Tapered plastic optical fiber coated with ZnO nanostructures for the measurement of uric acid concentrations and changes in relative humidity

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Tapered plastic optical fiber coated with ZnO nanostructures for the measurement of uric acid concentrations and changes in relative humidity

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ABSTRACT

Simple sensors are proposed and demonstrated using a tapered POF coated with ZnO nanostructures for measurement of different concentrations of uric acid in de-ionized water and changes in relative humidity (RH). The sensor operates based on intensity modulation technique. The tapered POF were fabricated by etching method using acetone, sand paper and de-ionized water to achieve a waist diameter of 0.45 mm and tapering length of 10 mm. The tapered fiber were then coated with ZnO nanostructures using sol–gel immersion method on ZnO seeded and non-seeded fiber. As the concentration of the uric acid was varied from 0 ppm to 500 ppm, the output voltage of the sensor using tapered POF with seeded ZnO nanostructures increased linearly with a higher sensitivity of 0.0025 mV/ppm compared to 0.0009 mV/ppm for unseeded tapered POF coated with ZnO. Both samples showed almost similar linearity of the response cuves of about 98.2%. The tapered POF with ZnO nanostructures interact with uric acid due to strong electrostatic interaction resulting in the increase in response with the increasing concentration. In addition, the seeded ZnO nanostructure could significantly enhance the transmission of the sensor that is immersed in solutions of higher concentration. On the other hand, for both samples, the change in the intensity of the transmitted light of the tapered POF coated with ZnO nanostructures decreases linearly with relative humidity. The tapered POF with grown (seeded) ZnO provides better sensitivity at 0.0258 mV/% with a slope linearity of 95.48%. The ZnO nanostructures that are exposed to an environment of humidity causes rapid surface adsorption of water molecules and changes in optical properties. The tapered POF coated with ZnO nanostructures using the seeding technique causes an increase in both effective RI of surrounding medium and absorption coefficient of the ZnO nanostructures surfaces and leads to larger leakage of light. Results show that tapered POF with seeded ZnO nanostructures enable to increase the sensitivity of fiber for uric acid detection as well as relative humidity. The proposed sensors provide numerous advantages such as simplicity of design, low cost of production, higher mechanical strength and easier to handle compared to silica fiber optic.

1. Introduction

Uric acid is a heterocyclic compound of carbon, nitrogen, oxygen, and hydrogen. Uric acid is a product of the metabolic breakdown of purine nucleotides. Monitoring of uric acid is essential because abnormal levels of uric acid lead to several diseases like gout, Lesh–Nyhan syndrome, renal failure, hyperuricaemia, and physiological disorders, while patients with Wilson's disease are observed to have low uric acid level [1]. High level of serum uric acid is also considered as a risk factor for myocardial infarction and stroke [2]. Therefore, the need for uric acid biosensors is tremendously increasing [3–6].

Several method of detecting uric acid has been reported by research such as amperometric principles and potentiometer [5]. On the other side, wide variety of nanostructures such as zinc oxide

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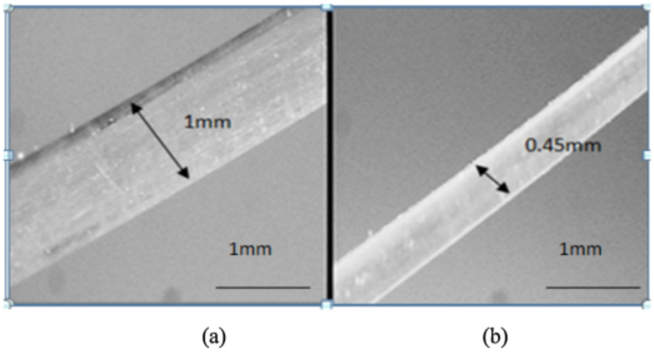


Fig. 1. (a) and (b) Microscope images of the original un-tapered, tapered POF, which have a cladding diameter of 1 mm and 0.45 mm, respectively.

(ZnO) nanowires, nanotubes and nonporous material have also attracted great interest to researchers due to its unique advantages in combination with immobilized enzymes and can enhance the direct electron transfer between the enzyme's active sites and the electrons [5]. ZnO nanowires grown on the surface of gold coated flexible plastic substrate results as good uric acid biosensor [7]. However, more research on reducing the cost of coating is carried out.

