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Multifunctional analysis of innovative PCM-filled concretes

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Abstract

This work presents the first results of the thermo-physical and mechanical performance analysis of new lightweight structural concretes with high thermal capacity including capsulated PCMs in a variety of mix designs and capsulation geometries. The thermal tests showed the promising phase change behavior of the composites, while the structural tests showed that their stress-strain behavior appears to be compatible with their use in structural applications, even with the highest PCM concentration (5% in weight). Overall, the multifunctional characterization showed the interesting potentialities of such new composites, given the dead load reduction obtained with their lightweight and thermal capacity increase.

Keywords: Phase change materials; energy efficiency in buildings; smart materials; multifunctional concrete; compressive strength; building envelope; thermal-energy storage; smart material.

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1. Introduction

Research and technology transfer are currently much focused on the development and real world application of new multifunctional and sustainable materials for energy saving in buildings [1-2]. More in details, materials have been acknowledged to be really responsible for building indoor thermal quality by means of their passive role for determining building thermal-energy efficiency and outdoor microclimate mitigation [3-4]. As a result of this significant research effort, the building sector can claim to produce nearly zero energy buildings with a high renewable energy percentage and a huge CO_2 and other greenhouse gas emission reduction in the atmosphere.

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At the same time, two major aspects still need to be carefully investigated: (i) the inner time lag between energy availability/generation and energy need in buildings, and (ii) the development of structural materials with a more sustainable LCA (life cycle assessment) and more efficient thermal behaviour [5-6]. Therefore, thermal energy storage (TES) systems are really under attention from the scientific and industrial community, being able to store heat or cold to be further utilized in a variety of environmental boundary conditions. Generally, three types of thermal energy storage systems can be distinguished: sensible, latent and thermo-chemical energy storage. In particular, latent heat storage systems use the phase change transition of a material identified as PCM (Phase Change Materials), e.g. solid-liquid transformation, in order to store the amount of latent heat in the form of phase change enthalpy, at a constant temperature [7]. Given the aforementioned interesting property of phase change materials, different researchers focused on developing a list of potential PCM candidates to be used for general purposes [8], and in building applications [9-11]. A suitable phase change material, in fact, at least needs to guarantee two crucial requirements: an adequate phase change temperature, and a large melting enthalpy. Nevertheless, the selection of these materials is also made by considering other physical, technical and economic aspects, such as possessing a good thermal conductivity, a small volume change and a low cost of production [12]. Despite the evident straining related to the material selection process, the incorporation of PCMs within building elements can be considered as a promising method to achieve a greater thermal inertia of the building envelope. Several in-lab tests indeed proved the increased heat storage capacity and the decreased thermal conductivity of different construction materials developed by using PCMs [13]. Furthermore, huge effects were also observed by Athienitis A.K. et al. and Kong X. et al. in reducing the interior peak temperatures of buildings equipped with such components [13-14]. By considering the wide variety of applications of PCMs into buildings, their inclusion into cement-based materials and, more specifically, in concrete represents a promising challenge, given the concrete responsibility over a huge amount of carbon dioxide emission in the building sector, needing a true “green” revolution toward energy efficiency, multifunctional capability and environmental sustainability [15]. In fact, concretes and cement-based composites, given their capability to easily incorporate small aggregates in their matrix, including nanomeric particles and fiber additives [16], can be considered as one of the most promising applications for PCMs. Most research studies concerning this topic investigated the capability of PCMs to improve the thermal performance of concrete, without drastically changing its mechanical properties [17-20], and sometimes even found interesting effects in terms of service life increase in different concrete components [21-22]. In order to ensure such acceptable properties, most of these studies focus on the integration of microencapsulated PCMs in cementitious admixtures, thus avoiding leakage problems which could negatively affect the compression resistance of the samples. Thiele A.M. et al., for example, specifically focused on the evaluation of the thermal and mechanical properties of microencapsulated PCM-concrete composite walls [23], while Hunger M. et al. investigated the effect of different percentages of microencapsulated PCMs (1%, 3% and 5%) on the material properties of self-compacting concrete [24] and Konuklu Y. et al. developed a review paper on the use of microencapsulated PCM in building applications [25]. In contrast with the huge piece of literature dedicated to the microencapsulated PCM-concrete topic, the use of macroencapsulated phase change materials embedded in concrete seems to be a less explored field from a research point of view. In an interesting study, for example, Memom S.A. et al. developed structural-functional integrated normal weight aggregate concrete (NWAC) using macroencapsulated paraffin-LWA, which conferred to the composite an acceptable compressive strength and interesting thermostatic properties [26]. A few works tried to optimize the use of lightweight aggregates as carrier unit for PCMs, in order to obtain a composite with high thermal efficiency and low deterioration [27-30].. Despite the research efforts carried out so far in the literature, the goal of achieving composite concretes filled with PCMs that are compatible with structural applications is yet to be attained, as several aspects need to be more carefully investigated. Among those, the following are especially worth mentioning: stress-strain constitutive behaviour, ductility, reliability, porosity, viscosity, shrinkage and more. In the overall scenario depicted above, the present research builds upon the previous contributions developing and testing new cement-based materials with PCM inclusions, by proposing a new structural concrete for multifunctional application in thermal-energy efficiency passive strategy in buildings. Additionally, a new type of PCM capsule is tested, consisting of a bead with including microcapsules filled with paraffin based PCM, in order to test its efficacy from both mechanical and thermal points of view. To this aim, the multifunctional characterization of these concretes is carried out and key results are presented.

