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Behavior of self- compacting concrete incorporating calcined pyrophyllite as supplementary cementitious material

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Abstract. The current trend of industrial concrete leans more towards the use of self-compacting concrete. These must have fresh properties well defined as fluidity, filling ability and resistance to segregation. However, to ensure the rheological stability, use mineral fines is required. In this work, powder of calcined pyrophyllite (CP) was used as cement substitution at level of 10% and 20% by weight. The interest is focused on the role played by the calcined pyrophyllite to produce SCC with reduced impact environmental.. Calcination of pyrophyllite powder was carried out at 750 °C. Its effect on the workability and mechanical properties of self-compacting concrete is analyzed. The results show that the properties of workability of SCC containing 10% of calcined pyrophyllite tested at fresh state (Slump Flow, T_{50}, passing ability and segregation resistance) are almost identical to those of the control SCC. Furthermore, the calcined pyrophyllite increases the compressive strength, tensile and flexural strength of SCC approaching without exceeding those of the control SCC. It seems that 10 % of calcined pyrophyllite is the optimum replacement rate which improves mechanical strength compared to 20%. Replacing cement with the calcined pyrophyllite aims to save cement and reduce the CO_2 emissions released during the manufacture of cement.

Key words: Calcined Pyrophyllite; Self-compacting concrete; Mechanical strength; workability.

1. Introduction

Self-compacting concrete SCC is a very fluid, stable and homogeneous concrete. It differs from ordinary concrete by its fresh properties and moldability. The SCC is formulated to obtain the optimal compromise between fluidity and resistance to segregation and to bleeding (ACI, 2003). However, to ensure the fresh stability of SCC, the use of mineral additives (fine) is required. But researches are still relevant to the understanding of the fresh and physical- mechanical behavior of SCC. The addition of finely divided siliceous materials to concrete mixes in order to improve the workability of fresh concrete and subsequently the durability of its hardened state is an established practice in modern concrete technology (Uysal & Sumer, 2011; Busari et al., 2019). Siliceous materials of low reactivity are utilized solely to improve the workability of fresh concrete deficient in fines (Ouldkhaoua et al., 2020). Generally, cementitious or pozzolanic materials are preferred because they eventually also contribute to the strength and durability of the hardened concrete (Belaidi et al., 2015; Belaidi et al., 2012; Boukhelkhal et al., 2016; Boukhelkhal et al., 2012; Aghabaglou et al., 2014; Dembovska et al., 2017; Ouldkhaoua et al., 2020).

A wide variety of natural materials as well as industrial by-products contain silica that has pozzolanic activity. To maximize its pozzolanic activity, natural pozzolans usually require grinding to cement fineness and may need to be calcined prior to its application (Krajèi et al., 2013; Mansour et al., 2012; Mansour et al., 2013). Indeed, the thermal treatment collapses the crystalline structure of the mineral by evaporating the lattice hydroxyl groups, creating a highly reactive amorphous alumina-silicate that has high affinity for reaction with cement hydration products (Tregger et al., 2010). It has been demonstrated by many researchers (Bijeljié et al., 2014; Madandoust & Mousavi, 2012) that kaolinit in form of metakaolin (calcined kaolinit) can be used as an excellent cement substitute. However, if other clay minerals' pozzolanic reactivity was improved, the use of calcined clays would present a good choice in addressing the cement production reduction problem (Rashad, 2015). Recently, natural pyrophyllite was discovered in Algeria. Being calcined and finely ground, becomes a pozzolanic material reacts with calcium hydroxide $Ca(OH)_2$ released during the hydration of cement to form the calcium silicate hydrates CSH and allow to improve the fresh and mechanical properties of concrete. The pyrophyllite ($AI_2O_3.4SiO_2.H_2O$) is a relatively rare mineral because it is not found everywhere in the word; few studies have been done on pyrophyllite used as a cementitious material. Therefore, it has generally been neglected in commercial circles, but its high content of silica and alumina and its physical and chemical properties make it useful in many industries, primarily in refractories, ceramics and various uses.