Apart of sensing uric acid, researchers are also studying on relative humidity sensors using coated and grown materials on fiber. Recently, evanescent wave sensors have been proposed and demonstrated for humidity measurement. For instance, Muto et al. demonstrated humidity sensors with sensitive coating where the water absorbed changes the refractive index of the coating layer and subsequently transforms the lossy fiber into a light guide [8]. Later, humidity sensing were demonstrated using agarose gel and nanostructured films that were deposited onto tapered fibers [9–14]. However, the fabrication procedure is very time consuming, dependent upon the design and has limited exposed interaction length.

In our previous work, we have demonstrated a simpler fabrication method of coating sensitive material [15–16] onto the tapered plastic optical fiber which received much attention for sensing applications [17–19]. As this is attributed to the characteristics of tapered fibers which allow a higher portion of evanescent field to travel inside the cladding and thus it is more sensitive to the physical changes of its surrounding. In comparison with silica based fiber, plastic optical fibers (POFs) show several advantages such as ease of handling, mechanical strength, disposability and easy mass production of components and system [20]. Besides that, POFs stand out for their greater flexibility and resistance to impacts and vibrations, as well as for greater coupling of light from the light source to the fiber [21]. In this paper, tapered POF were coated with ZnO nanostructure grown using the seeded and non seeded technique for sensing uric acid concentration and detecting changes in relative humidity (RH). The ZnO coating on the tapered fiber induce changes of the optical properties in response to an external stimulus. Besides that, looking at the compatibility of ZnO crystal structure, there is no apparent difference between polymers and silica, as both do not have defined crystal structures. The deposition of ZnO nanostructures have been demonstrated [22–23]. The measurement is based on intensity modulation technique where the intensity of the transmitted light is measured for different uric acid concentration and relative humidity changes.

2. Experiment

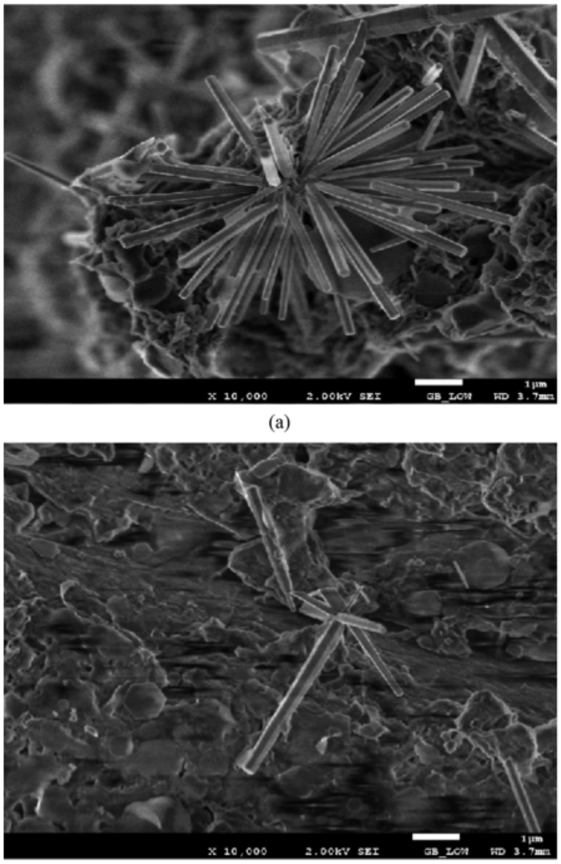
The tapered POF are prepared based on chemical etching technique using acetone, de-ionized water and sand paper. The POF used has an overall cladding diameter of 1 mm, a numerical aperture of 0.51 and an acceptance angle of 61°. The refractive index of the core and cladding are 1.492 and 1.402, respectively. The acetone was applied to the POF using a cotton bud and neutralized with the de-ionized water. The acetone reacted with the surface of the polymer to form milky white foam on the outer cladding which was then removed by the sand paper. This process was repeated until the tapered fiber has a stripped region waist diameter of 0.45 mm. The total length of the tapered section was 10 mm. Finally, the tapered POF fibers were cleansed again using de-ionized water. These tapered plastic fiber were then coated with seeding and non-seeding ZnO nanostructures.

The preparation for ZnO nanostructures on the fiber consists of two primary steps: First, the seed layer to grow the ZnO nanostructures on the fiber was developed using a simple manual dip coating technique. The seeded solution was prepared using zinc acetate dehydrate ($Zn(CH_3COO)_2 \cdot 2H_2O$) as a precursor dissolved in isopropanol with molarity of 0.025 M. The solution was stirred at 60 °C for 2 h in ambient to yield a clear and homogenous solution. Then, the solution was cooled down to room temperature for the coating process. The fiber was manually dipped into the seeding solution and was dried at 50 °C to evaporate the solvent and to remove the organic residuals. This coating and drying method was repeated for five times to increase the thickness of the fibers.

