LIBS HYDROGEN ISOTOPES DETECTION: SIGNIFICANCE IN NUCLEAR/FUSION TECHNOLOGY

M. Trtica [1*], J. Savovic [1], M. Kuzmanovic [2], D. Rankovic [2], J. Stasic [1]

[1] VINCA Institute of Nuclear Sciences – National Institute of the Republic of Serbia, University of Belgrade, Belgrade, Serbia; e-mail: etrtica@vinca.rs
[2] Faculty of Physical Chemistry, University of Belgrade, Belgrade, Serbia

The aspect of the detection of hydrogen isotopes in nuclear materials was analyzed in this review. Particular attention was paid to the spectroscopic methods for the detection of hydrogen isotopes in fusion reactor materials since their presence can induce severe problems in terms of material degradation and shortening of its lifetime. The main focus is on laser-induced breakdown spectroscopy, an optical emission method that is efficient, reliable, and fast. Recent results obtained at the VINCA Institute will also be presented.

Keywords: *laser-induced breakdown spectroscopy, hydrogen isotopes detection, fusion and fission technology, nuclear materials*.

ОБНАРУЖЕНИЕ ИЗОТОПОВ ВОДОРОДА С ПОМОЩЬЮ ЛАЗЕРНО-ИСКРОВОЙ ЭМИССИОННОЙ СПЕКТРОСКОПИИ

M. Trtica [1*], J. Savovic [1], M. Kuzmanovic [2], D. Rankovic [2], J. Stasic [1]

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[1] Институт ядерных наук VINCA – Национальный институт Республики Сербия, Белградский университет, Белград, Сербия; e-mail: etrtica@vinca.rs
[2] Белградский университет, Белград, Сербия

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Проанализированы спектроскопические методы обнаружения изотопов водорода в материалах термоядерных реакторов, поскольку их присутствие может оказывать влияние на деградацию материала и срок его службы. Лазерно-искровая эмиссионная спектроскопия рассмотрена как эффективный, надежный и быстрый метод оптической эмиссии.

Ключевые слова: *лазерно-искровая эмиссионная спектроскопия, детектирование изотопов водорода, технологии синтеза и деления, ядерные материалы.*

Introduction. Nuclear energy is an important energy source for modern technologies and has practical applications in our daily lives. Key features include high power density, reliable power supply, and a CO_2-free energy source. In this framework, fusion energy can be considered as clean energy. The use of materials with exceptional properties is extremely important for the long-term operation of nuclear facilities. During the operation period, degradation of these materials may occur due to certain phenomena/processes. Monitoring of these processes is crucial as they can negatively affect the material properties. Fusion technologies use hydrogen isotopes dominantly in the form of fuel. In fission technology, on the other hand, the primary hydrogen sources are hydrogen-based moderators (light (H_2O) or heavy water (D_2O), or metal-hydrides, e.g., ZrH_2). Developing sensitive methods for hydrogen isotope detection in nuclear, fusion and fission technology is of great interest and challenge nowadays [1–3]. This review paper is mainly devoted to the analysis of methods relevant to H-isotopes detection in nuclear materials, emphasizing the laser-induced breakdown spectroscopy (LIBS) method, due to its unique properties. Some of the results of the LIBS application for H-isotopes detection in fusion and fission technology are presented. Also, this review covers our research on LIBS detection of H-isotopes conducted recently at the VINCA Institute.

