ULTRALIGHT MINERAL FOAMS FOR SUSTAINABLE INSULATION APPLICATIONS

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ABSTRACT

The intention of the construction industry to contribute on cutting back on the global CO_2 emissions is an issue that goes beyond any other question. A most important leverage remains on the reduction of energy consumption of buildings in operation. In this, high-performance insulation materials may play a major role. However, so far various insulation products widely used in buildings have come under discussion due to large uncertainties regarding their recyclability and, because of their poor performance when it comes to fire safety.

The Institute of Construction and Building Materials (WiB) at the TU Darmstadt is examining the performance of non-flammable and high-performance mineral-bounded foams for insulation purposes for many years. As partner in a joint research project for energy active factory construction called "Energy Transfer and Application factory - ETA-Factory", a full-scale production hall was successfully built with a 30 to 40 cm thick insulating layer of mineralized foam applied, and an average density of 180 kg/m³. For this, a mix design was developed at WiB, in a semi-continuous production method for ultra-light and very robust foams in fresh condition, jointly with industrial partners. With this mineral foam based insulation layer, the whole building envelope of the ETA-factory was made of cement-based materials only.

As a consequence of this successful application of light mineral foam, research has been driven forward aiming at solving its most experienced weaknesses, viz. the not yet completely satisfying thermal conductivity ($\lambda \approx 0.065$ W/(m $\cdot$ K)) and the relatively high chemical and drying shrinkage and, especially when regarding the associated crack formation. Significant progress has been made in both areas, resulting in mineral foams with densities of less than 100 kg/m³ and with a thermal conductivity comparable to common insulation materials. In addition, in conjunction with a successful project application, a closed-loop recycling concept has be developed to achieve a significant capturing of CO_2, whenever reusing recycled mineral foam in cement production facilities.

Keywords: mineral foams, non-flammable, high-performance mineral bounded foams.

INTRODUCTION

It is possible to produce cement-based foams in a very wide density range (Table 1). Due to their good thermal insulation properties, high fire resistance and long durability, lightweight cementitious foams are recommended to achieve a successful energy efficiency in buildings. A large number of studies have already been carried out on the composition, physical properties and applications of foam concretes with a density ranging between 500 kg/m³ and 600 kg/m³ (Narayanan 2012, Othuman 2011, Panesar 2013, Ramamurthy 2009, Ranjani 2012). There is also some research published on very-lightweight foams with densities around 200 kg/m³ (Akthar & Evans 2010, Gilka-Bötzow 2016, Pott 2006) (see also Figure 1). While so far only little research has been done on the compositions and the properties of ultra-light mineral foam (ULMF) with densities lower than 160 kg/m³ or even approaching 100 kg/m³. This is probably due to their increased instability, as mineralized foams with bulk densities below 160 kg/m³ no longer have a completely closed pore structure (Gilka-Bötzow et al. 2016). There is no research focussing on the mechanical and thermal properties of such low-density foam. Several researches reported that the thermal conductivity of ultra-lightweight foam concretes decrease with reduction of the apparent density. However, for densities below 150 kg/m³, this diminishing relationship is not obvious (Chen 2014, Gilka-Bötzow 2010, Jiang 2016, Liu 2014).

Table 1: Dry density ranges and proposed categorization for porous cementitious foams with aggregates D < 2 mm.

dry density range [kg/m³]	category
1400 – 1800	(reinforced) foam concrete
800 – 1400	foam mortar
400 – 800	mineralized foam
400 – 160	(very) lightweight mineralized foam
< 160	ultra-lightweight mineralized foam