Following this, another solution has been prepared to grow ZnO nanostructures on the bare tapered fiber and ZnO seeded tapered fiber. This is done by dissolving 0.01 M zinc nitrate hexahydrate ($Zn(NO3)_2 \cdot 6H_2O$) and 0.01 M hexamethylenetetramine (HMTA) in 100 ml deionized water. The deposition process of ZnO nanorods on the fibers is performed using sol–gel immersion method by suspending the bare and seeded-ZnO fibers in the growth solution at 60 °C for 15 h.

Fig. 1 (a) and (b) shows the microscope images of the original un-tapered, tapered POF, which have a cladding diameter of 1 mm and 0.45 mm, respectively. Fig. 2 shows the FESEM images with ZnO nanostructures grown on seeded tapered fiber (Fig. 2(a)) and on non seeded tapered fiber (Fig. 2(b)).

Fig. 3 shows the experimental setup for the proposed sensor to detect different uric acid concentration using the fabricated tapered POF with ZnO nanostructures. The setup consists of a light source, an external mechanical chopper, the proposed probe, a highly



(a) (b)

Fig. 2. FESEM images with ZnO nanostructures grown on seeded tapered fiber (a) and on non seeded tapered fiber (b).

sensitive photo-detector, a lock-in amplifier and a computer. The input and output ports of the tapered POF are connected to the laser source and photo-detector, respectively. The light source used in this experiment is a He–Ne laser operates at a wavelength of 633 nm with an average output power of 5.5 mW. It was chopped at a frequency of 113 Hz by a mechanical chopper to avoid the harmonics from the line frequency which is about 50 Hz to 60 Hz. The 113 Hz frequency was chosen as an odd number to prevent multiplication of 50 Hz and 60 Hz besides it is an acceptable value of output and stability. In addition to that, an increase to the value of chopper frequency causes the output voltage and stability to decreases. The light source was launched into the tapered POF with ZnO nanostructures placed in a Petri dish filled with the uric acid solution. The output lights were sent into the silicon photo-detector (818 SL, Newport) and the electrical signal was fed into the lock-in amplifier (SR-510, Stanford Research System) together with the reference signal of the mechanical chopper. The output that is resulted from the lock-in amplifier was connected to a computer through an RS232 port interface and the signal was processed using Delphi software. The reference signal from the chopper was matched with the input electrical signal from the photo-diode. This allows a very sensitive detection system that will remove the noise generated by the laser source, photo-detector and the electrical amplifier in the photo-detector. In the experiment, the performance of the proposed sensor was investigated for various uric acid concentrations. During the experiment, the errors caused by temperature were considered negligible and the temperature was kept constant at 25 °C.

The setup used to measure relative humidity using the fabricated tapered POF coated with ZnO nanostructures is shown in Fig. 4. Laser source (He–Ne) is launched into the tapered POF placed in a sealed chamber with a dish filled with saturated salt solution. The sealed chamber is constructed with a hole and the tapered POF is introduced through it into the sealed receptacle and suspended to saturated salt solutions in order to simulate different values of relative humidity. In the experiment, the performance of the proposed sensor was calibrated for relative humidity ranging from 50% to 80% using 1365 data logging humidity–temperature meter.

3. Results and discussions

Fig. 5 shows the variation of the transmitted light from the tapered POF coated with ZnO nanostructures against the concentration of uric acid solution. Inset of Fig. 4 shows the refractive index of the uric acid solution (as measured by using METTLER Toledo RE40D refractometer) against the uric acid concentration. As the concentration of uric acid increases from 0 ppm to 500 ppm, the refractive index of the solution also increases from 1.3330 to 1.3336. As shown in the figure, the output voltage from the photo-detector, which corresponds to the transmitted light intensity linearly, increases as the concentration of uric acid solution increases. It is found that the sensitivity of the tapered POF with

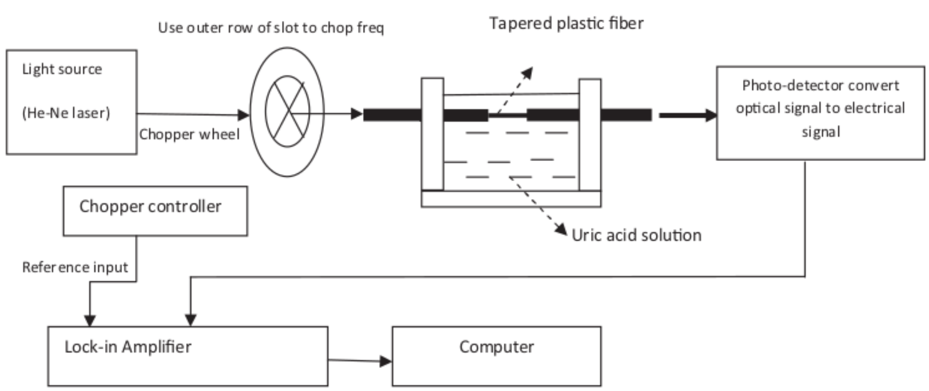


Fig. 3. Experimental setup for the proposed biosensor to measure uric acid concentration using a tapered POF with coated with ZnO nanostructures.