Focusing on fusion technology, H-isotopes, apart from the fuel form, can be present within the reactor's first wall/plasma facing material (PFM) and structural materials (SM). An important fusion reaction is a reaction between deuterium and tritium (DT reaction). In this context, there are two main approaches at the moment [4]: magnetic confinement fusion (MCF) and inertial confinement fusion (ICF), or the so-called magnetic and laser concept, respectively. In the laser concept, two directions are included, i.e., direct-drive and indirect-drive [4]. The fusion reactor is a complex system and the design for the two approaches is shown in Fig. 1 – the MCF approach at ITER (International Thermonuclear Experimental Reactor) [5] (Fig. 1a), and the ICF approach at HiPER (Laser Fusion European Project) (Fig. 1b) [4, 6]. Both reactor designs include the first wall/plasma facing and structural materials (Fig. 1a,b). Candidate materials for PFM can be tungsten, beryllium, molybdenum, etc. [4, 5], and for SM high-quality steels like austenitic stainless steel 316L and ODS steel (Oxide Dispersion Strengthened steel) [5, 7, 8]. A proper selection of materials for fusion reactor technology applications is essential, and they should satisfy requirements such as the high capability to withstand thermal loading, good resistivity to the action of neutron and electromagnetic fluxes, low affinity to absorption of H-isotopes, and fast induced radioactivity decay [8, 9]. As a result of the DT reaction, the intense neutron and electromagnetic fluxes, alfa-particles, heat flux, and H-isotopes can be present inside the fusion reactor. Regarding the presence of hydrogen isotopes, it should be emphasized that they can be accumulated in the first wall/PFM [3, 4, 10], and can also be present in the SM due to transmutation reactions [4, 8, 10]. The accumulation and presence of H-isotopes in the materials, plasma facing as well as structural metals, can induce their degradation via the appearance of blistering, swelling, and cracking of the surface [7, 11, 12]. Figure 1c shows some of these effects obtained by the action of D-ions from low-flux D-plasma on the tungsten (PFM) surface [7]. The implantation of D-ions was done by the plasma source [7]. Blistering (Fig. 1,c1) and cracking (Fig. 1,c2–c4) of the W surface were recorded in the course of this irradiation. Mainly, the direct consequence of the impact of H-isotopes on the materials/metals is their damage and drastic lifetime reduction. In this context, monitoring the presence of H-isotopes in the fusion reactor materials is highly important.

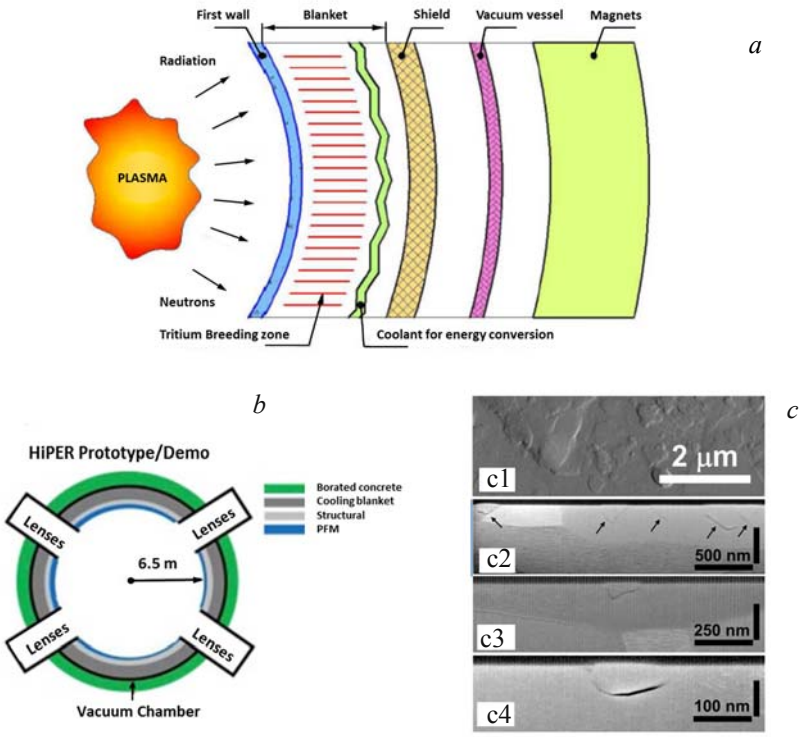

Fig. 1. Partial view (schematic) on (a) ITER [5] and (b) HiPER fusion reactor [4, 6]; (c) tungsten morphology exposed to low-flux D-plasma [7], c1 appearance of the blister and c2–c4 the cracking.

Detection of hydrogen isotopes. Methods used in practice for detecting hydrogen isotopes in metals can be classified as nuclear and non-nuclear [3, 13]. Several nuclear methods should be pointed out – nuclear re-

action analysis with LOD (limit of detection, volume-based) estimated at 100 at. ppm; neutron radiography; neutron reflectometry; and the notched neutron spectrum technique. The accuracy of the last-mentioned technique can be estimated at the level of about 0.4 wt. ppm. Apart from these methods, there are other non-nuclear methods [3, 13], like thermal desorption spectroscopy; elastic recoil detection analysis; glow discharge optical emission spectroscopy; laser-induced desorption spectroscopy, and LIBS. This review focuses on Laser-Induced Breakdown Spectroscopy due to its originality, uniqueness, and potential.