Pyrophyllite is a soft, white or pale colored silicate mineral. Hydrated aluminum silicate $Al_2Si_4O_{10}(OH)_2$, comprised as the main constituent of some schistose rocks. Pyrophyllite deposits are usually associated with metamorphic rocks, in which the mineral forms packets of pyrophyllite slate or compact agalmatolite aggregates. It sometimes occurs in hydrothermal quartz and ore veins. Pyrophyllite is both fire- and acid-resistant. It is used commercially as a highly aluminiferous raw material for refractory products and as a chemically stable and insulating material. It has Mohs hardness of 1-2 and specific -gravity of 2.6-2.9. Pyrophyllite (from the Greek words for fire and leaf) gets its name from the fact that it exfoliates when water is driven off upon heating, leaving a flaky mass. In pyrophyllite, two silicate layers are sandwiched with a gibbsite layer between them. Thus the overall structure of natural pyrophyllite can be imagined as stacked silicate-gibbsite-silicate sandwiches. After calcination pyrophyllite powder lost its sticky property when combined with water and its color changed from light grey to light orange.

In the literature, the raw pyrophyllite is used as an additive or substitute materials at different amounts into a conventional porcelain mixture (Mukhopadhyay et al, 2009). Extensive and relevant studies on the use of pyrophyllite in building materials such as mortar or concrete are even scarcer (Anggraini et al, 2014). Demez & Karakoç, (2020) assessed the residual mechanical properties of high strength concretes made with pyrophyllite aggregate after high temperature. Terzic´ et al, (2020) studied the utilization possibility of activated pyrophyllite in building materials as a pozzolanic mineral additive. Researches of Lauw and Besari (2001) reported that the calcined pyrophyllite decreased strength of lightweight concrete. They showed that it can be effectively used as a partial replacement of cement but to improve its pozzolanic activity, calcination temperature and finesse must be increased.

The available studies on the application of pyrophyllite are predominantly limited to its utilization in ceramic materials (whitewater, tiles, porcelain) as a replacement of quartz, clay or feldspar (Amritphale et al., 2006; Mukhopadhyay et al., 2010).

Therefore, the general idea behind our investigation was to use Algerian calcined pyrophyllite powder as cement substitution at a rate of 10% and 20% to assess its potential in self-compacting concrete providing additional benefits. This, for economic and environmental reasons by reducing the cost of manufactured cement and CO_2 gas released. Its effect on the workability and mechanical properties of self-compacting concrete was studied.

2. Materials and Experiment

2.1 Materials

Cement CEM II / A 42.5 was used. Natural ground sand with a maximum size of 3 mm was used as natural fine aggregate with a specific gravity of 2.69 g/cm^3, fineness modulus of 2.31 and its water absorption value is 4.5%. Coarse aggregates 3/8 and 8/15 with specific gravity of 2.66 (g/cm^3) were also used. Superplasticizer MEDAFLOW RE 25, a high range water reducer polycarboxylate was used to achieve the required workability of the SCC mixes. Superplasticizer having a pH of 7, a density of 1.06 and chlorine content < 1 g/l and a 25% solids content. Used pyrophyllitte comes from the region of Bechar located 1150 km south-west of the capital Algiers in the form of rock pieces. It was crushed and pulverized using a mill, sieved through a sieve of 80 microns and then calcined at 750 ° C for 3h.

Physical properties and chemical compositions of the calcined pyrophyllite CP and cement are given in Table 1 and Table 2. Furthermore, XRD analysis of pyrophyllite and calcined pyrophyllite are shown in Figure 1 and Figure 2.

Table 1. Physical properties of Calcined pyrophyllite CP and Cement

Caracteristics	CPC	Cement
Specific surface area (cm^2/g)	6200	4400
Specific gravity (g/cm^3)	2.55	2.99
Mineral activity (mg) $Ca(OH)_2$/g	118.3	-

Table 2. Chemical composition of CP and Cement (%, by weight)

Oxydes	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	P_2O_4	TiO_2	PF
CPC	53.77	27.33	8.03	1.23	0.70	1.24	2.86	0.64	0.15	1.10	2.94
Cement	20.71	5.45	3.63	60.4	2.15	2.37	0.65	0.23	-	-	4.28

Chemical analysis of the calcined pyrophyllite CP shows that it contains a significant rate of silica and alumina. Hence it is categorized as alumino-silicate material. The oxide composition of calcined pyrophyllite used in this investigation was about 53.77 % silica, 27.35% alumina and 2.94% was loss on ignition. In addition, calcined pyrophyllite contains impurities as MgO, SO_3, K_2O, Na_2O, P_2O_4 and TiO_2. Moreover, figure 1 show that the crystalline phases of the pyrophyllite are quartz, kaolinit and gypsum, ilite, pyrophyllite and hematite. But after calcination at 750 ° C for 3 hours, kaolinit and gypsum disappeared and new minerals were formed as the anhydride.

