

Development and characterization of nanostructured-perlite-cementitious surface compounds

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Abstract The effect of perlite loading on the thermal resistivity, solar reflectance and indirect tensile strength of Nanostructured Cementitious Binder is studied. The main objective of this research is to constitute structural light-weight surface compounds and to improve their thermal resistivity and reflectivity with suitable mechanical performances as a surface compound. Portland White Cement (PWC) was partially substituted by nano clay. An optimum blend of type I PWC, and nano clay was determined based on indirect tensile strength. The perlite dosage was added to the optimum cement-nano clay blend at different ratios (10, 20, 30, 40, 50, 60 and 70%) by weight. Superplasticizer with different ratios was used to study the performance of the 70% perlite mixture. The mixes were prepared using water of consistence. The wet compounds were molded in PVC molds and left for 24 h, then demolded and cured in humid air (20 ± 1 °C & 100% RH) for 28 days. The molds are 50 mm in diameter and 27 mm height. The thermal resistivity and indirect tensile strength of the different compounds were evaluated for the cured samples at 7 and 28 days. It was found that the 2% nano clay improved the indirect tensile strength of the neat PWC. As expected the addition of the perlite has reduced the indirect tensile strength but increased the thermal resistivity of the compound by 250%. The increase of perlite loading has not affected the solar reflectivity of the compound. Sokalan HP 80 polycarboxylate ether superplasticizer was successfully used to recover about 50% of the loss in the indirect tensile strength of the newly

formulated Nanostructure Perlite Cementitious Surface Compounds (NPCSC) containing 70% perlite loading.

Introduction

The thermal and energy performance of buildings depends on the thermal characteristics of the building envelope, and particularly on the thermal resistance of the insulation material used. The performance of building materials is mainly determined by their thermal conductivity, which describes the ability of heat to flow across the material under a specific temperature gradient. The value of the thermal conductivity of a particular material is subject to variation, due to changes in both moisture content and temperature. In actuality, thermal insulation in buildings is exposed to significant and continuous temperature variations, due to varying outdoor air temperature and solar radiation. Thermal conductivity of concrete increases with increasing cement content [1] and thermal conductivity of aggregates. Silica Fume (SF) causes a decrease in the thermal conductivity and an increase in the specific heat of cement paste [2]. SF also causes an increase in the electrical resistivity [3, 4]. However, the effect of SF and Fly Ash (FA) on the thermal conductivity of Lightweight Aggregate Concrete (LWAC) has not fully been studied. Since the thermal conductivity of crystalline silica is about 15 times that of amorphous silica it is natural for the concretes with amorphous silica to have lower conductivity [5–7]. The amorphous silica in the cement paste, which is the continuous phase in concrete, may also contribute to lowering the thermal conductivity. Admixtures, such as SF and FA are used in concrete to improve the mechanical properties, decrease the rate of hydration, decrease the

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alkali aggregate reactivity and decrease the permeability of concrete. However, their effects on the thermal conductivity have received little attention [8–13].

Nano silicate has proven to be an effective reinforcement in a variety of polymeric systems [14–20]. Using clay in a polymer matrix leads to many largely reported improvements, such as: increased tensile strength and modulus, increased stiffness, accelerated crystallization (almost certainly because the clays act as nucleating agents), improved flame retardancy, enhanced barrier to gas (oxygen and carbon dioxide), water and hydrocarbons (gasoline, methanol and organic solvents) permeation, lower density, better scratch resistance even for very modest clay loadings (1–5%), and increased heat deflection temperature [21]. Polymer clay nanocomposite can claim parallel enhancements in other material properties, which, in turn, may represent the major promise for any potential future uses of polymer clay nanocomposite, given that tensile properties can also be improved by other means.

Nano-SiO₂ has been used to increase the strength, flexibility, and aging resistance of polymers [20]. The effect of nano-SiO₂ on the mechanical properties of High-volume Fly Ash High-strength Concrete (HFAC) were studied [22]. The hydration heat, the strength and the pore size distribution were also investigated. A comparison was made between fly ash incorporating nano-SiO₂, fly ash, and nano-SiO₂ alone in terms of weight change after immersion in saturated lime solution. Addition of nano-SiO₂ to high-volume high-strength concrete leads to an increase of both short-term strength and long-term strength. The pozzolanic activity of nano-SiO₂ can activate fly ash, and the weight increment of fly ash incorporating nano-SiO₂. The addition of fly ash leads to higher porosity at short curing times, while nano-SiO₂, acting as an accelerating additive, leads to more compact structures, even at short curing times [22].