Laser-induced breakdown spectroscopy (LIBS) is a modern optical spectroscopy method [14–19] that can be used for qualitative and quantitative elemental analysis of almost any kind of material. Due to its unique characteristics, LIBS has found numerous applications that range from science via industry, geology, and nuclear technology to medicine. LIBS is an emission spectroscopy technique where plasma initiated by laser irradiation of the target serves as an excitation source. When the laser radiation intensity is sufficiently high (for nanosecond pulses in the span of 10^8 to 10^{11} W/cm^2), the target is ablated, and plasma is generated above the surface. This high-temperature plasma (>10000 K early after its creation) consists of atoms, ions, and free electrons. Initially, high electron density of the plasma immediately after laser ablation gives rise to a strong continuum emission and spectral line broadening caused by the Stark effect. As the plasma evolves in time and cools down, characteristic atomic and ionic emission lines of analyte elements can be monitored. Typically, temporal gating of the emission is used to discriminate the line emission from the continuum background.

A typical experimental LIBS setup is shown in Fig. 2a. The setup is comprised of a pulsed laser, optics for focusing laser radiation and collecting plasma emission, the dispersive device (spectrometer), a detector of plasma emission, and a computer for system control and data processing. Time-resolved measurements require a delay generator to synchronize the plasma production and the emission spectra acquisition. One characteristic of the LIBS method is its highly flexible experimental setup. The experimental design could be optimized to fulfill the requirements of a specific application by selecting the laser (wavelength, pulse duration, energy) and its focusing conditions, the type of gas and pressure of the surrounding atmosphere, a system for radiation detection (spectrograph coupled with detector), and signal acquisition parameters.

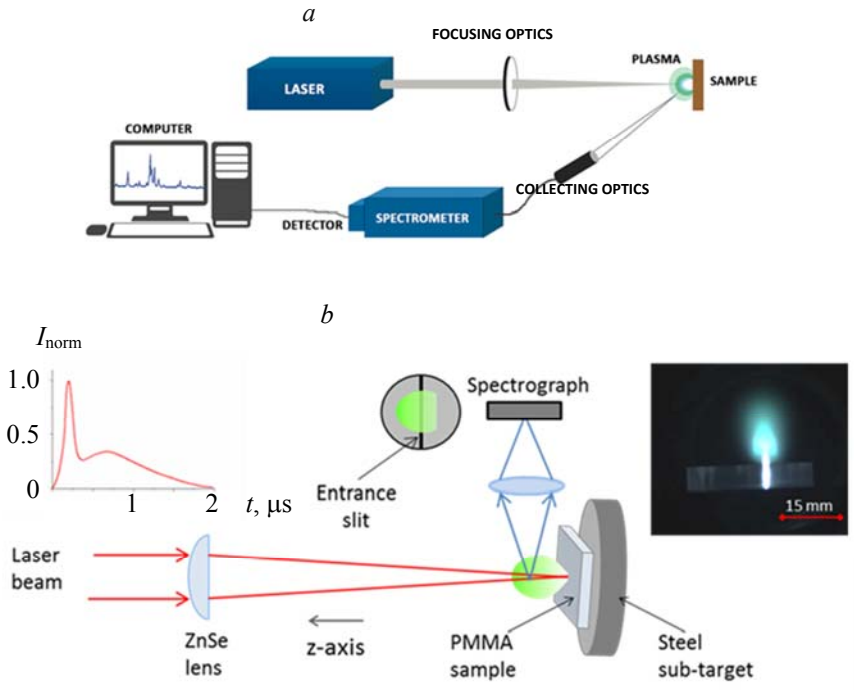

Fig. 2. (a) Typical experimental setup for LIBS [18]; (b) LIBS VINCA Institute experiment on PMMA polymer; inset – CO_2 laser pulse shape and view of the plasma [1].

