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Thermodynamical and Experimental Study of the Effects of Ball Clay-Silica Fume Combination on the Hydration and Strength

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ABSTRACT: The production of ordinary Portland cement (OPC) as the most expensive constituent of concrete is associated with destructive environmental effects and significant energy consumption. Thus, the use of supplementary cementitious materials such as Natural or synthetic pozzolans is a fundamental solution that affects the kinetics of hydration of cementitious materials. In this article, to evaluate the effect of ball clay in the presence or absence of silica fume on the performance of cementitious materials, compressive strength and ultrasonic tests were performed at replacement weight percentages of 0, 10 and 20% for ball clay and 0 and 7% for silica fume. Also, thermodynamic modeling during hydration was carried out based on the Gibbs free energy minimization. The results showed that mere presence of ball clay does not have a positive effect, but it can improve the structure and strength of cement in the presence of silica fume, since it resulted in complete consumption of portlandite and consequently increasing C-S-H gel. In addition to examine the type and volume of phases formed during hydration, the optimum substitution of ball clay and silica fume was studied. Accordingly, the best overall performance of ball clay was observed in the 10% substitution in the presence of 7% silica fume.

Keywords: Ball Clay, Compressive Strength, Hydration, Thermodynamic Modeling, Ultrasonic Pulse Velocity.

INTRODUCTION

Concrete is of the selected materials in the construction industry and is also the most widely used material after water. This shows the important role of cement. Given the extent of construction activities and the growing need for cement consumption, access to high quality cement at reasonable cost has always been an important issue. Meanwhile, it should be noted that cement production requires high amounts of energy, and thus the cement industry is among polluting industries as almost one ton of carbon dioxide is produced per ton of cement. Therefore, pollutant emissions by burning fuel of furnaces in cement factories are released into the environment (Gartner, 2004). Cement manufacturers are seeking to reduce both the amount of energy required to produce cement and also the amount of gases released in the process of cement production. For this

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purpose, they are seeking for alternatives for cement (Juenger et al., 2011). The substance that is intended to replace part of the cement should reduce the negative factors such as the need for limestone, CO_2 production, high energy consumption and costs. On the other hand, it should lead to an increase in cement properties such as strength and durability. Meanwhile, natural and synthetic pozzolans and minerals can be considered as an alternative for cement due to their positive effects on cement properties. Natural or synthetic pozzolans affect the microstructure and hydration kinetics of cementitious materials.

Ball clay is one of the natural pozzolans. Ball clay is a fine-grained sedimentary clay with considerably high plasticity. It turns white at high temperatures of about 1,200 °C in the presence of oxygen. Ball clay has a natural origin and is also known as a secondary clay, meaning that it has been transported by natural weathering away from its place of origin. Its natural color can vary from pale yellow to black. The color change is dependent on the presence of other minerals or those deposited in ball clay (Hosterman, 1949; Andreola et al., 2009; Purohit et al., 2016). Ball clays are primarily used in the ceramic and refractory industries as they provide resistance, plasticity and performance to ceramic parts. Despite its positive pozzolanic properties, ball clay has not been considered to be used in the manufacture of cement and concrete products. This is despite the fact that natural and synthetic pozzolans like ball clay and silica fume are available abundantly. By substituting a proper portion of them in cement, in addition to reducing cement consumption and improving the mechanical properties and durability, they may play an important role in sustainable development and maintaining favorable environmental conditions.

Like other chemical systems, physiochemical processes of cement hydration are governed by the laws of thermodynamics. Since thermodynamics concerns the initial and final states of systems, it is capable of calculating a set of stable phases or a combination of aqueous phases of hydrated cement paste or the cement paste in equilibrium with its surrounding. Theoretical calculations of stable phases are flexible and are comprehensive enough to account the complexities of cement systems. In recent decades, the basic aspects of cement chemistry have received much attention. The recent developments in the field of cement technology have led many research groups around the world to develop thermodynamic models with the aim to examine the possible reaction mechanisms during cement hydration and the complexities of hydrated cement systems as well as improving the ability to predict their performance over time.