2. Materials and methods

2.1. Development of the composites

The present paper concerns the analysis of three different concrete typologies:

- Concretes with no PCM additives, used as reference material for comparison purpose – *noPCM-C*;
- Concretes with microencapsulated PCMs (5% in weight of concentration) – *microPCM-C*;
- Concretes with macrocapsulated PCMs (5% in weight of concentration) – *macroPCM-C*.

The nominal transition phase temperature of the selected PCMs corresponded to 18°C for all the prepared samples. The chosen PCM material was an organic paraffin included into microcapsules in the microPCM-C. Such microcapsule ensured a PCM content in capsule of 85–90 wt.% and their size corresponded to 17-20 microns, keeping the temperature stability up to 250°C, given the inert and stable polymeric shell composition of the capsule [32]. The macroPCM-C composites were developed by including the same concentration of macrocapsuled PCM included into a patented new matrix with 3-5 mm of diameter including the same microcapsules of the microPCM-C sample. The PCM content in bead is 80% for the macroPCM-C. Figure 1 clarifies the phase changing materials geometry and the composites configuration.

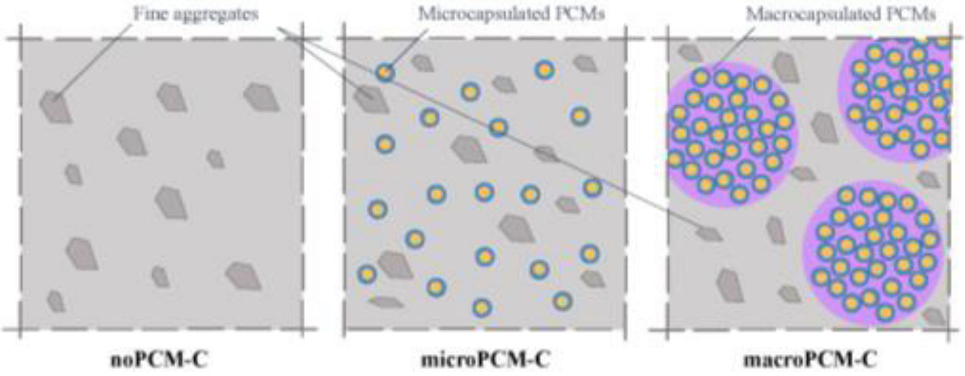

Fig. 1. Schematic representation of the tested concretes (not to scale).

This work concerns the analysis of the mix designs of concrete including micro and macrocapsulated PCMs with 5% in weight by means of addictive procedure during the mix design. Therefore, no substitution of any aggregates has been carried out while preparing the PCM-based materials into the structural concretes. For thermal and structural purposes, several sample configurations have been developed. In particular, concrete 19×19×5 cm^3 slabs were prepared for thermal tests in the climate chamber, and 10×10×10 cm^3 cubes (according to [33]) were prepared for structural tests and outdoor thermographic analyses. Table 1 reports the mix design of the tested samples. A polycarboxylate plasticizer was added in order to obtain similar good workability of the mixes. The cement was pozzolanic, type 42.5. Two types of aggregates were utilized: sand having nominal dimensions between 0 and 4 mm, and medium gravel with nominal dimensions between 4 and 8 mm. Figure 2 reports some photo evidence of the specimens where no visual difference has been observed in comparing normal and composite specimens, even if the superficial properties of the specimens will be tested by means of solar spectrophotometer for more detailed analysis.