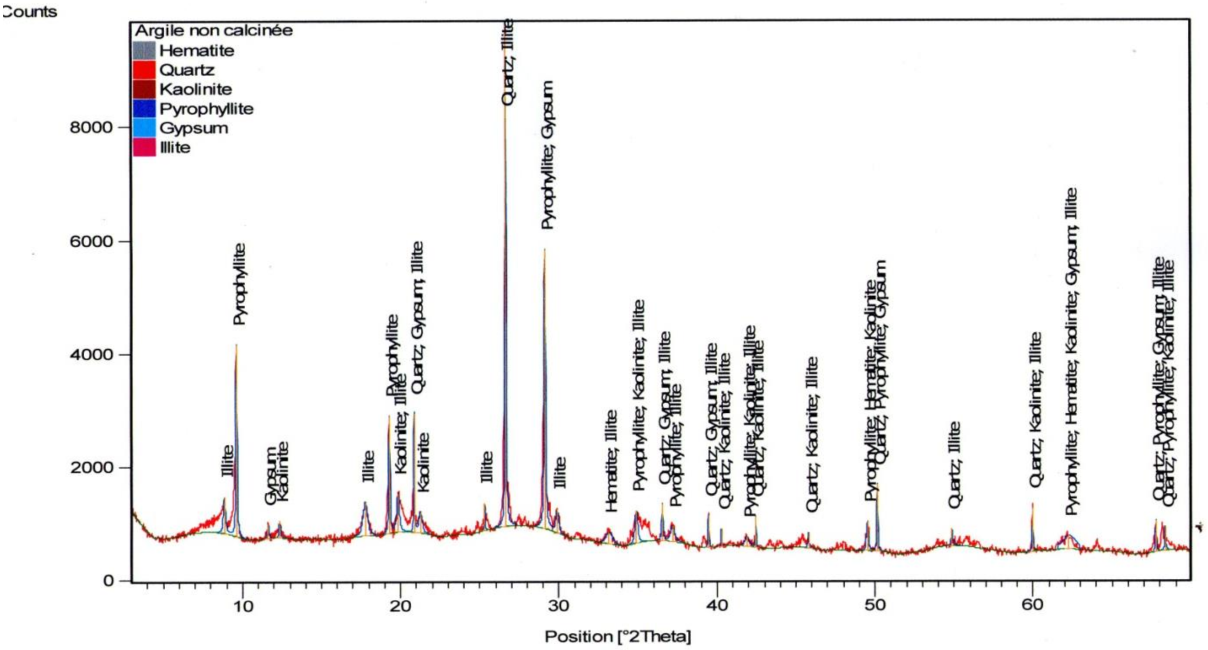


Fig 1. X-ray diffraction of the pyrophyllite (Cu kα filter Ni).

According to figure 2, XRD analysis confirms that applied thermal cycle was not enough to decompose the illite and quartz. Temperature of 750 ° C was chosen based on the works of Samet et al., (2007), Rabehi et al., (2014) and Rashad, (2015). At this temperature, there is a departure of the water content (the dehydroxylation) and the formation of material with an amorphous structure which makes it more reactive than the starting clay. The heat treatment causes the transition of the crystalline phase ordered to a disordered phase by a collapse of the crystal lattice**.**