One of the problems with laboratory studies is implementation of all the factors affecting hydration to obtain results in the shortest time. Hence, along with laboratory studies, theoretical modeling and simulation as well as new methods with lower costs have received much attention to achieve reliable results in the shortest time. Thermodynamic modeling of cement hydration and aggressive environments has been studied on some materials (Zajac et al., 2014; Lothenbach et al., 2015; Martin et al., 2015; Schöler et al., 2015; Winnefeld et al., 2015; Kunther et al., 2016; Le Saoût et al., 2017; Martin et al., 2017; Tarighat et al., 2018, 2019).

Hydration process is significantly influenced by cement chemistry and microstructure so that a slight change in cement ingredients causes significant differences in the hydration products. That is why use of cement supplements affects the amount and type of hydrates formed and, consequently, the volume, porosity and the durability of concrete systems. To the best of our knowledge, ball clay has not been used in the manufacture of concrete and there is no information on its effects on mechanical properties and durability of concrete. In addition to experimental studies in this work, the impact of silica fume and ball clay on the compressive strength of concrete and wave velocity in concrete is investigated by thermodynamic modeling. The thermodynamic model is used to study the behavior of the cement paste and the impact of ball clay on cement hydration in the presence and absence of silica fume.

In the present study, in addition to experimental studies on the impact of silica fume and ball clay on the cement hydration and concrete compressive strength, thermodynamic method was used for modeling the behavior of hardened cement paste in order to better understand the cement hydration as well as the impact of ball clay in the presence and absence of silica fume.

MATERIALS AND METHODS

Materials

Portland cement type CEM I 42.5 N was used in this study. The used ball clay was processed after milling at a constant temperature of 750 °C for 3 hours. The chemical composition of cement, silica fume and ball clay (XRF results) is presented in Table 1. The aggregates used in this study included natural sand and twice-washed gravel prepared from mines around Tehran. The ratio of water to cementitious materials was 0.45 and the experiments were conducted at room temperature (25 °C). Table 2 shows the characteristics of the specimens used in thermodynamic modeling.

Characterizations of Ball Clay

XRF, PSA (particle size analysis), DTA (differential thermal analysis) and TGA (thermal gravimetry analysis) tests were used to identify the physical and chemical behavior of the ball clay.

In Figure 1 the particle size distribution of ball clay used in this study is presented. As it is clear, particles ranging in size from 0.4 to 2000 µm while 95% of particles are less than 100 µm which are finer than OPC particles. The finer particle size of ball clay is beneficial in filler effects between cement particles. Also, the information derived from the PSA indicated that surface area of ball clay was 1.23 m^2/g.

In Figure 2, the results of the DTA test are shown for ball clay. The DTA test is a measure of the difference in the temperature of the test sample and the control one, while both samples are heated with the same heating program. The graph peak indicates the type of thermal event that has emerged.

Table 1. Chemical composition (wt.%) and the physical characteristics of the OPC, Ball clay and Silica fume

	OPC [a]	**Silica fume** [a]	**Ball clay** [a]
SiO_2	21.32	91.7	51.94
Al_2O_3	3.83	1.2	19.45
Fe_2O_3	2.76	1.1	5.43
CaO	62.02	0.8	12.42
MgO	3.44	0.9	2.27
SO_3	2.09	0.2	0.31
Na_2O	0.12	0.7	0.93
K_2O	0.73	1	4.38
P_2O_5	0.05	0.1	0.17
TiO_2	0.44	<0.1	0.71
MnO	0.2	-	-
LOI	2.98	2.02	1.77

[a] XRF data

Table 2. Mix designation, % cement replacement

S.No	Mix designation	OPC	Silica fume	Ball clay
1	SF0 BA0	100	0	0
2	SF0 BA10	90	0	10
3	SF0 BA20	80	0	20
4	SF7 BA0	93	7	0
5	SF7 BA10	83	7	10
6	SF7 BA20	73	7	20