The present study aims at developing and researching an ULMF – which is stable in the fluid and hardened state. To realize such an ULMF with a density of 100 kg/m³, an air void content over 95 % has to be reached by adding large amounts of prefabricated foaming agent (Gilka-Bötzow et al. 2016). Coalescence and fragmentation of bubbles in fresh foam concrete is unavoidable due to thermodynamic instability in the foam. This phenomenon usually leads to collapse of the foam concrete during the hardening process, which in turn leads to an increase in density and a deterioration in physical properties (Tscheuschner 1999, Mathes 2004). Studying the stability of the aqueous foams is useful to understand the mechanism that leads to collapse and to improve the stability of ULMF. Moreover, the cement slurry as the so called continuous phase of a fresh cementitious foam, plays also an essential role. If the slurry has poor rheological properties, the fresh cementitious foam will desintegrate while mixing or during the hardening process (Gilka-Bötzow & Koenders 2015). The rheological behaviour of concretes may typically be changed by adding superplasticizer and/or by altering the water content. A good understanding the effect of these parameters on the stability of fresh and hardened mixtures is critical for preparation of a stable ULMF.
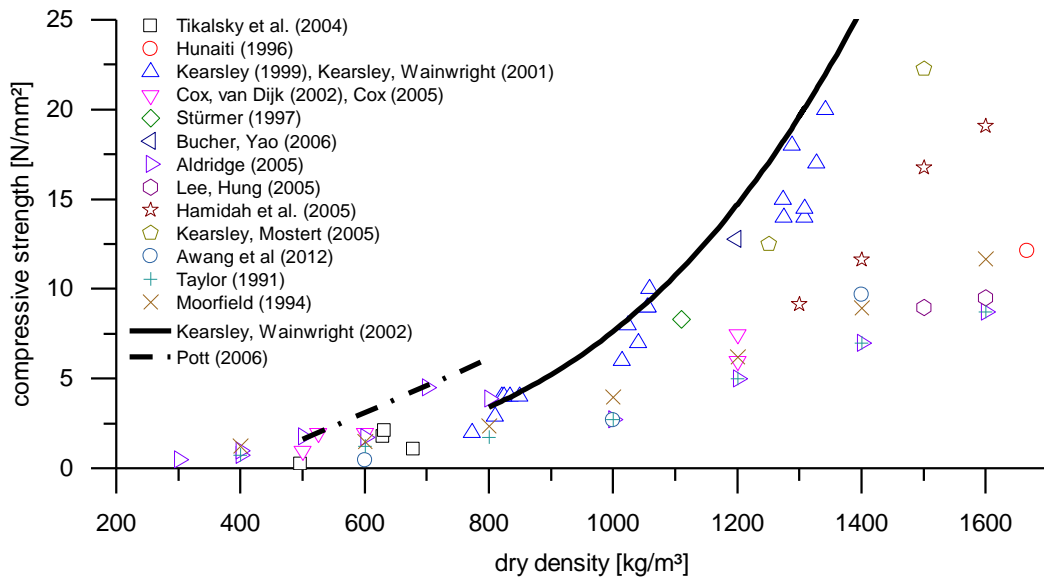
Figure 1: Relationship between 28 days compressive strength and bulk density of "foamed concrete" being the result of a meta-study.

In the present study, for creating a stable ULMF with densities below 100 kg/m³, aqueous foam stability was researched by comparing six foaming agents with the same density (80 kg/m³) over time. The effect of different aqueous foam densities (60 kg/m³ and 80 kg/m³) on the properties of hardened cementitious foams were determined. ULMF was prepared using Portland cement, silica fume, lime-stone flour and several chemical admixtures. The thermal conductivity, density, porosity and compressive strength of the hardened ULMF at age of 28 day were tested.

MATERIALS AND METHODS

Raw Materials
Materials used for this research included OPC (CEM I 52.5 N, Leimen plant, Germany), silica fume, lime-stone flour with a particle size from 0 µm to 20 µm. Besides, hardening accelerator (formiat) and shrinkage reducing agent (calciumsulfoaluminat) were added to the cement slurry. In addition, six foaming agents were used to compare the foam stability over time. Three of them are based on synthetic surface active substances (denoted S_1, S_2, S_3), two are protein-based (denoted P_1, P_2) and one of them is a so called biotenside (denoted B_1).

Mix Design
The mix design was done using a rational mix design method (Gilka-Bötzow et al. 2016). The composition of the ULMF is reported in Table 2. Two densities of aqueous foams (60 kg/m³ and 80 kg/m³) were prepared by using a pre-foaming method. A PCE-superplasticiser was used to enhance the workability of the cement slurry and to reduce

water consumption. The superplasticiser had a low content of defoaming agent. It had also a slight retarding effect, as nearly all foaming agents have.

