained pozzolanic power and consequently more existence of  $\text{Ca}(\text{OH})_2$  in the paste.
- SEM images of the pastes cured for 28 days in water showed the filling effect of activated pozzolans which was due to existence of pre C-S-H. The microstructural improvement in APNS75 and APNS55 were much better than APNS65. This was due to more existence of pre C-S-H in activated pozzolans at 55°C and 75°C compared to activated pozzolan at 65°C. The improvement in APNS65 was better than PNS sample.
- EDS analysis of the pastes showed that APNS75 and APNS55 with a similar microstructural improvement had a close and higher Ca/Si and Ca/(Si+Al) ratios than other samples. Also, APNS65 and PNS sample with a similar microstructural improvement showed a close Ca/Si and Ca/(Si+Al) ratios.
- Although the microstructural improvement in APNS75 was better than other samples, there was more  $\text{Ca}(\text{OH})_2$  in the paste. However, with a similar microstructural improvement in APNS55 there was less  $\text{Ca}(\text{OH})_2$  in the paste compared to APNS75. This shows despite total  $\text{Ca}(\text{OH})_2$  consumption in both activated pozzolans at 55°C and 75°C and more activation time at 55°C, the remained pozzolanic power of activated pozzolan at 55°C was higher than activated pozzolan at 75°C. This shows the effect of activation temperature on the remained pozzolanic power of activated pozzolan.

## 5. Acknowledgement

This paper is dedicated to S. Zahra Razavi Khoosfi for her special helps and support to this study.

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