Superplasticizers have been employed as additives in a variety of cementitious compounds including cement concrete mixtures. There are four clinker phases of cement namely C₃S, C₂S, C₃A and C₄AF. In fresh cement paste without superplasticizer, C₃S and C₂S have a negative zeta-potential while C₃A and C₄AF have a positive zeta-potential. This leads to a faster coagulation of the cement grains [23]. Cement particles behave as a colloid in suspension with each particle carrying charge. Superplasticizer can be added to modify the suspending liquid and control particle charge. A repulsion force is created when the polymer layer is present at the surface of particles and the layers overlap. This happens whenever the polymer molecules become more compacted as the two layers are squeezed together. The surface potential of the cement phase becomes negative and the particles start repelling each other. Polycarboxylate ether adsorbs more selectively to C₃A and C₄AF than C₂S and C₃S. In addition, long

polyether side chains lead to steric repulsion between the cement particles and increases the workability. Longer main and side chains lead to better dispersion and water reduction.

The main objective of this research is to constitute structural lightweight cementitious surface compounds with high thermal resistivity, high solar reflectivity and suitable mechanical properties for building skin and other applications. Sokalan HP 80 polycarboxylate ether superplasticizer is used in an attempt to recover some of the loss in the indirect tensile strength caused by the addition of high loading of perlite insulation particulates.

Materials and experimental

Materials

The materials used in this investigation are Portland cement (type I) provided by Lehigh Cement Co., perlite provided by Airlite Processing Corporation of Florida, and nano clay. The chemical compositions of the PWC and perlite are summarized in Table 1. The nano clay used in this investigation is montmorillonite clay (Hydrated sodium calcium aluminum silicate). It is Cloisite 30B modified montmorillonite, which was supplied by Southern Clay Products. Cloisite 30B is a natural montmorillonite modified with an ammonium salt. It is used as an additive for plastics to improve various plastic properties, such as mechanical reinforcement, heat distortion temperature, coefficient of thermal expansion and thermal and moisture barrier properties.

Sokalan HP 80 polycarboxylate ether; modified in water supplied by BASF Chemical Company was used as a superplasticizer in this investigation. The superplasticizer was added to the mixed water in ratios of 0.5, 1, 1.5 and 2% by weight of nano clay-cement binder.

Compound preparation and identification

The PWC was partially substituted by nano clay as illustrated in Table 2. The dry PWC and nano clay were shear mixed for 5 min. Then the perlite was added to the nano clay cement binder (NCB) at different weight ratios based on a unit weight of NCB as illustrated in Table 2. The superplasticizer was added to the higher loaded blends of perlite as shown in Table 2. The blended compounds were prepared by slow mixing for 3 min using the standard water of consistency. The compound was molded in a PVC mold for mechanical and thermal conductivity tests. The samples were kept in molds for 24 h, and then demolded. The specimens were cured at 100% relative humidity and

Table 1 The chemical composition of starting material

Oxide composition	Portland cement (%)	Perlite (%)
CaO	66.3	0.6
SiO ₂	22.5	72.7
Al ₂ O ₃	5.4	12.9
Fe ₂ O ₃	0.4	3.4
MgO	1.0	0.6
SO ₃	2.8	–
Na ₂ O	0.17	0.2
K ₂ O	–	0.2
TiO ₂	–	0.9
Ignition loss	1.7	3.0

20 ± 1 °C for 28 days. The specimens were dried at a temperature of 105 °C for 24 h in an oven. The mechanical, thermal resistivity and solar reflectivity tests were performed on dried specimens.