Fig. 2. Tested specimens of concretes with no PCMs and with micro and macrocapsulated PCMs.

Table 1. Mix design of the tested concretes.

Sample name	noPCM-C	microPCM-C	macroPCM-C
Components	kg/m^3	kg/m^3	kg/m^3
Cement	524	446	446
PCMs	-	102	102
Sand	951	816	816
Gravel	638	548	548
Water	234	223	223
Plasticizer additive	2.62	6.77	4.46

2.2. Thermo-physical analysis of the composites

The thermal characterization of the samples was carried out by means in-lab analysis tools such as (Figure 3):

1. Solar spectrophotometer and integrating sphere, type Shimatzu SolidSpec-3700, according with the ASTM standard ASTM E1980 – 11 [34];
2. Thermal emissometer, using a AE1 RD1, according with the Standard ASTM C1371-04a [35];
3. Transient Plane Source (TPS) method, using a Hot Disk 2500 system, in accord with the ISO 22007-2 standard [36].

The first test allowed to characterize the optical finishing of the concrete surface, in order to assess the variation imputable to the PCM inclusion, while the second test allowed to assess thermal conductivity and thermal diffusivity, and to derive the specific heat of the tested samples. In this apparatus, the probe, i.e. a kapton-covered flat tungsten spiral, behaves both as a heat source and as a resistance thermometer [37]. The probe was placed in a single sided configuration, for the large irregularity of one side surface of the specimens, installing a superinsulating material on the other side of the probe, with known thermal properties. The experimental campaign was carried out in laboratory at ambient temperature, i.e. at about 20-22°C, thus with most of the PCMs in the composite already in the liquid phase. Each measurement, in both the devices, was performed three times for each specimen. Then, the mean values were calculated and the final data were reported in the following section.

2.3. Field thermal analysis of the composites

The superficial temperature profile of the prepared specimens was registered during a sunny day when the specimens were exposed to outdoor conditions, subject to direct solar irradiance, before and after the transition phase temperature, by assuming the fusion trend during the course of the measurement.

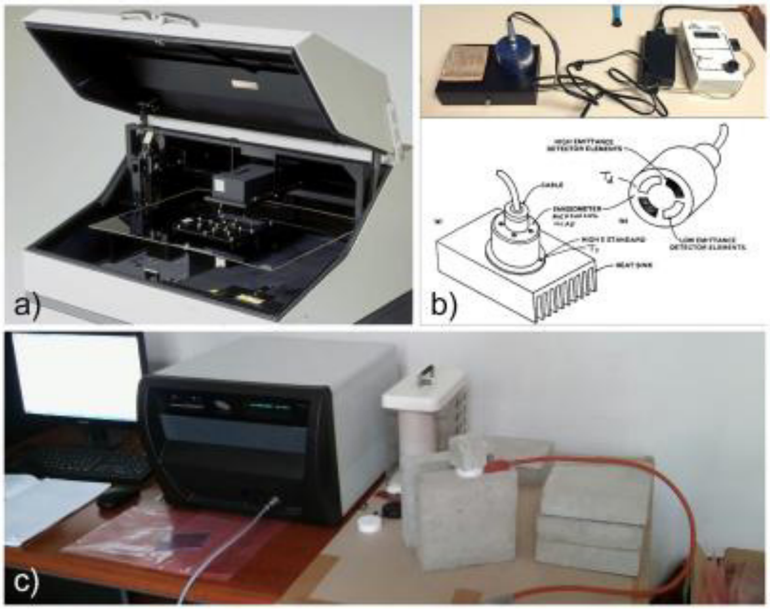

Fig. 3. Lab experimental apparata for thermal and optic-energy tests: (a) solar spectrophotometer, (b) thermal emissometer, (c) hot disk.