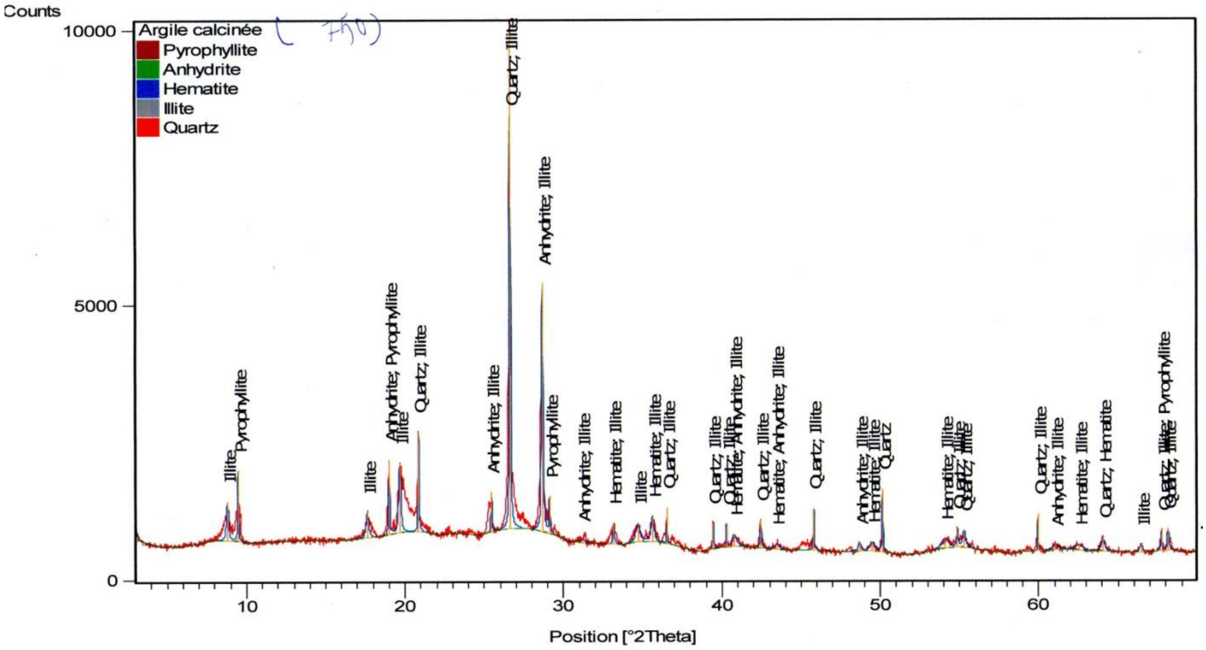


Fig 2. X-ray diffraction of the calcined pyrophyllite (Cu kα filter Ni)

2.2. Formulation of self compacting concrete

Based on the Japanese method (Okamura & Ozawa, 1994), the self-compacting concrete was formulated. The basic data are optimized such as: Gravel to sand ratio G/S was fixed to 1, the ratio of water to cement blended W/B = 0.38 with the cement content C = 450 kg/m^3, a rate of 10% and 20% of calcined pyrophyllite used as cement substitution was chosen and finally, a dosage of superplasticizer was kept constant at 1.7% by weight. Three compositions of SCC mixes were prepared, a control self-compacting concrete (SCC1) without CP and two concretes SCC2 and SCC3 containing 10% and 20% of CP. The mix proportion of self compacting concretes is presented in Table 3.

Table 3. Mix proportion of self-compacting concretes (kg.m^{-3})

SCC/Constituent	SCC1 0% CP	SCC2 10% CP	SCC3 20% CP
Cement	450	405	360
Limestone Fillers	45	45	45
Calcined pyrophyllite CP	-	45	90
Sand	875	875	875
Coarse aggregate G3/8	346	346	346
Coarse aggregate G8/15	519	519	519
Water	182	182	182
Superplasticizer	8.42	8.42	8.42

2.3. Mixing, Casting, Curing and Testing Specimens

For each SCC mixture, three prismatic samples (70x70x280mm) and three cylindrical samples (110x220mm) were cast into steel molds according to standard NF EN 12390-2 (2012). The molds are kept in a room at ambient temperature, and after 24 hours demolded. To ensure proper curing of SCC concrete, the specimens were placed in a tank of water at a temperature of 20° C±2°C, this prevents water loss and to ensure normal operation of hydration process of cement. The concretes were then tested in compression according to NF EN 12390-3 (2012) standard, flexural according to NF EN 12390-5 (2012) and tensile according to NF EN 12390-6 (2012) at maturities 7d, 14d and 28 days.