Table 2: Mix design

ρ aqueous foam	cement	water	aqueous foam	PCE	silica fume	lime-stone flour	hardening accelerator	shrinkage reducer	w/f
kg/m³									
60	58	23.9	57	0.3	5.8	5.8	1.2	2.9	0.33
80			76						

Production Procedure
The foam concretes were prepared according to the proportions listed in table 2, at 20 °C. The process is specified as follows: Firstly, water with superplasticiser was poured into a colloidal mixer, and then the half amount of Portland cement and additives were added gradually. After stirring for 90 s, the other half of cement and additives were poured into the colloidal mixer followed by another 90 s stirring. The aqueous foam was produced using a foam generator to the aimed density. The generator is equipped with a proportional mixer to add a fixed amount of foaming agent (2 %) to the water. Compressed air then flows into the fluid. With this, the kinetic energy is converted into surface energy. The so generated foam was then thoroughly mixed with the base mixture. The demoulding process was conducted after 14 days, then samples were fully covered with polyethylene film and cured at 20 °C until 28 days. After that all specimens were oven dried at 45 °C until constant weight was achieved.

Experimental Tests
To determine the velocity of foam decay, bulk aqueous foams were filled in flat bottom glasses with a height of 120 mm and a diameter of 60 mm. After that the foam height was recorded every 30 min from the beginning up to 120 min. Based on this, the decay rate was calculated as the quotient of the measured height and the initial height. The thermal conductivity was measured using prismatic standard cement test samples (160 mm · 40 mm · 40 mm) by the transient plane heat source method according to the EN ISO 22007-2. The compressive strength test was conducted with the cubic samples (15 mm · 150 mm · 150 mm) according to the EN 12390-3. In order to be able to observe the entire failure process of the mineralized foam, the compressive test was continued up to a compaction of more than 50 %. The loading rate was set to 0.2 MPa/s. The thermal conductivity as the compressive strength of the foam concrete were calculated as the average value of three parallel specimens. The total porosity was calculated from the skeletal density determined by a helium pycnometer and the bulk density calculated by dividing volume of sample by its mass.

RESULTS AND DISCUSSION

Aqueous foam

Figure 2 shows the decay of the tested aqueous foams after 120 min. Although all foams had the same density (80 kg/m³), the protein-based foams (P_1, P_2) show a more closed air void structure that allows to contain a greater amount of small air voids. This also guarantees a more stable foam network. In contrast, the synthetic foaming agents induced coarser air pores (S_1, S_2, S_3). However, the aqueous foam produced with the biotenside foaming agent (B_1) collapsed rapidly from the top downward.
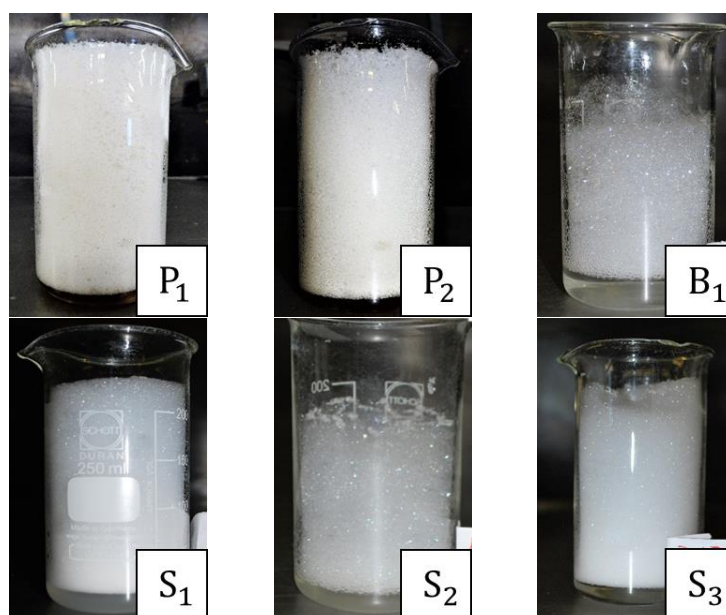