The IR camera detected the infrared energy emitted by the samples and visualized its surface temperature in a very fast and accurate way for direct comparative purposes. In this work, the superficial temperatures of the investigated 10×10×10 cm^3 noPCM-C and PCM-filled concrete samples were firstly registered at lower temperature than at the melting level, therefore the same cube samples were exposed for about one hour to the outdoor environmental conditions in a clear sky summer day characterized by an outdoor temperature of about 28-30°C and a relative humidity of about 50% at 12:00 pm. After one hour of exposure, the superficial temperature of the samples was once again measured by means of the thermal camera. The procedure was repeated a second time after three hours.

2.4. Mechanical characterization of the samples

The mechanical characterization of the specimens was carried out by means of a universal testing machine for compression tests. In particular, the Controls Advantest machine with a maximum capacity force of 5000 kN was used and instrumented with three linear displacement transducers placed at 120 degrees. The complete compressive stress-strain curves, including their unstable post-peak branches, were obtained by means of displacement control tests. The test speed was chosen as 2 µm/s, with a maximum displacement of 5000 µm. In this way, the analysis allowed to assess the maximum compressive force of each specimen and its final axial strain. The tests were carried out in accordance to the provisions of UNI EN 12390-3 [39]. Figure 4 reports the picture of the experimental setup and of the specimen positioning, with the displacement transducers.

Fig. 4. Lab experimental setup for mechanical tests: (a) the devices for compression test (b) and the specimen positioning with the displacement transducers.

3. Results

3.1. Results of the transient plane source method for measuring thermal conductivity and thermal diffusivity

The thermal conductivity analysis showed how the samples present non-negligible differences in terms of thermal conductivity and diffusivity. In particular, the thermal conductivity of the new macrocapsulated PCM was much affected by such inclusions, since it showed higher conductivity, able to produce a general decrease of thermal insulation capability of the macroPCM-C composite. At the same time, its capability to propagate the thermal field in the macroPCM-C decreased, as showed in the right graph of Figure 5, confirming also the inner morphological variation of the material imputable to the macrocapsule addition. The microcapsulated PCM concrete showed instead a decreased thermal conductivity, maybe imputable to the small and closed pores created for the inclusion of the PCM microcapsules. Conversely, its capability to propagate the thermal wave in transient conditions increased. Therefore, the promising preservation of the same thermal conductivity properties of the macroPCM-C showed the promising application of such new capsule for thermal-energy storage incorporation into cement based materials, as the main scope of the work.

3.2. Results of the optic-energy characterization

The thermal emittance analysis, as expected, did not show any contribution imputable to the PCM inclusion into the concrete composites under investigation. In fact, the thermal emittance values were measured around 0.88-0.99 as typically observed for cementitious materials [38]. On the other side, the solar reflectance measurements reported in Figure 6 performed in different positions of the sample cubes, highlighted the effect of PCM inclusion in decreasing solar reflectance capability of the concretes, for higher superficial roughness of the composites. In fact, the visible range of the spectrum (380 – 780 nm) did not show important differences on the lateral side of the sample, which was much better polished for workability reasons.

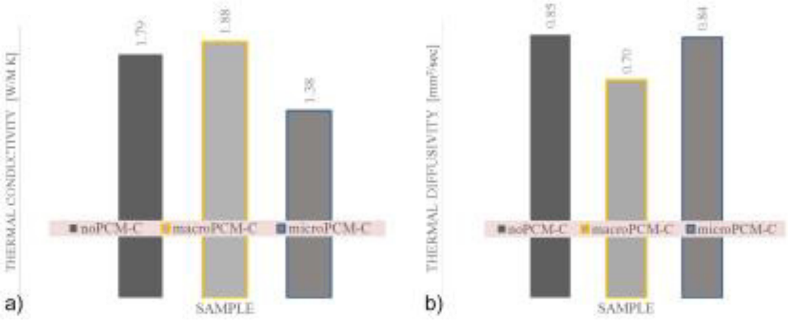

Fig. 5. (a) Thermal conductivity and (b) diffusivity results by means of hot disk method.