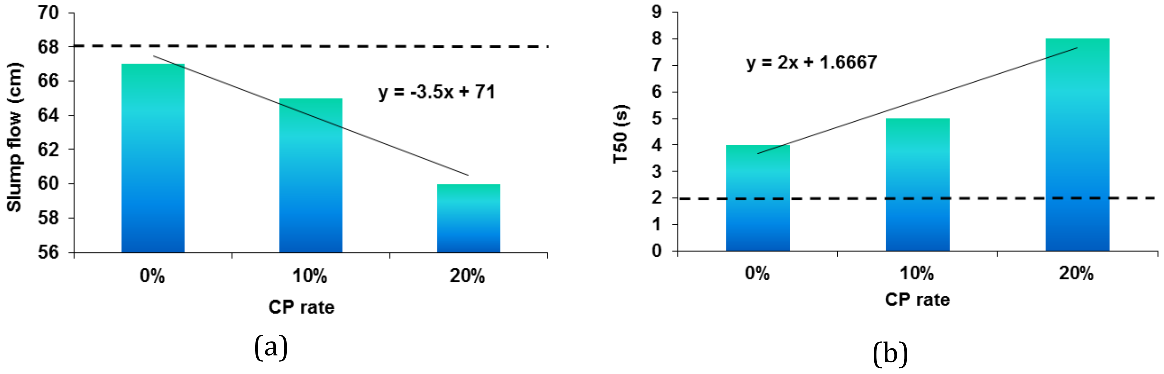
Before casting, slump flow and T_{50} test of mobility of the fresh SCC (NF EN 12350-8, 2010), L-Box test (NF EN 12350-10, 2010) and sieve stability test (NF EN 12350-11, 2010) were carried on each SCC mixture. Slump flow test is utilized to weigh flowability of SCC in lack of impediments. T_{50} test is used to weigh horizontal free flow in lack of impediments. A greater flowability is an indication of lower time. L-Box test measures filling and passing ability of SCC. Sieve stability test is performed to assess the stability of SCC.

3. Results and Discussion

3.1. Influence of calcined pyrophyllite on fresh properties

The results obtained on self-compacting concrete tested at fresh state (Flow test, L-Box, Sieve stability), show that all elaborated SCC with and without calcined pyrophyllite have good fresh properties (slump flow diameter, slump flow time T_{50}, passing ability and stability) that comply with the requirements prescribed by EFNARC (2005).

Figure 3-(a) illustrates the slump flow diameter of SCC. It is shown that for all SCCs, the slump flow was between 55cm and 68 cm, which is an indication of good deformability. When CP is incorporated in SCC, the slump flow results come under SF1 classes according to the EFNARC (2005) guidelines. The presence of calcined pyrophyllite decreased the slump flow diameter of self-compacting concrete. The reduction is 3% for SCC2 and 11% for SCC3. These results are confirmed by the values of the time T_{50}. Figure 3-(b) shows that all SCC have slump flow times which are in the field of SCC (greater than 2 sec). T_{50} is classified into two classes in EFNARC (2005) guidelines i.e., VS1 and VS2, for VS1 class outcome is ≤2 and for VS2 class outcome is >2. In this study, outcomes come under VS2 class.



(a) (b)

Fig 3. Influence of the rate of calcined pyrophyllite on (a)- Slump flow, (b)- Flow time T_{50} of SCCs.

Results showed that the time of flow to reach the 50 cm diameter increased with increase of CP rate. The presence of CP decreased the flowability and made the SCC more viscous compared to SCC1. Indeed, it was found during tests that when the CP rate increased, the concrete became less fluid and its placement in the test specimens was slow. The reduction in slump flow is probably due to the high fineness of CP compared to that of the cement, which can absorb the water and consequently decrease the flowability. Similar results have been reported in other researches when the calcined kaolin was used (Melo & Carneiro, 2010; Hassan et al., 2010).

Moreover, the passing ability determined by L-Box is greater than 0.8 for all SCC. According to EFNARC guidelines (2005), L-Box test results ≥ 0.80 comes under PA1 classes with 2 rebars and the test results ≥0.80 comes under PA2 classes with 3 rebars. This is means that the risk of blockage is avoided. The passing ability decreased in the presence of CP when its rate increased from 10% to 20% (Figure 4). Compared to SCC1, the reduction of passing ability is 6% and 9% for SCC2 and SCC3 respectively.

Figure 5 illustrates effect of calcined pyrophyllite on the stability to segregation of SCCs. According to results, a decrease of segregation resistance of SCC was obtained with the increase of CP rate which translated into an increase of the percentage of milt. But, all the studied SCCs are stable (Milt < 15%) and are classified as SR2. The SCC1 without CP is characterized by high stability (Milt < 5.14%) and consequently a high resistance to segregation and to bleeding. SCC2 and SCC3 are homogeneous and less stable than SCC1. This is due to increase of the amount of CP fines in SCC witch facilitate the passage through the sieve and increases the milt weight.