The LIBS method has numerous advantages and versatile applications. It can be applied to solids, liquids, and gases; it is a non-contact and minimally destructive method; a typical ablated mass is of the order of nano- or even picograms; the technique requires minimal or no sample preparation, and the measurements

are rapid and thus suitable for real-time analysis. Compared to analytical techniques like ion- and electron-microprobe or flame atomic absorption, LODs for LIBS are much lower, around a few ppm or lower. However, LIBS has some disadvantages as well. LIBS depicts higher LODs relative to ICP-AES, where LODs are at the ppb level or lower. The accuracy and precision of the measurements are typically a few percent, while the reproducibility mainly depends on the variations in laser pulse energy.

The necessity of increasing the sensitivity and detection power of the LIBS technique resulted in the permanent search for methods for signal intensity enhancement. In that context, various approaches have been applied [18] – double laser pulse excitation of plasma; a combination of laser with an electric discharge (spark/arc/glow discharge); interaction between microwave radiation and laser-induced plasma; magnetic field confinement of laser-ablated plasma; methods based on the sample surface preparation such as nanoparticles deposition; target pre-heating, and others.

The possibility of LIBS detection/monitoring of hydrogen isotopes in fusion technology, i.e., analysis of reactor materials (PFM and SM), was considered in detail in [20–26]. Such investigations are especially relevant in the course of the last five years. In [20] the double-pulse LIBS was used to observe deuterium-exposed tungsten (plasma-facing material). In [21] remote *in situ* LIBS was employed to detect deuterium presence in molybdenum, also a candidate for PFM. In addition, the application of LIBS for monitoring protium ($_1H^1$) was presented in [22, 23]. Ashikawa, et al. [22] considers the depth distribution of protium in graphite target (candidate for PFM), while in [23], analysis of protium in molybdenum target (candidate for PFM) was performed. In [24] LIBS was used to monitor tritium and impurities in the first wall/PFM, W-Be, W, and W-Al. In these investigations, deuterium was a proxy for tritium, while Al was used as a proxy for Be due to the high toxicity of Be.

Fission. Although the review is focused on fusion technology, it should be mentioned that the significance of H-isotopes detection/monitoring in fission technology is also vital. Hydrogen isotopes can be present in the form of moderators based on water (light (H_2O) and heavy (D_2O) water) and in metal-hydride configuration, e.g., ZrH_2, YH_2 [3, 27]. H-isotopes can be present in the fuel form as well, such as in a SNAP (Systems Nuclear Auxiliary Power) reactor [3, 27], where the fuel is a mixture of ^{235}U (93% enriched) and ZrH_x. Further, hydrogen can be incorporated into the structures of some fission reactor components [3, 28, 29]. Namely, it is well known that rod-shaped fission fuel is situated in tubes made of zirconium-based alloy (zircaloy), e.g., zircaloy-4 with excellent properties. Water can react with zircaloy during the reactor operation, producing Zr-oxide and hydrogen [3, 28, 29]. Hydrogen can further be incorporated into the zircaloy tube wall causing its degradation [30]. In this context, monitoring the hydrogen content in the zircaloy tube is highly important. Attempts to monitor H-isotopes in zircaloy by LIBS are described in [28, 31, 32], where LIBS techniques based on long laser pulses (nanoseconds laser pulse length (LPL); Nd:YAG laser) [28, 31] and ultrashort pulses (~35 fs LPL; Ti:Sapphire laser) [32] were applied. It should be noted that in all of these experiments, successful registration of hydrogen in zircaloy was achieved.

Apart from H-isotopes detection, another unique application of LIBS should be emphasized. Namely, in [33], stand-off LIBS was employed for monitoring the contaminant materials zirconium and molybdenum (zirconium-molybdate form) in the spent nuclear fuel. LIBS was based on a Nd:YAG laser, and was situated several meters from the basket where the spent fuel was located. Nd:YAG laser radiation was transmitted through a lead-glass window, one meter thick, while the entire process was carried out inside the concrete chamber.