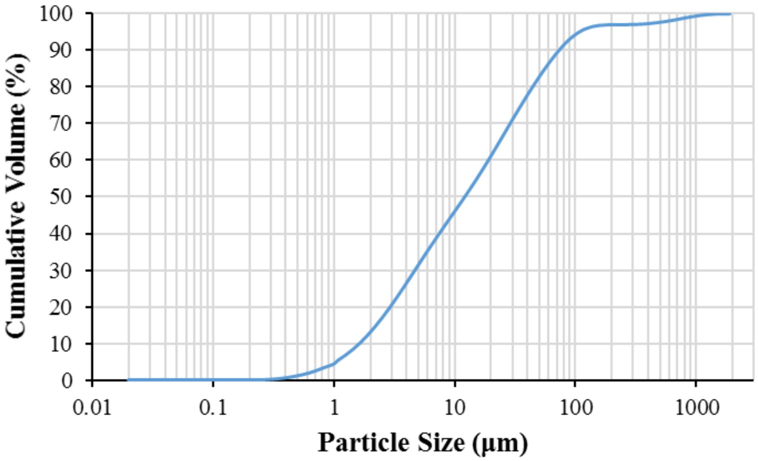

Fig. 1. Particle size distribution of ball clay used in this study

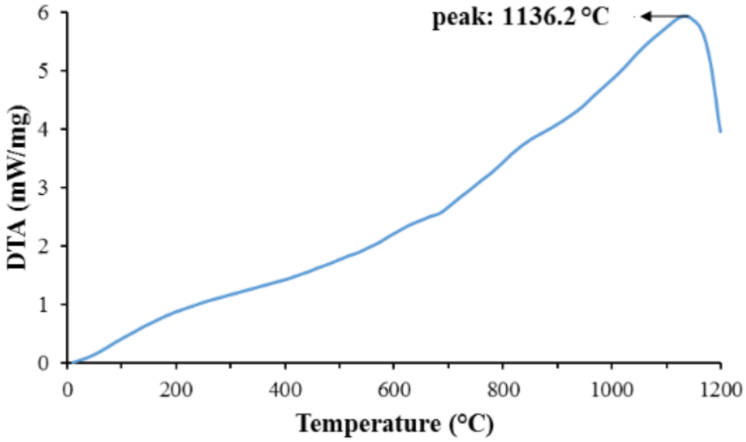

Fig. 2. The results of DTA test on the ball clay used in this study

Considering the Figure 2, the structure change of the crystal in ball clay is not shown, because the DTA curve has no sharp peak. The first change was seen at a temperature of about 1136.2 °C, which is due to the molecular outflow of water from the ball clay crystal network.

In Figure 3, the graph of TGA falls up to 640 °C with a slight moderate gradient, indicating the weight loss is due to the evaporation of surface water at lower temperatures and the loss of water of clay at higher temperatures. Afterwards, the graph falls with a sharp slope from 640 °C to about 710 °C. This weight loss is due to disordering the crystal shape and released water from ball clay structure, all result in increasing potential reaction. Then the slope of the graph decreases gently, which is probably related to carbonate and quartz minerals staying on transitional stages.

Experimental

Mix Design Specifications

Six different substitutions were used in this study. A control design was prepared without ball clay and silica fume substitution. In other mix designs, 0 and 7% cement was replaced with silica fume and 0, 10 and 20% cement was replaced by ball clay. The concrete mix proportions are illustrated in Table 3.

Preparation and Maintenance of Specimen and Tests

In this study, cubic molds with dimensions of 10 cm were used for compressive strength testing. After constructing each concrete mix design, the slump test was immediately performed to determine the fresh concrete flow. In case the slump was appropriate (a slump between 80 and 120 mm), the concrete was molded. The mold was opened after 24 h. Then, the specimens were placed in a water pond for curing to the ages of 7 and 28 days. The 90-day specimens were removed from water after 28 days and were maintained at room temperature to reach the age of 90 days. To ensure the accuracy of test results and to reduce errors, three cubic specimens were prepared for each mix design and age.

Thermodynamic Modeling

The thermodynamic approach is used to achieve a better understanding of cement hydration. Previous studies (Gartner and Jennings, 1987; Berner, 1988; Brown, 1989; Reardon, 1992) have demonstrated that cement hydration follows the basics of physical chemistry by minimizing the free energy of the system. According to these studies, thermodynamic equilibrium models have proved useful in investigating cement hydration.