Figure 2: Decay of aqueous foams after 120 min

Figure 3 shows the decay rate over time of the various aqueous foams based on the different foaming agents. The decay rate of the aqueous foams with different densities produced with P_1 is shown in Figure 4. When exposed to gravity, the aqueous foam structure changes from circular foams to hexagonal lattices. The curvature at these boundaries create a low pressure zone which results in compression between adjacent bubbles, while further accelerating drainage and reducing the distance between the adjacent membranes. The underlying aqueous foams gradually shrunk until they disappeared, and the upper aqueous foams gradually enlarged, while the membranes between the aqueous foams became thinner (Bikerman 1973). As can be seen in the Figure 2 and Figure 3 the collapse of the aqueous foam was accompanied by an increase in water volume. From the Figure 4 it is apparent that the aqueous foam with low density (50 kg/m³) collapsed much more obvious and faster than the high density foam (80 kg/m³). This provides a guidance on the choice of an aqueous foam with an appropriate density for the mineralizing process.
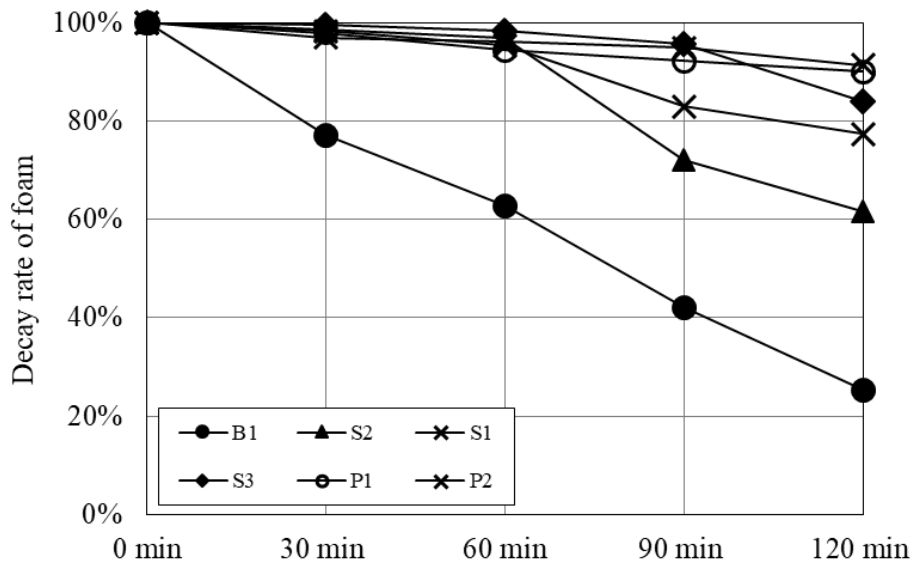
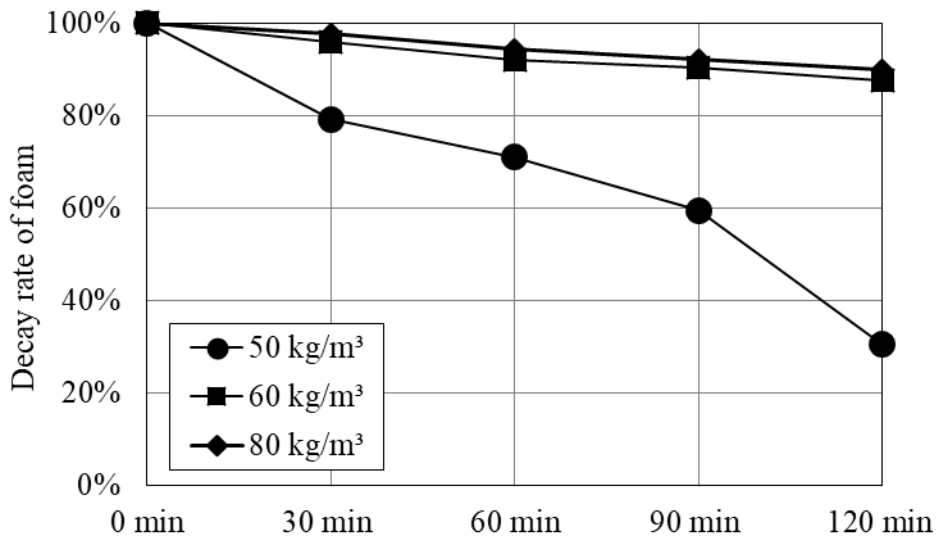Figure 3: Decay rate of aqueous foams based on the different foaming agents.

Figure 4: Decay of aqueous foams (P_1).