VINCA Institute experiments. Hydrogen detection in various materials that are interesting for fusion technology has been tested at the VINCA Institute. Two sets of experiments were conducted using proxy targets for the reactor wall materials bearing hydrogen – polymer polymethyl methacrylate (PMMA; $(C_5O_2H_8)_n$), i. e. metal (titanium). The experimental LIBS apparatus shown in Fig. 2b was used to monitor hydrogen in these samples. The apparatus is similar to those used in references [1, 18, 34, 35] and is based on a pulsed TEA CO_2 laser, utilizing the time-integrated space-resolved measurement. The TEA CO_2 laser produces a specific pulse (Fig. 2b, inset), consisting of the initial spike (FWHM of ~100 ns) and a tail (~2 μs in length). This specific pulse shape affects the laser-target interaction [36]. In one approximation, the spike initiates plasma above the sample, while the tail additionally heats it.

PMMA polymer. Plasma generation on this target was only possible using the metal sub-target (Fig. 2b). The laser intensity was relatively low, of the order of 50 MW/cm^2, unlike in [37, 38], where the plasma was produced using much higher laser intensities. Generally, the studies of PMMA polymer are interesting for several reasons: it is a good proxy material for some fusion materials containing hydrogen; laser-ablator interaction is of high importance in direct-drive inertial confinement fusion (DDICF). Namely, in DDICF, DT

fuel is placed inside a capsule where the outer layer is typically plastic; the study of laser-organic material/polymer interaction is scarce in the literature, and besides nuclear technology, LIBS as a modern spectroscopic method can be used for identification of the given polymer (based on C/H ratio) [39].

In our experiments, in order to induce plasma above the PMMA, an accumulation of 150 pulses was needed (Fig. 2b, inset), i. e., plasma was created after the channel through the polymer was made, and laser radiation reached the sub-target surface. Shock-wave plasma created in this manner propagates through the PMMA channel, heating the polymer and causing various effects like melting, vaporization, dissociation, etc. [1]. Figures 3a,b show hydrogen spectral lines H_α and H_β detected from PMMA. The electron number density evaluated from the H_β line profile [1] was around 10^{16} cm^{-3}. Aside from the hydrogen detection, other PMMA-originated and plasma-generated species were registered such as C, O, C_2, and CN. Figures 3c,d depict the registered emission spectra of C_2 and CN molecules. It should be pointed out that, in order to create CN molecules, the addition of a small quantity of air was required. Finally, based on experimental results, the ionization, rotation, and vibrational temperature were estimated at 16500, 4500, and 7000 K, respectively.