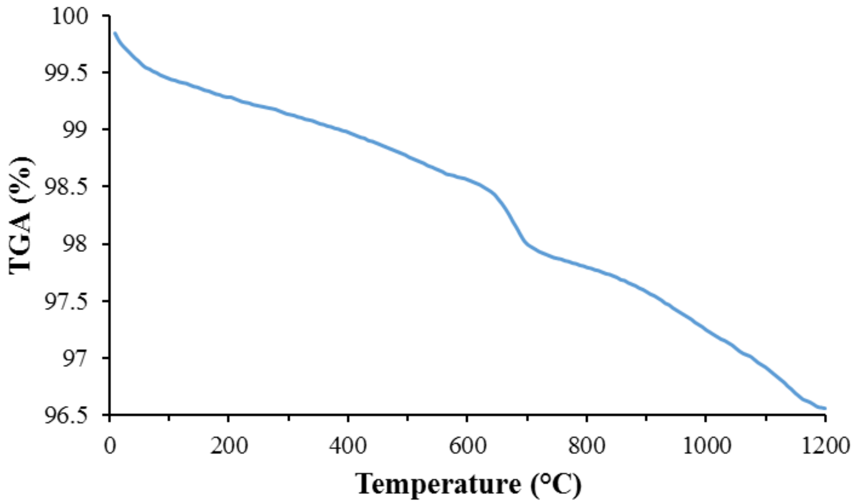

Fig. 3. The results of TGA test on the ball clay used in this study

Table 3. The concrete mix proportions

Binder	400 g
Water	180 g
Coarse aggregate	1274 g
Fine aggregate	546 g

Superplasticizer was used 0.5% by weight of binder.

Today, thanks to the increase in computational power, software tools for thermodynamic calculations are easily accessible and researchers are more tempted to use them in order to investigate the complexities of cement systems. Actually, phase equilibria studies help to understand the behavior of complex reactions of the hydrated cement. It can also help in the interpretation of the experimental observations. Due to the use of cement additives in mixed cement, it is undoubtedly a great help to better understand the complex phase equilibrium in new concrete systems (Lothenbach et al., 2010).

In this study, thermodynamic modeling was performed using Gibbs free energy minimization with the help of GEM software package. Thermodynamic modeling can be used for cement systems to calculate stable phases and allows prediction of the composition of hydrated phases under different conditions. It can help to further optimize complex multi-component cement systems while reducing time and cost.

The cement database cemdata 14 was used in the thermodynamic modeling. This database is based on Nagra/PSI geochemical database and contains thermodynamic data (dissolution products, Gibbs free energy, enthalpy, entropy, heat capacity) for a number of cement phases such as calcium silicate hydrate and AFm, Aft and hydrotalcite phases.

RESULTS AND DISCUSSION

Compressive Strength

In this study, compressive strength tests were performed on 10 cm cubic specimens after 14, 28 and 90 days. Figures 4 and 5 and Table 4 show the results for different ages and different substitution percentages. As can be seen, replacing cement with 7% silica fume and 10% ball clay improves the compressive strength (up to 12%). The role of ball clay and silica fume is important for two reasons. First, pozzolanic reactions leading to an improved bond between the hydrated cement matrix and aggregate in the mix. This improved bond is due to the conversion of the calcium hydroxide (portlandite), which tends to form on the surface of aggregate particles, into calcium silicate hydrate (C-S-H gel) due to the presence of reactive silica (Cheng-Yi and Feldman, 1985). Second, these pozzolanic materials act as a filler and can fill the pores in the concrete structure due to very fine particles. In case of natural pozzolan ball clay, the impact of the first factor becomes apparent slowly with aging. In the presence of active silica fume pozzolan and ball clay, the non-hydrated ball clay granules can play a role in load-bearing.

By increasing the substitution percentage of ball clay from 0% to 10% and 20%, a marginal change in the compressive strength is observed. Therefore, ball clay alone does not have a positive effect on the compressive strength.