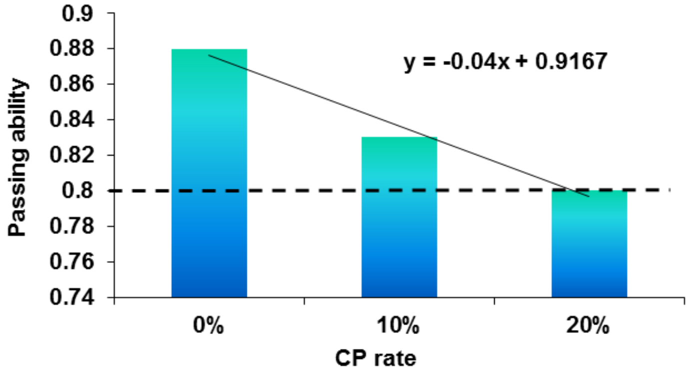


Fig 4. Influence of the rate of calcined pyrophyllite on the passing ability H_2/H_1 of SCCs.

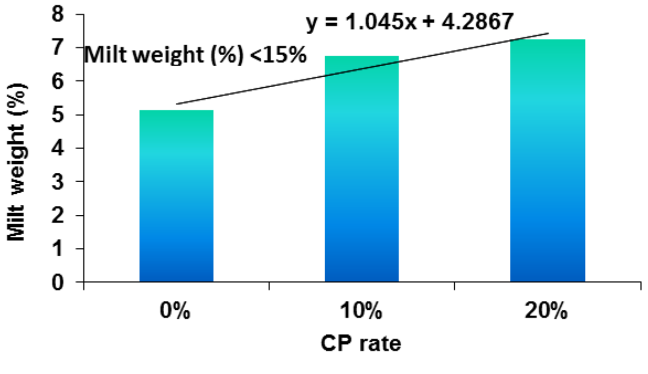


Fig 5. Influence of the rate of calcined pyrophyllite on the segregation rate/ Milt weight of SCCs.

In general, reduction in workability was attributed to few reasons such as greater fineness of addition than cement (Kim et al., 2012; Mo et al., 2018), and the rough morphology of particles could interlock and agglomerate together (Kim et al., 2012).

The three (03) SCC concretes all have a slump flow greater than 60 cm, a quantity of milt less than 15% and a passing ability greater than 80%, which confirms their self-compacting behavior. It seems that 10% is the optimum rate for good fresh properties of SCC. This concrete is classified as having a normal workability, plastic consistency and stability without risk of segregation. So, behavior of SCC2 at fresh state is similar to that of SCC1.

Moreover, a correlation between Milt of sieve stability test and T_{50} was suggested. It is clear from the obtained results that the increase in T_{50} is associated to the increase of the milt and the decrease in resistance to segregation. The curve that describes this relationship is polynomial and the two properties are 100% related. The relation is expressed by the equation (1):

$$\text{Milt weight} = -0.3658\,(T_{50})^2 + 4.9125 - 8.6567 \quad (1)$$

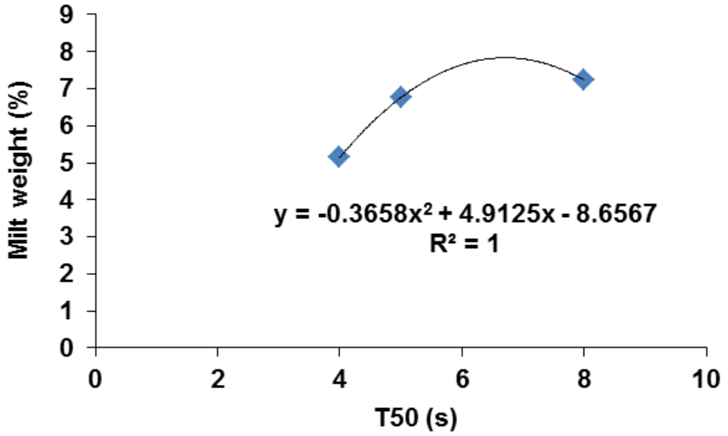
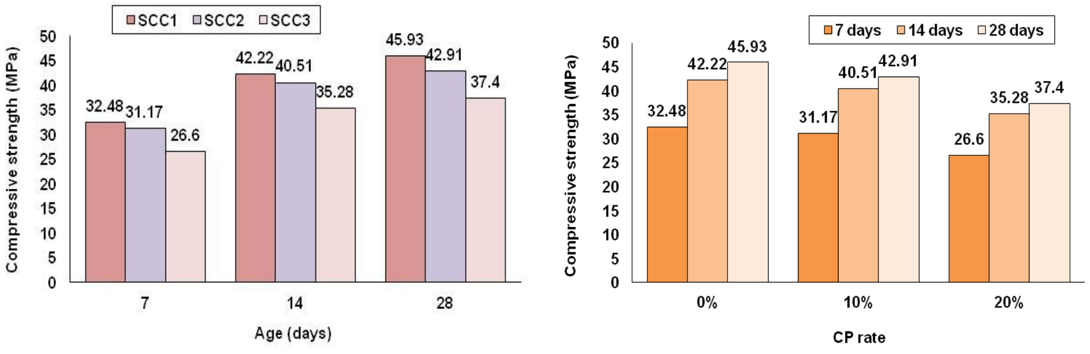