Testing

Thermal conductivity

The thermal conductivity of the Nanostructured Perlite-Cementitious Surface Compounds (NPCSC) was determined using Quickline-10 according to ASTM E-1530; guarded heat flow meter method. In this method the sample is held under a reproducible compressive load

Table 2 The dry mixes composition of blended binder and compound (mass%)

Mixes	PWC	Nano clay		
C0	100	0		
C1	98	2		
C2	97	3		
C3	96	4		

Mix ratio per unit weight of nano clay cement binder (NCB)*				
Mix	NCB	Perlite	Superplasticizer	Mixed water
M1	1	0.1	0	0.575
M2	1	0.2	0	0.875
M3	1	0.3	0	1.175
M4	1	0.4	0	1.475
M5	1	0.5	0	1.775
M6	1	0.6	0	2.075
M7	1	0.7	0	2.375
S1	1	0.7	0.005	1.75
S2	1	0.7	0.01	1.6
S3	1	0.7	0.015	1.45
S4	1	0.7	0.02	1.35

* 98% PWC + 2% nano clay

between two polished metal surfaces, each controlled at a different temperature. The lower contact surface is part of a calibrated heat flux transducer. As heat flows from the upper surface through the sample to the lower surface, an axial temperature gradient is established in the stack. By measuring the temperature difference across the sample along with the output from the heat flux transducer, the thermal conductivity of the sample can be determined when the thickness is known. An axial load 140 kPa was applied on the test sample to insure good contact. The thermal resistivity is the reciprocal of the conductivity.

Water absorption and apparent porosity

Water absorption of each sample is measured according to ASTM C140-01 [24]. The procedures are as follows. The specimens were immersed in water at room temperature (22 °C) for 24 h and then weighed while suspended by a thin wire and completely submerged in water, W_i (immersed weight). The specimens were then removed from the water and allowed to drain for 1 min by placing them on a wire mesh. Visible surface water was removed with a damp cloth. The new weight was taken and recorded as W_s (saturated weight). The specimens were then dried in a ventilated oven at 105 °C for not less than 24 h and until two successive weights at intervals of 2 h show an increment of loss not greater than 0.2%. This final weight of dried specimens was recorded as W_d (oven-dry weight). The absorption is calculated as.

$$\text{Absorption\%} = [(W_s - W_d)]/W_d \times 100$$

Apparent porosity was determined from water absorption of the sample. Apparent porosity was determined by dividing volume of absorbed water by the apparent volume of the sample [25].

Indirect tensile strength

The indirect tensile tests are performed on a Sintec-5D MTS machine using 50 × 25 mm cylinder samples (see Fig. 1). Three samples per batch are tested, with the average strength values reported in this paper. The loading rate on the cylinder is 0.05 mm/min. The indirect tensile strength is determined as follows:

$$\sigma_t = 2P_{\max}/\pi Dt$$

where σ_t is the indirect tensile strength, P_{\max} is the maximum applied load, D is the diameter and t is the thickness.

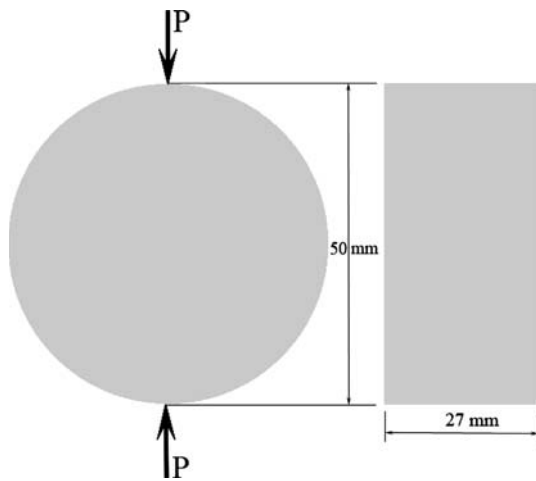


Fig. 1 Specimen geometry and loading configuration

Reflectivity

An RT-060 reflectance/transmittance integrated sphere is used for measuring the surface reflectance. Reflectance is the percentage of incident flux reflected by a material. The integrating sphere has the unique ability to collect flux reflected by a surface over the entire angular range of reflectance distribution. A reference material of known reflectance is used for calibration. The RT-060 is designed for use in the 250–2,500 nm wavelength range.