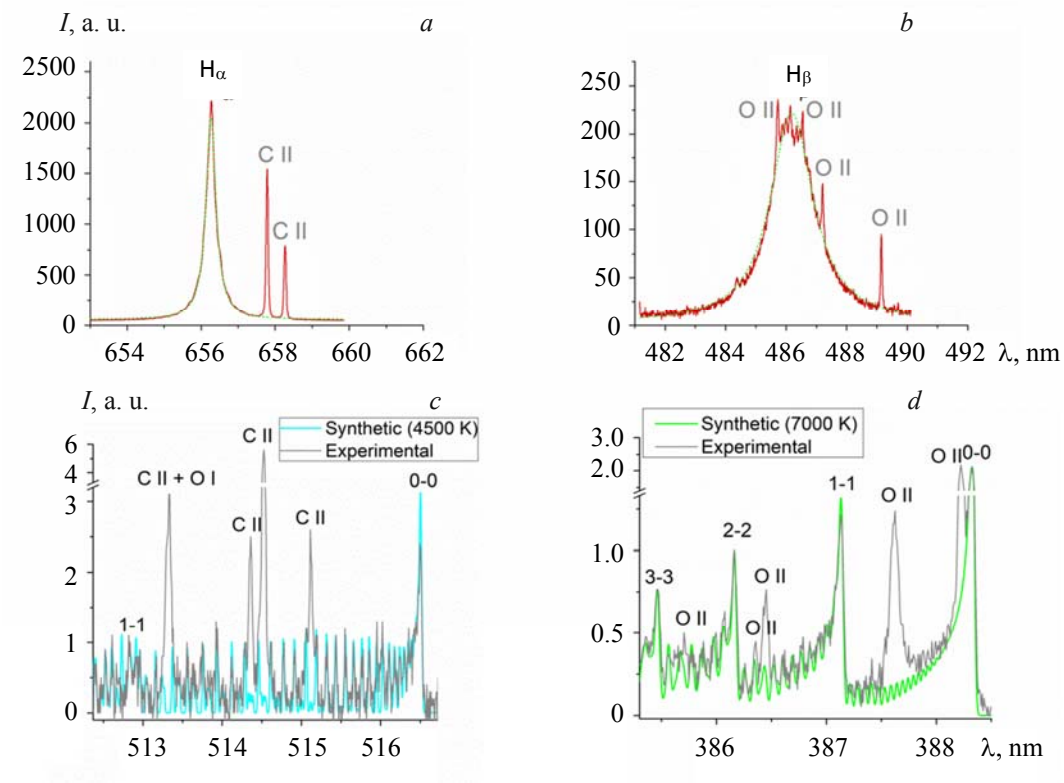

Fig. 3. LIBS spectra acquired from plasma above the PMMA-steel sub-target system: (a) H_α 656.27 nm line; (b) H_β 486.13 nm line; (c) part of spectra of C_2 Swan and (d) CN violet system [1].

Titanium target. Titanium, especially its alloys, is an attractive material in nuclear fusion technology [8]. Titanium has excellent properties, like high melting and boiling temperature, good mechanical and anticorrosive characteristics, and low activation. Its weakness as a reactor material is its affinity to hydrogen. In the VINCA Institute experiment, Ti was employed as a proxy metal for other metals used in fusion reactors that can contain hydrogen. Analysis of the hydrogen-isotope (protium) inside Ti is relatively scarce in the literature [40, 41]. In [40] nuclear reaction and XRD analysis were employed, while in [41] it was LIBS based on a Nd:YAG laser. In our experiment, LIBS based on a pulsed TEA CO_2 laser was applied. Hydrogen was monitored in two Ti-samples (Fig. 4), i.e. the one containing only the hydrogen incorporated during the production stage, and the second one with additionally incorporated hydrogen. Namely, it is well known that during the Ti production process, hydrogen accumulation in titanium can occur [41]. In our LIBS experiments, hydrogen was detected in plasma induced on both samples. Hydrogen presence was monitored via the H_α line. The laser intensity was ~50 MW/cm^2, and the plasma was induced in a vacuum ambiance.

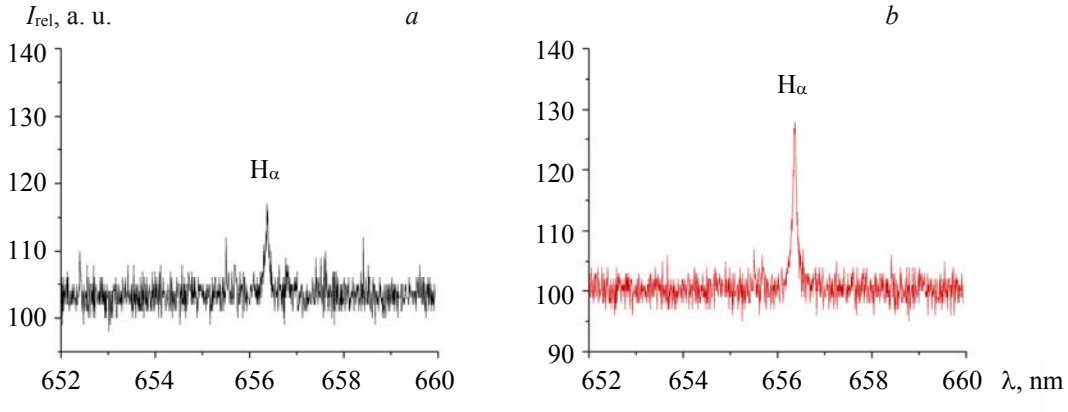

Fig. 4. LIBS signal of H_α line in Ti-sample with (a) "production" and (b) "additionally incorporated" hydrogen.

Conclusions. This review focuses on the monitoring/detection of hydrogen isotopes in nuclear materials. Special attention was devoted to the optical emission spectroscopy method, laser-induced breakdown spectroscopy, for detecting hydrogen isotopes in fusion reactor materials because their presence can induce severe problems in terms of material degradation and lifetime reduction. The fusion reactor materials, among others properties, should have a low affinity to H-isotopes; thus, hydrogen monitoring is essential. Compared to other available methods, the advantages of LIBS are its efficiency, reliability, and speed of analysis. The investigations presented in the literature, including our research at the VINCA Institute, showed that LIBS is a highly promising method for applications in nuclear/fusion technologies.

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