Comparing SF7BA0 and SF7BA10 mix designs, the compressive strength of the concrete containing 7% silica fume and 10% ball clay is higher than that only containing 7% silica fume (Figure 5). Improved compressive strength of concrete in the presence of ball clay is due to the conversion of the calcium hydroxide into calcium silicate hydrate which will be shown in thermodynamic modeling as well as the filling role of ball clay particles. According to the results, the compressive strength of all concrete specimens increases with aging. This shows the progress and completion of the hydration process and pozzolanic reactions with aging. As a result, the formation of more C-S-H gel leading to an increase in the compressive strength.

Comparing SF7BA10 and SF7BA20 shows that the compressive strength decreases with increasing the ball clay substitution from 10 to 20% of the cementitious materials (Figure 5). This reduction in the compressive strength is due to high levels of cement replacement in the mix design containing 7% silica fume and 20% ball clay, leading to an increase in the number of non-hydrated ball clay granules and the formation of more pore solution in this mix design and reduction of C-S-H gel which will be indicated in thermodynamic modeling. The compressive strength reduction is visible at the ages of 14 and 28 days. However, at the age of 90 days, this decline is slightly reduced as the pozzolanic role of ball clay is activated.

Table 4. Compressive strength of concrete samples (MPa)

S. No.	Mix designation	Compressive strength (Mpa) 14 days	28 days	90 days
1	SF0 BA0	25.4 (0.6)	29.9 (0.6)	34.6 (1.1)
2	SF0 BA10	24.7 (1.4)	25.1 (0.8)	32.5 (0.3)
3	SF0 BA20	24.2 (0.3)	26.8 (1.8)	33.4 (0.6)
4	SF7 BA0	27.4 (1.9)	33.5 (0.5)	34.9 (0.8)
5	SF7 BA10	27.7 (1.4)	33.7 (0.5)	36.7 (1.3)
6	SF7 BA20	26.3 (0.4)	29.5 (1.2)	36.0 (0.9)

Results are average of three replicates and standard deviations are shown in bracket

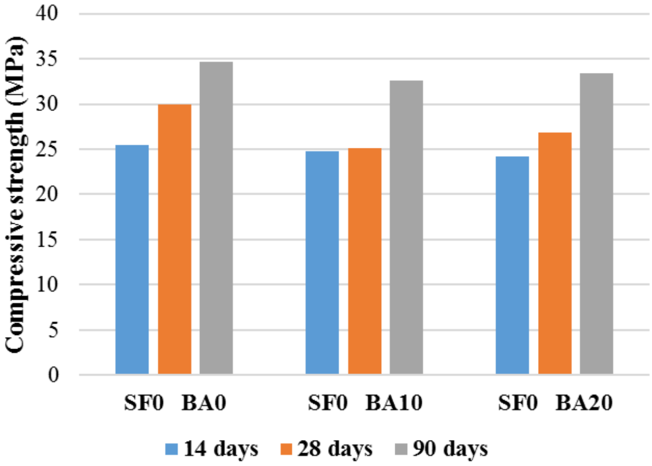

Fig. 4. Comparison of compressive strength of concrete samples at the age of 14, 28 and 90 days with different ratios of Ball clay

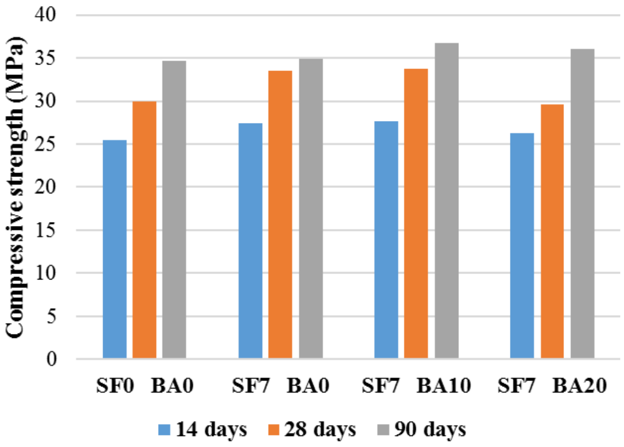

Fig. 5. Comparison of compressive strength of concrete samples at the age of 14, 28 and 90 days with different ratios of Ball clay in the presence of 7% silica fume

Cement substitution with appropriate levels of silica fume increases the compressive strength than the control specimen (Kadri and Duval, 1998; Bhanja and Sengupta, 2003; Pradhan and Dutta, 2013). This is mainly due to the aggregate-paste bond improvement and forming a less porous and more homogenous microstructure in the interfacial region because silica fume acts mainly at the interface paste-aggregate, where there is a higher concentration of calcium hydroxide and greater porosity than in paste (Bentar et al., 1987; Gleize et al., 2003; Siddique and Khan, 2011).