Results and discussion

Performance of nano clay binder

The relationship between the indirect tensile strength and the nano clay loading for white cement pastes hydrated for 7 and 28 days are shown in Fig. 2. As it can be seen, the indirect tensile strength increases as the replacement of

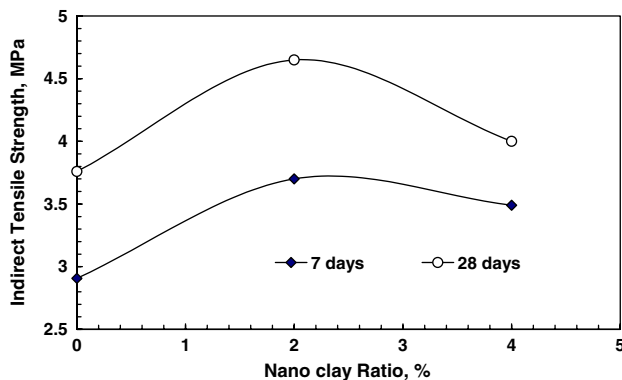


Fig. 2 Variation of indirect tensile strength with the ratio of nano clay to White Portland Cement (WPC) hydrated at 7 and 28 days

PWC by nano clay increases up to 2% by weight; for both 7 and 28 days of curing. However, at 4% nano clay replacement of PWC, the tensile strength decreased for both curing periods. These results show that the indirect tensile strength of hydrated cement paste is enhanced with nano clay replacement. At 2% nano clay the increase of the indirect tensile strength was 28 and 25% for the 7 and 28 days hydration periods, respectively.

Basically, the nano clay can enhance the tensile strength of hardened cement paste by two mechanisms. The first mechanism is the packing effect of small nano clay particles that act as filler in interstitial spaces inside the skeleton of hardened microstructure of cement paste increasing its density as well as its strength. The second mechanism is the pozzolanic effect that combines silicon elements in nano clay with the lime elements of calcium oxide and hydroxide in cement to enhance the bonding strength and solid volume, resulting in higher tensile strength of hardened cement paste.

The pozzolanic reaction between the calcium hydroxide and amorphous silica is normally slow during a prolonged period of moist curing but it reacts rapidly in alkaline environments such as the pore solution of fresh Portland cement paste [1]. There is a decrease of tensile strength of blended cement pastes at higher loadings of nano clay due to the fact that the existence of silicon could transform the original C-S-H (calcium silicate hydrate) gel (lower Ca/Si ratio) in to C₅S₆H₅ (tobermorite) and increase the extent of silicate polymerization [26]. Air voids may also form leading to a weak structure as indicated in Table 3.

The thermal resistivity, reflectivity, water absorption and porosity of nano clay binder was also measured as illustrated in Table 3. There is a slight decrease in the thermal resistivity of the Nano Clay Binder (NCB) as the replacement of nano clay increases. The reflectivity is almost constant while the water absorption has not changed considerably. The porosity of NCB has increased by about 8% for both 2 and 4% replacement of nano clay. The above results reveal that the optimum replacement of PWC is about 2% nano clay and hence this ratio will be used through out the study as the control binder.

Table 3 Physical properties of nano clay binder

Properties	Nano clay binder		
	0%	2%	4%
Thermal Resistivity (m·K/W)	1.92	1.88	1.82
Reflectivity (%)	97	98	98
Water absorption (%)	14.8	15.58	15.96
Porosity (%)	24.8	27.17	27.2

Effect of perlite loading on the mechanical properties and fracture behavior of NPCSC

The variations of indirect tensile strength with perlite loading in the Nanostructured Perlite-Cementitious Surface Compounds (NPCSC) hydrated for 7 and 28 days are shown in Fig. 3. As expected the indirect tensile strength of both hydration times decreases as the perlite loading increases. The indirect tensile strength of the nano clay binder (NCB) at 7 and 28 days were found to be 3.7 and 4.65 MPa, respectively. This indicates that the replacement of white cement by 2% nano clay during the first 7 days attained about 80% of the 28-day indirect tensile strength. The NPCSC with 70% perlite loading attained about 28% of the indirect tensile strength after 28 days of hydration. The development of indirect tensile strength of the NCB was much higher than the NPCSC. The phenomenon can be explained as the improvement of the bond between cement grains due to the pozzolanic reaction of nano clay with calcium hydroxide to produce additional calcium silicate hydrate deposited in the pore system. The addition of perlite to the NCB as a lightweight aggregate leads to weakness of the nanostructured perlite-cementitious surface compounds bond due to its filler effect. Nevertheless, the workability of the compound was excellent and it can be applied to walls or roofs without any problem.