Mineralized Foam

Figure 5 shows the representative stress-strain curves of the tested ULMFs with the aqueous foam density 60 kg/m³ and 80 kg/m³, respectively. Figure 6 shows in more detail the same curves near the coordinate origin. All the stress-strain curves of the ULMFs demonstrated similar trends regardless of the aqueous foam density. First the foam bubbles on the surfaces collapsed under the applied load, later the inner structure of the foam broke as a second failure mechanism. The curve for the compression process can be roughly divided into four stages:

Stage I (o-a, strain approx. 0 % – 3 %): First the strain increases rapidly with the stress (elastic platform). This trend can be traced back to the collapse and compaction of the

surface voids, which often have poor mechanical properties. For that there are several complementary reasons. First the pores on the surface are not closed, but kind of hemispheric open pores. Therefore, the surface of the sample has for geometrical reasons a lower bearing capacity. Moreover on the one hand there is too much water on the underside of the specimen due to the drainage of the aqueous foam. On the other hand later there is always too little water near the surface during hydration due to superficial drying, which causes a gradient of humidity inside the open pore structure of the material. The pore structure gets gradually compacted with the increasing stress. Stage II (a-b strain approx. 3 % – 15 %): The strain continues to increase almost linear with the stress but at a lower rate than stage I.

Stage III (b-c strain approx. 15 % – 70 %): The strain increases with a wavering stress. At this stage, the stress reaches its maximum. This stage is characterized by a continuous collapse of the air-void structure within the sample.

Stage IV (c-d strain approx. 70 %): The stress begins to decrease. During this process, the residual strength of ULFM tends to be constant while the strain increases slightly until reaching a plateau.
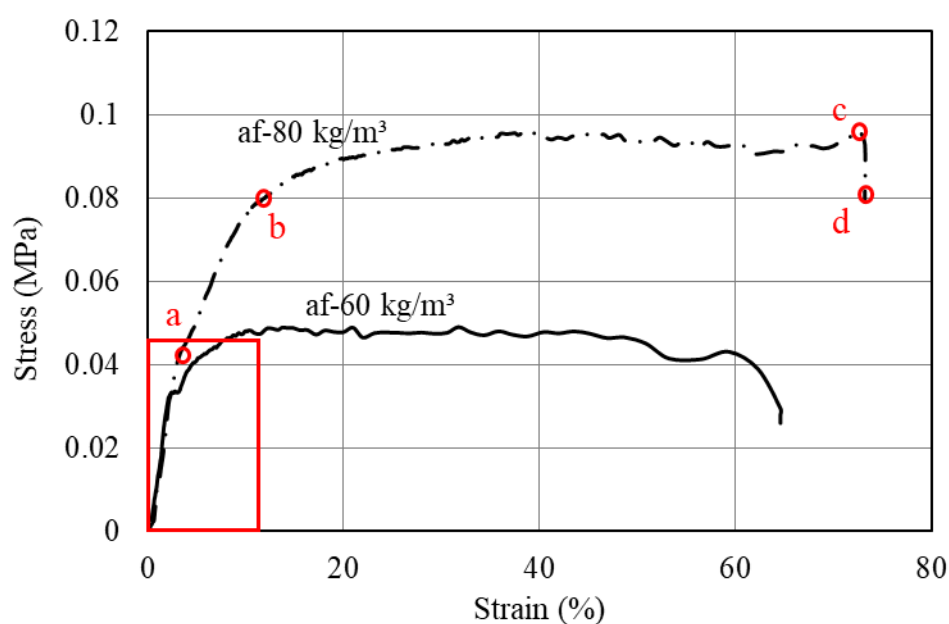

Figure 5: Strain-stress diagrams of the tested ULMFs with different densities.