Nevertheless, the upper side of the sample showed a bigger mutual difference among the samples, given the relatively low weight of the microcapsules and macrocapsules responsible for their buoyancy within the concrete mass. Such difference was mostly evident in the visible part of the spectrum, but also in the overall composite upper surface reflectance trend (Figure 6.a). In general, it can be seen how the PCM inclusion does not affect the concrete solar reflectance in a significant way to change its thermal behavior due to solar response.

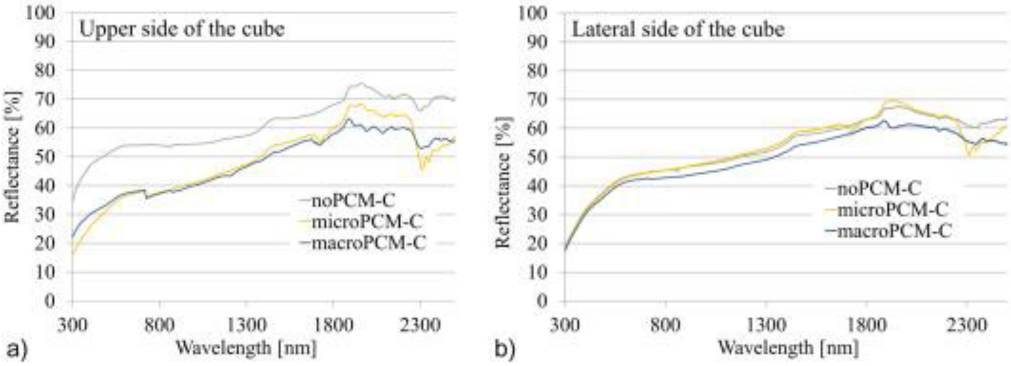

Fig. 6. Solar reflectance measurements of the three samples, (a) taken on the upper side of the cube, and (b) on the lateral side of the cube.

3.3. Results from the infrared thermography

The surface temperature of the samples was analyzed by means of multiple subsequent IR thermography, taken before and after the melting of the PCMs inside the composite. Table 2 reports the temperature difference calculated as the superficial temperature before and after the exposition, during which the melting has been supposed to happen. The exposition has been planned at 12:00 pm – 1:00 pm and at 3:30 pm – 4:30 pm during the same sunny day, that registered a maximum temperature of 35°C with 980 W/m^2 of global solar radiation over a horizontal plane, registered by means of a weather station positioned over the testing field. The macroPCM-C concrete showed the best capability to decrease the superficial temperature, and therefore confirmed the PCM melting during the course of the exposition of the specimens. In fact, the IR thermography was taken over the upper surface of the sample, which was the one with the highest density of macrocapsules, as specified in the solar reflectance analysis.

Table 2. Superficial temperature analysis.

Sample name	T difference [K] at 12:00 – 1:00 pm	T difference [K] at 3:30-4:30 pm
noPCM-C	+3.9	+4.6
microPCM-C	+1.8	+3.5
macroPCM-C	-0.2	+1.4

3.4. Results from the mechanical tests

Table 3 reports the mean and characteristic values of the compressive strength of the samples, R_m and R_{ck}, respectively, evaluated by testing 5 cubic specimens for each type. These results show that the PCM inclusion resulted in a reduction of compressive strength that, however, might still be compatible with structural applications, especially if a weight content of PCM smaller than the 5% is considered. It is also worth noting that the R_m of the macroPCM-

C is higher than the one corresponding to the equivalent concentration of microcapsulated PCM concrete, but, at the same time, the coefficient of variation (CoV) of the compressive strength of the macroPCM-C composites is much higher than the one of the microPCM-C ones. This seems to demonstrate that the concrete filled with microcapsule is more reliable than the one filled with macrocapsules. This is due to the fact that macrocpsules affect the braking methodologies, since a fracture of the capsules itself has been ofserved in the fracture surface, after the mechanical tests. In parallel with the axial compression tests, the density measurements showed that the composites with PCMs presented a much lower density with respect to the neat sample noPCM-C, demonstrating how the PCM-filled concretes could behave as lightweight concrete, with the following structural benefits, and as “thermally-capacitive” concrete, from the thermal-energy perspective. Such decrease has been motivated by considering the relatively lower mechanical properties of the microcapsules containing PCMs.

Table 3. Results of the structural performance tests on the concretes.