The stress–strain behavior of the NPCSC, M3 and M7 hydrated for 28 days, is shown in Fig. 4. These data represent the average of 5 identical specimens from each mix. It is observed that the M3 compound displays brittle behavior. This is manifested in a sudden drop of the stress after a maximum was reached. The fracture surface is very smooth as shown in the micrograph in Fig. 4 (M3-30%). However for the NPCSC M7-70%, the stress strain

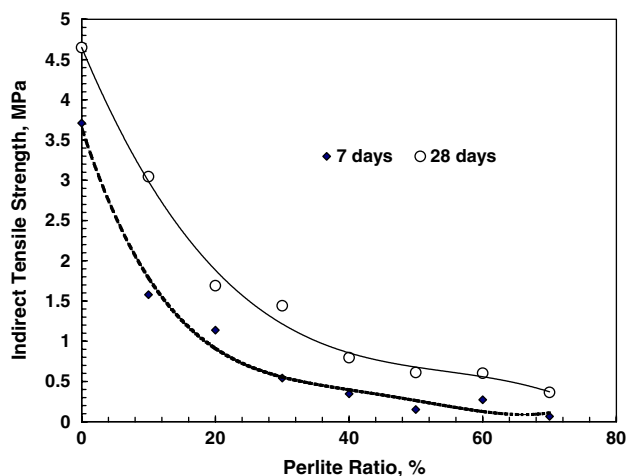


Fig. 3 Variation of indirect tensile strength with perlite ratio of nanostructured perlite-cementitious surface compounds hydrated at 7 and 28 days

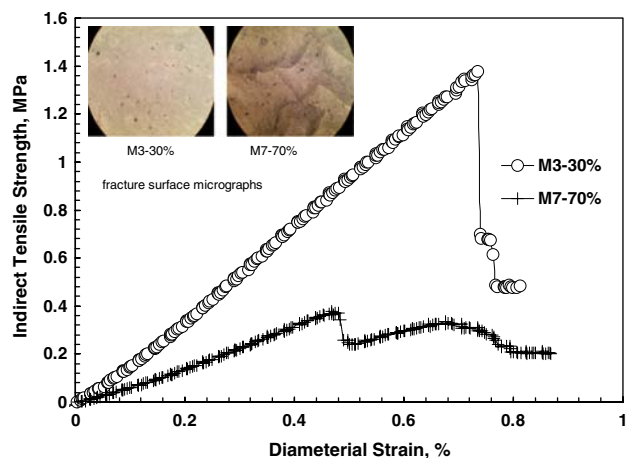


Fig. 4 Stress–strain behavior of the nanostructured perlite-cementitious surface compounds M3 and M7 hydrated for 28 days

behavior is different from the NPCSC M3-30%. There is a small drop in the stress followed by an increase in the stress and the strain, then a smooth decay in the stress followed by a constant stress plateau. This is a so called ‘grace failure’, which is manifested in a rough fracture surface with many facets (micrograph M7-70%, Fig. 4). This behavior is indicative of a higher resistance to crack propagation or fracture toughness. The stress–strain responses appear to be dependent on the amount of the perlite in the mix. As the loading of the perlite increases the roughness of the fracture surface increases. The fracture surface is smooth in specimens containing a low amount of perlite or no perlite. Observations of the surface of the groove in specimen (M7) indicate that the offsetting segment of the crack path was tortuous, and in many cases branching and discontinuities characterized it. Therefore, higher loading of perlite toughens the mix.

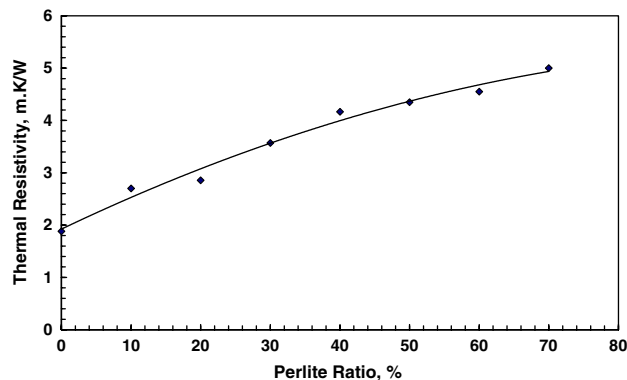


Fig. 5 Variation of thermal resistivity with perlite ratio of nanostructured perlite-cementitious surface compounds hydrated for 28 days