Figure 6 shows the same curves as before up to 10 % strain, with stage I and II respectively being fitted by a straight line. According to experimental observations and abovementioned analysis, when the stress reaches the intersection σ_1 and σ_2, the broken pores were further compacted forming a layer of dense concrete powder. At this point, it can be considered that the maximum compressive strength has been reached.
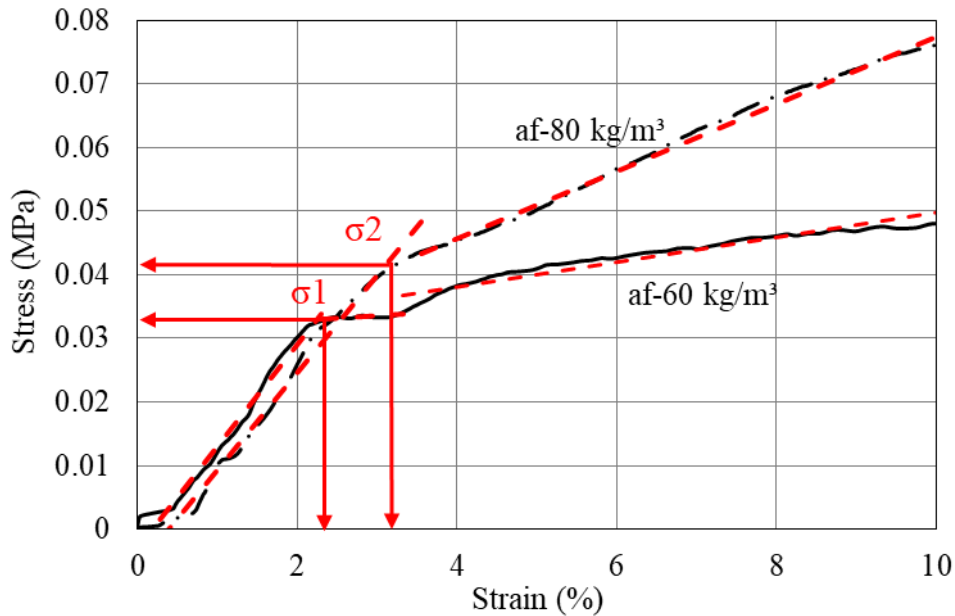
Figure 6: Detail of the strain-stress relation of the tested ULMFs with different densities.

Besides the compressive strength, the different aqueous foam densities have a considerable effect on other physical properties of the ULMFs as shown in **Error! Reference source not found.**. It is apparent that a low aqueous foam density results in a lower compressive strength and also in a slightly lower dry apparent density of the ULMF. In addition, it has a significant effect on thermal conductivity. All these phenomena are most probably due to the respective pore structures that are formed. Depending on the bulk density of the aqueous foam used, the porosity differs in pore size distribution, but also in the type of the formed pore walls. These can have different thicknesses and tightness.

Table 3: Experimentally determined physical properties of the mineralized foams investigated

aqueous foam density	compressive strength	thermal conductivity	dry apparent density	total porosity
[kg/m³]	[MPa]	[W/(m · K)]	[kg/m³]	[%]
60	0.030	0.048	97.2	96.6
80	0.041	0.053	98.3	96.4

However, there is no obvious difference in the total porosity between them. It indicates that the water content of the aqueous foam in the present case has no major influence on the total porosity which includes also the porosity of the solid material framework. With this, the mentioned water amount is directly proportional to the created apparent porosity and so indirectly proportional to the density of the mineralized foam. For these reasons, together with the real water cement ratio, the amount of water in the aqueous foam, relative to the total water content, rises disproportionally with declining densities (Gilka-Bötzow et al. 2016). So, not by considering the water in the aqueous

foam in the mix design, as is reported in many publications (e. g. Narayanan & Ramamurthy, 2000), which is normally only acceptable for higher dry densities.

CONCLUSION

The present study aims at developing an ultra-light mineral foam (ULMF) as a thermal insulation material to reduce the energy consumption of buildings and to enable a sustainable product. A robust ULMF with a dry density of about 100 kg/m³ and an air void content of 98 % was developed, which showed a 28 day compressive strength around 0.04 MPa and a thermal conductivity of about 0.053 W/(m · K). The stability of aqueous foams was researched by comparison of different foaming agents over time. Protein-based foaming agent, in general, can create a highly stable foam with a closed cell structure, which provides a more stable network of air voids than synthetic foaming agents. Moreover, the density of aqueous foam affects its stability, thereby affecting the properties of ULMF. The increase of density of aqueous foams (from 60 kg/m³ to 80 kg/m³) has a larger influence on the increase of the compressive strength (40 %) than on the thermal conductivity (10 %) of ULMFs. In extended tests on the compressive strength of the mineralized foams the total failure mechanism was investigated and classified in four stages. More knowledge about this behaviour can be useful in the context of understanding the energy-dissipating behaviour of these materials.

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