Fig 6. Relation-ship between flow T_{50} and Milt of sieve stability test.

3.2. Influence of calcined pyrophyllite CP on hardened properties

3.2.1. Compressive strength

Figure 6 shows that the increase of compressive strength as a function of the age is substantially similar for all samples tested SCC (Figure 6-(a)). Due to its high volume in Portland cement (450kg/m^3), the control mix has for all ages the highest values of strength. Moreover, incorporation of calcined pyrophyllite as cement substitution decreases the compressive strength (Figure 6-(b)).



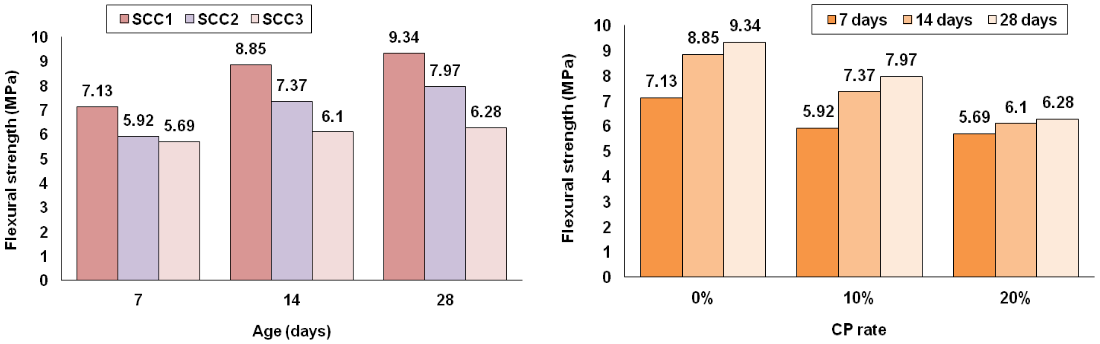
(a) (b)

Fig 6. (a)-Compressive strength, (b)-Effect of CP rate on compressive strength.

The reduction is 4% and 18% at 7 days, 4% and 16% at 14 days, 7% and 19% at 28 days for SCC2 and SCC3 respectively. The rate of 10% CP generates the lowest reduction at all ages. The strengths of SCC2 are almost comparable to those of SCC1. This decrease is due to the reduction of C_3S and C_2S minerals. These are the two main minerals that ensure the development of short and medium-term strength because they lead to the formation of CSH increasing the strength (Chinje and Billong, 2004; Baronio and Binda, 1997). Also, the water destined to cement wetting was mobilized by the CP due to its high fineness, which delayed the hydration process, therefore a decrease in strength has been recorded. Another reason is the low reaction rate of calcined pyrophyllite due to fact that cement type (CEM II) had a negative influence unlike rapid reaction rate of metakaolin and its ability to accelerate cement hydration. (Badogiannis et al., 2015; Akcay et al., 2016; Lenka and Panda, 2017; Barkat et al., 2019).

3.2.2. Flexural strength

Figure 7-(a) shows the increase of flexural strength as function of the age for the three compositions of SCC. The strengths of SCC2 and SCC3 at maturities of 7, 14 and 28 days do not reach those of SCC1. Substituted cement by calcined pyrophyllite CP reduced flexural strength at all maturities (Figure 7-(b)). Reduction is 17% and 20% at 7 days, 17% and 31% at 14 days, 15% and 33% at 28 days for SCC2 and SCC3 respectively. The lowest reduction is obtained for concrete containing 10% of CP.