Effect of perlite loading on thermal resistivity and solar reflectivity of NPCSC

The thermal resistivity of the NPCSC as a function of perlite loading is presented in Fig. 5. As can be seen, the thermal resistivity increases with the increasing perlite ratios. The variation of the thermal resistivity shows that the compound containing 70% perlite by weight has a thermal resistivity of 2.5 higher than the compound with no perlite. The improvement in thermal resistivity is due the high insulation properties of the perlite particles coupled with the pores network formed. When a small quantity of the nano particles were uniformly dispersed in the cement pastes; the hydrate products of cement will deposit on the nano particles due to their great surface area during hydration and grow to form conglomeration containing nano particles as nucleus. The nano particles located in the cement paste, as nucleus will further promote and accelerate cement hydration due to their high activity. Also the nano clay can react with calcium hydroxide crystals liberated in cement pastes during the hydration process. Therefore, this pozzolanic reaction process leads to the generation of calcium silicate hydrate through reaction with calcium hydroxide. Thus, the NPCSC can be used as a thermal insulation siding or coating for roofs. The developed NPCSC can reduce heat gain in warm climates and reduce heat loss in cool climates. This will lead to energy savings and indoor comfort.

The relationship between the solar reflectivity of the NPCSC, hydrated for 28 days, and the perlite loading is shown in Fig. 6. It is clear that, the reflectivity of the NPCSC has very minimal change with the perlite loading. The presence of some traces of mineral in the perlite can slightly reduce the reflectivity. This is not significant in the current study. High reflectivity of building skins is always good in summer and has little negative effect in winter, because of the reduced sunshine periods. There is no

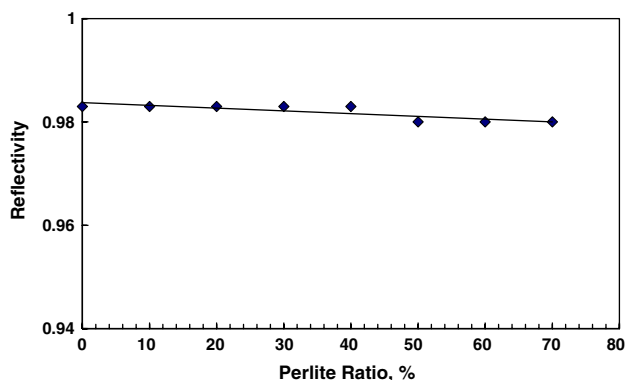


Fig. 6 Variation of reflectivity with perlite loading in nanostructured perlite-cementitious surface compounds hydrated for 28 days

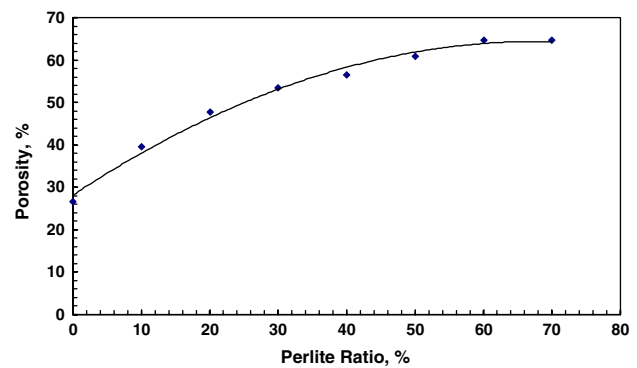


Fig. 7 Variation of porosity with perlite loading in nanostructured perlite-cementitious surface compounds hydrated for 28 days

substantial thermal mass effect, especially if the roofs are thermally insulated under the outer envelope.

Effect of perlite loading on porosity of NPCSC

Figure 7 shows the variation of porosity of nanostructured perlite-cementitious surface compounds hydrated for 28 days with perlite loading. The porosity of the surface compound increases as the perlite loading increases. Basically, the increase of porosity in the NPCSC leads to an increase in the thermal resistivity without appreciable decreases in the solar reflectivity.