The most important feature of silica fume is ultra-small size of its particles which significantly accelerates its pozzolanic activity. Silica fume and ball clay particles are located between the coarse cement particles and fill the space between the cement particles due to hydration.

Ultrasonic Testing

In this project, ultrasonic tests were performed on 10 cm cubic specimens after 28 days according to standard ASTM C597. The results of this experiment are shown in Figures 6 and 7 and Table 5.

This is non-destructive testing of concrete and is generally carried out for measuring the velocity of waves in the concrete to determine the extent of its homogeneity and for detecting the presence of cavities, joints and cracks in the concrete, changes in the concrete properties with time (e.g. hydration) or changes resulting from environmental effects such as fire, ice, chemical attacks as well as determining the quality of the concrete. Obviously, the higher velocity of ultrasonic waves reflects less cavities and pores in the concrete. In other words, the particles are closer to each other and thus the waves pass the length in a shorter time. As a result, the mechanical properties and durability are improved.

Table 5. Ultrasonic wave velocity for 28 day (m/s)

S. No.	Mix designation	Ultrasonic wave velocity (m/s)
1	SF0 BA0	3865 (13)
2	SF0 BA10	4009 (20)
3	SF0 BA20	4016 (18)
4	SF7 BA0	3930 (5)
5	SF7 BA10	4015 (18)
6	SF7 BA20	4022 (24)

Results are average of three replicates and standard deviations are shown in bracket

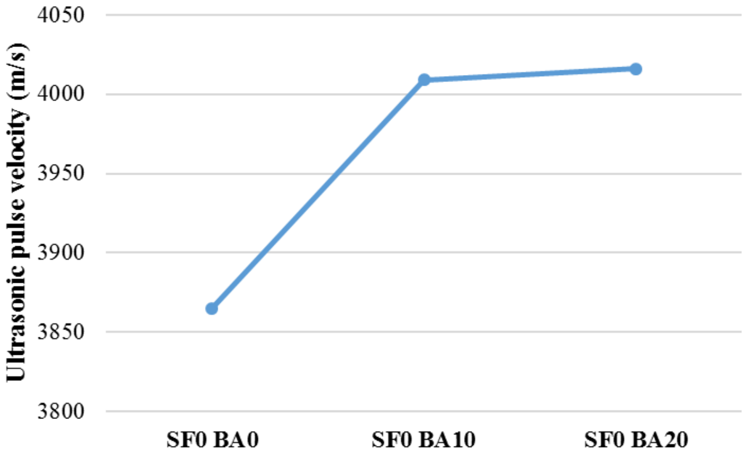

Fig. 6. Comparison of ultrasonic wave velocity with increasing substitution of Ball Clay for 28 day (m/s)

Fig. 7. Comparison of ultrasonic wave velocity with increasing substitution of Ball Clay in the presence of 7% silica fume for 28 day (m/s)

It is seen that in all mix designs, the velocity of sound is higher than that in the control. This may be attributed to the pozzolanic reactions, the filling effect of pozzolans as well as the formation of more C-S-H gel by pozzolanic reactions. This leads to a decrease in the porosity and denser concrete specimens. As a result, the waves pass the length at a higher velocity in less time. The mix designs containing both silica fume and ball clay show a higher growth. The highest wave velocity occurs in the specimen containing 7% silica fume and 20% ball clay, because ball clay and silica fume particles are located between the coarse particles of ordinary cement and fill the space between them due to hydration.