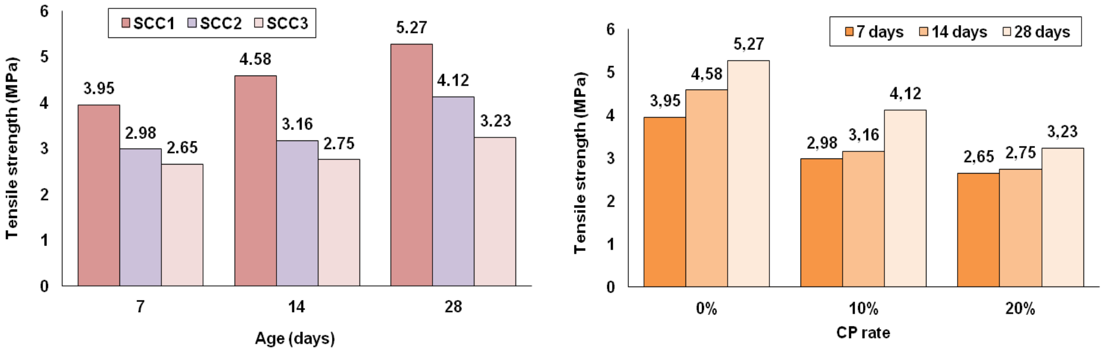


(a) (b)

Fig 7. (a)-Flexural strengths, (b)-Effect of CP on flexural strength

3.2.3. Tensile strength

Figure 8-(a) clearly shows an increase in tensile strengths according to age for all SCC. Moreover, incorporation of calcined pyrophyllite decreased the tensile strength of SCC2 SCC3 at maturities 7, 14 and 28 days (Figure 8-(b)). Significant reduction was obtained. It is 25% and 33% at 7 days, 31% and 40% at 14 days, 22% and 39% at 28 days for SCC2 and SCC3 respectively. SCC2 mixture is the concrete witch developed tensile strengths approaching those of the control.



(a) (b)

Fig 8. (a)-Tensile strength, (b) Effect of CP on tensile strength.

4. Conclusions

This investigation was conducted to assess the fresh and hardened properties of reduced environmental impact SCC made with calcined pyrophyllite. From the obtained results following conclusions can be drawn:

- The slump flow of SCCs is between 55cm and 68 cm, which is an indication of good deformability. It decreases in presence of calcined pyrophyllite. The lowest reduction of 3% was obtained for SCC containing 10% CP.
- In the other hand, the flow time T_{50} increased with the increase of the rate of calcined pyrophyllite from 10% to 20% and a slowdown in the flow of SCC was observed.
- The passing ability is greater than 0.8 for all SCC. This is means that the risk of blockage is avoided According to EFNARC guidelines (2005). The passing ability decreased in the presence of calcined pyrophyllite when its rate increased from 10% to 20%. The 6% lowest reduction is obtained for SCC incorporating 10%.
- The segregation resistance decreased increase of CP rate. But, all the studied SCCs are stable (Milt < 15%) and are classified as SR2. SCC2 and SCC3 are homogeneous and less stable than SCC1
- It seems that 10% is the optimum rate for good workability properties of SCC2 compared to 20%. This concrete is classified as having a normal workability, plastic consistency and satisfactory stability is to say no risk of segregation.
- Behaviour of SCC2 contained 10% of calcined pyrophyllite at fresh state is similar to that of control SCC1 concrete.
- An excellent relationship between Milt of sieve stability test and flow time T_{50} was suggested**.** The two properties are correlated at 100%.
- The use of calcined pyrophyllite as 10% and 20% cement substitution in concrete SCC slows down the hardening process of concrete, consequently producing lower strengths of SCC concretes.
- At 28 days, for self-compacting concrete containing 10% CP, reduction is 7%, for compressive strength, 15% for flexural strength and 22% for tensile strength.
- The rate of 10% CP seems to generate mechanical strengths approaching those of the SCC control.

Workability and mechanical tests show results that support the use of cement with calcined pyrophyllite as part of an economical and environmental self-compacting concrete.

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