Effect of superplasticizer dosage on the mechanical and thermal behavior of NPCSC

The physical and mechanical properties of the developed NPCSC containing different dosages of superplasticizer hydrated for 28 days are given in Table 4. The addition of 1.5% by weight of superplasticizer increases the indirect tensile strength from 0.36 to 2.19 MPa. This is about a 6-fold increase in the indirect tensile strength of the NPCSC with 70% perlite loading. The adsorption of polycarboxylate ether superplasticizer on the surface of cement particles causes negative charges on the cement particles. The alkaline hydrolysis of the ester groups can lead to carboxylate salts and the ethers. As a result, the main chain, which contains the carboxylate groups, is adsorbed on to the surface of the cement particles, while the lateral chains, comprising ethers, break away from the main chain [27]. This leads to an electrostatic repulsion of cement particles with each other. The polycarboxylate ether contains carboxylate groups (COO^-), which react with Ca^{++} ions liberated during cement hydration will form $[\text{R}-\text{COO}^-]-\text{Ca}-[\text{OOC}-\text{R}]$ on the surface of the C-S-H gel or $\text{Ca}(\text{OH})_2$ crystals. The interweaved net

Table 4 Physical properties of nanostructured perlite cementitious surface compounds containing different dosages of superplasticizer hydrated for 28 days

Properties	Superplasticizer doses (wt.%)				
	0	0.5	1	1.5	2
Indirect Tensile Strength (MPa)	0.36	1.18	1.87	2.19	1.82
Thermal Resistivity (m-K/W)	5	3.7	3.4	3.3	3.3
Reflectivity (%)	98	98	98	98	98
Water Absorption (%)	97.6	42.7	34.9	32.1	33.9
Porosity (%)	64.7	40.6	34.9	33.7	34.3

structure which consists of a large ion bonded molecular system is bridged by means of $\text{Ca}(\text{OH})_2$ [28]. This results in dense structure which bridges the pore network system and enhances the heat transfer conductivity and hence reduces the thermal resistivity of the NPCSC.

It is seen from Table 4 that the maximum indirect tensile strength is achieved at 1.5% by weight of superplasticizer. At that dosage, the thermal resistivity decreases from 5 to 3.3 m-K/W. This is translated to a 34% loss in the thermal resistivity. This is due to the low porosity formation, which leads to a dense structure around perlite particles. The reflectivity of the NPCSC containing different doses of superplasticizer is not changed as seen from Table 4. The water absorption was reduced from 97.6 to 32.1% as the superplasticizer load increased and the porosity decreased from 64.7 to 33.7%. Good dispersion of the cement particles induced by the addition of superplasticizer can be attributed to the collapse of the slump behavior of the cementitious mixture.

Basically, the gain in tensile strength of the NPCSC corresponds to loss in its thermal resistivity. This is due to the decrease of porosity, which leads to presence of thermal bridge around perlite particles with low thermal resistance and it become able to transfer heat through it by conduction. The addition of superplasticizer considerably improves the indirect tensile strength of the NPCSC with some decreases in the thermal resistivity. Thus, a compromise has to be drawn between some of these competing properties. This again depends on the intended application. A little sacrifice in the resistivity may be needed when a stronger NPCSC is required and vice versa. Nevertheless, the newly developed NPCSCs are suitable for application in the building industry especially as skins in hot climates due to their superior thermal resistivity and solar reflectivity.

Conclusions

Nanostructured cementitious surface compound containing 70% by weight perlite was developed. An optimization of the physical, thermal and mechanical properties relevant to the building industry were made. The main conclusion

derived from the current study can be summarized as follows:

- The replacement of Portland White Cement by 2% nano clay improves the indirect tensile strength of the hardened pastes by 25% compared to the cement without nano clay at 28 days of hydration.
- The loading of perlite in the nanostructured perlite-cementitious surface compounds (NPCSC) up to 70% by weight of nano clay cement binder increases the thermal resistivity by 250% compared to the control sample (0% perlite).
- A reflectivity of about 0.98 has been achieved even with higher loading of perlite for the developed NPCSC.
- The use of perlite in NPCSC resists the formation of microcracks and increases the toughness.
- Sokalan HP 80 polycarboxylate ether superplasticizer was successfully used to recover about 50% of the loss in the indirect tensile strength of the newly developed nanostructure perlite-cementitious surface compounds (NPCSC) containing 70% perlite loading.

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