Sample name	R_m [MPa]	CoV	R_{ck} [MPa]	Density [kg/m^3]
noPCM-C	44,39	0,09	33,52	2265±38
microPCM-C	25,25	0,04	21,80	2018±38
macroPCM-C	29,48	0,14	15,03	2023±46

4. Discussion

Starting from the previous published works on PCM based latent thermal energy storage into building materials through capsulation process, this work was aimed at developing and testing the different thermal and mechanical properties of new composite concretes with a new kind of PCM macrocapsule and to compare it with a more typical microcapsulated PCM doped concrete. The new macrocapsules, basically consisting of agglomerated microcapsules within the cement based mix design, were tested and the increasing concentration of such macrocapsules produced very promising results from the multiperspective approach it has been implemented in this work, as summarized in the conclusions section of this paper. The experimental analysis carried out in the lab for investigating thermal features of the new composite material and its mechanical performance showed that a new lightweight material with good structural performance was developed, also characterized by a relatively higher thermal inertia, despite the lightweight characteristic imputable to the lightweight macrocapsule matter and the generated porosity. The implemented methodology showed to highlight the overall behavior of the new composite material and the mechanical tests also demonstrated how the PCM inclusion does not affect the concrete ductility, which is an interesting finding and useful property for concretes to be used in seismic areas.

Therefore, the prototyped concretes demonstrated interesting potentialities from both mechanical and thermal-energy perspectives, deserving further investigation in future developments of this work, in particular for what concerns the new macroPCM-C sample. In particular, thermal cycles in environmental simulation chambers will be carried out in order to identify the thermal progress of the material transition phase, with varying PCM concentration and by also taking into account workability issues. Meanwhile, further mechanical tests will be carried out, primarily to investigate the stress-strain constitutive behavior of the composite concretes with varying PCM content and the statistical properties of the standard compressive strength.

5. Conclusions

This research was aimed at developing and testing the multifunctional performance of new PCM-filled structural concretes for building applications. Such concretes were prepared by adding 5% in weight of encapsulated phase change material for thermal-energy storage applications. Moreover, the included PCMs were selected in order to identify the best capsule geometry for structural and thermal purpose. In particular, the same paraffin PCM with melting temperature at 18°C was selected and industrially capsulated in two ways. The microcapsules included about 85-90% of PCM in small capsules with diameter of about 17-20 microns. The second configuration concerned the macrocapsuled PCM, consisting in a sort of matrix with PCM microcapsules inside, having a whole diameter of 3-5 mm and a whole PCM concentration of 80%. The prototyped concretes were therefore tested from a thermal-energy and mechanical point of view, in order to evaluate the effective potentialities of such composite multifunctional application in constructions. The optic-energy analysis by means of solar spectrophotometer showed that, in the upper surfaces in particular, PCM inclusion modified the superficial configuration of the concretes, by lowering their solar reflectance capability mainly due to the roughness increase. Nevertheless, the thermal emittance was not affected by the PCM addition at all. The thermal conductivity of the macrocapsulated PCM-filled concrete showed to be relatively higher than the standard one, and much higher than the microcapsulated PCM-filled concrete, demonstrating that the big capsules produce relatively lower thermal insulation capability, but with a smaller thermal diffusivity compared to the microPCM-C and the noPCM-C (standard concrete). In fact, the thermal conductivity is always around 1.8 W/mK in the macroPCM-C, and it shows the promising finding that the PCM inclusion does not decrease its thermal conductivity by means of the polymer shell. Therefore, the PCM contribution during the phase change could be better expressed in the macroPCM than in the microPCM sample, showing how the innovative macrocapsule deserves further investigation because it can represent the optimized version of the PCM-filled concrete typically filled with microcapsules such as the microPCM-C.

The mechanical characterization also showed that, despite a non-negligible reduction of compression strength has been detected, the concretes still presented promising mechanical resistance to be classified as structural concretes. Interesting results showed that the PCM capsule inclusion produced a non-negligible decrease of density of the materials, i.e. about 10-11%, making it able to be classified as a lightweight concrete, able to reduce the dead loads of the structure. At the same time, the prototyped material behaves as thermal capacity optimized material for building energy efficiency applications, as showed by means of the IR-thermography.

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