Hydration Products

The clinker phases in the cement paste are hydrated to form hydration products such as C-S-H gel, portlandite, hydrotalcite and arcanite. Ball clay introduces Mg and K to the system which upon hydration gives a hydrotalcite and arcanite, respectively. Portlandite is composed of platelet crystals which do not play an important role in the strength. The C-S-H gel with three-dimensional crystals plays the primary role in strength. Thermodynamic modeling of cement hydration shows that in the presence of silica fume by increasing ball clay level, the portlandite is consumed as far as it lost completely at about 14% substitution and consequently C-S-H gel increases. It is because when ball clay reacts, silica and alumina enter the system. Silicates react with portlandite and by its consumption create extra C-S-H gel. Upon complete consumption of portlandite, C-S-H gel is reduced and the amount of pore solution increases more sharply and because of that with increasing the amount of ball clay from 10% to 20% substitution, the compressive strength decreases as shown in Figure 5. Hydrotalcite is not changed, but arcanite is slightly increased and then negligibly decreased (Figure 8). In total, the rate of change of phases is in good agreement with experimental observations.

CONCLUSIONS

Based on the tests conducted on the concrete specimens to the age of 90 days as well the results of thermodynamic modeling, the following conclusions were derived.

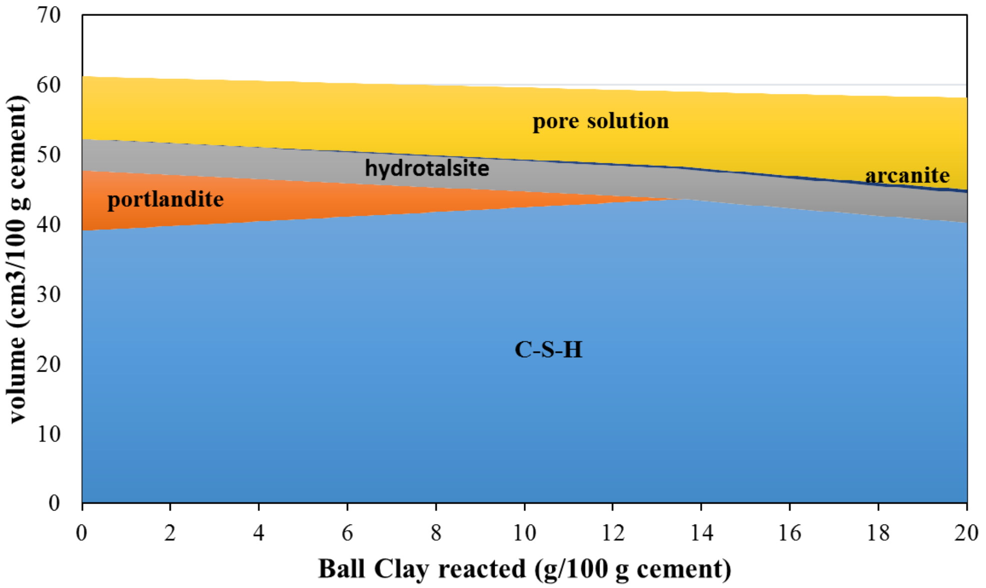

Fig. 8. Modeled phases changes with increasing substitution of Ball Clay in the presence of 7% silica fume

- The use of cementitious materials composed of Portland cement, silica fume and ball clay improved the compressive strength of the concrete (up to 12%). The mix designs containing 7% silica fume and 10% ball clay showed an improved compressive strength. This increase in the compressive strength is due to pozzolanic reactions that cause the conversion of the calcium hydroxide into calcium silicate hydrate and filling properties of silica fume and ball clay. Whereas, increasing the ball clay substitution level from 10% to 20 w% of the total cementitious materials decreased the compressive strength because the ball clay particles do not participate in the hydration process leading to formation of more pore solution.
- The use of silica fume and ball clay together significantly increased the velocity of waves flowing through the concrete specimens. This reflects the high density and quality of concrete. The maximum impact of these two materials was seen in the mix design containing 7% silica fume and 10% ball clay. The reason is that silica fume and ball clay particles are located between the coarse particles of ordinary cement and fill the space between them due to hydration.
- According to the results of thermodynamic modeling, ball clay substitution with part of cement caused consumption of portlandite and thus increased the volume of C-S-H gel during hydration process. This led to an improvement in the compressive strength of the concrete specimens. This improvement decreases at high substitution levels which is according to experimental results.

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