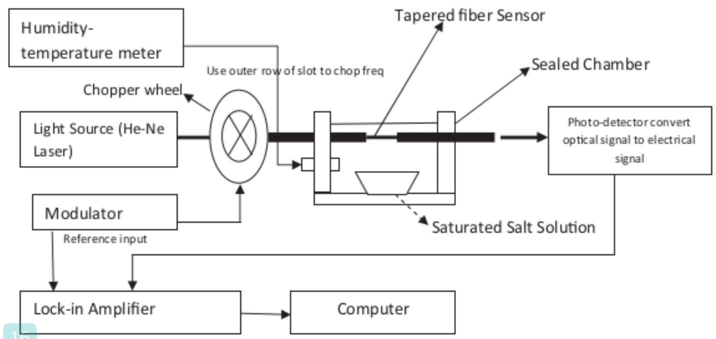


Fig. 4. Experimental setup for the proposed relative humidity sensor using a tapered POF with ZnO nanostructures.

non-seeded ZnO is 0.0009 mV/ppm with a slope linearity of more than 98.19% and limit of detection of 18.9 ppm. On the other hand, the sensitivity of tapered POF with seeded ZnO is 0.0025 mV/ppm with a slope linearity of more than 98.28% and limit of detection of 5.6 ppm. Chemical can be classified as electrolytes and non-electrolytes depending on the dissociation of their ions in solutions [24]. Uric acid is electrolyte, a substance containing free ions that make the substance electrically conductive. The tapered POF coated with ZnO nanostructures interact with uric acid due to strong electrostatic interaction and response with the increasing concentration [7]. However the seeded ZnO nanostructure could significantly enhance the transmission of the sensor that is immersed in solutions of higher concentration. These results show that the proposed sensor is applicable and useful for the detection of biomolecular concentration such as uric acid as the sensor has the ability to provide real time measurement.

Fig. 6 shows the sensing result of the transmitted light from the tapered POF coated ZnO nanostructures grown using the seeded and non seeded technique against the relative humidity. The change in the intensity of the transmitted light of the tapered POF coated ZnO nanostructures decreases linearly with relative humidity. It is found that the sensitivity of the tapered POF coated non seeded ZnO nanostructure is 0.0057 mV/% with a slope linearity of more than 91.67% and limit of detection of 13.84%. On the other hand, the sensitivity of tapered POF caoted ZnO nanostructures grown on seed fiber is 0.0258 mV/% with a slope linearity of more than 95.48% and limit of detection of 0.143%. The limit of detection (LOD) is calculated by dividing the standard deviation with the sensitivity, which indicates that the system is more efficient when the value of LOD is lower. According to Liu et al., the effective reflective index (RI) of ZnO composite varies from 1.698 to 1.718 with relative humidity change from 10% to 95% [14]. The ZnO composite are exposed to an environment of humidity causes rapid surface adsorption of water molecules. The optical properties of ZnO composite surfaces are modulated by the surface adsorption of water molecules. The increase of water molecules being absorbed on ZnO composite results in an increase of relative humidity [14]. The increasing water molecules cause an increase in both effective RI of surrounding medium and absorption coefficient of the ZnO composite surfaces and leads to larger leakage of light.

Reversibility of the results is another important factor in the operation of any sensor system, so in the next study, this parameter is tested for the reported system. The results of the output measurement as a function of concentration are recorded for two different runs and the results are compared. As can be noticed from Fig. 7, the maximum difference between the two runs is about ±0.05 mV, which is acceptable for a full-scale output of 7.15 mV. Fig. 8, gives the maximum difference between the two runs to about ±0.15 mV, which is acceptable for a full-scale output of 2.5 mV.

The performance characteristic of the proposed sensor is summarized in Tables 1 and 2. Overall, the sensor is observed to be sufficiently stable with standard deviations of 0.017 mV and 0.014 mV for tapered POF coated with ZnO nanostructures tested on uric acid with different concentration as being recorded for the

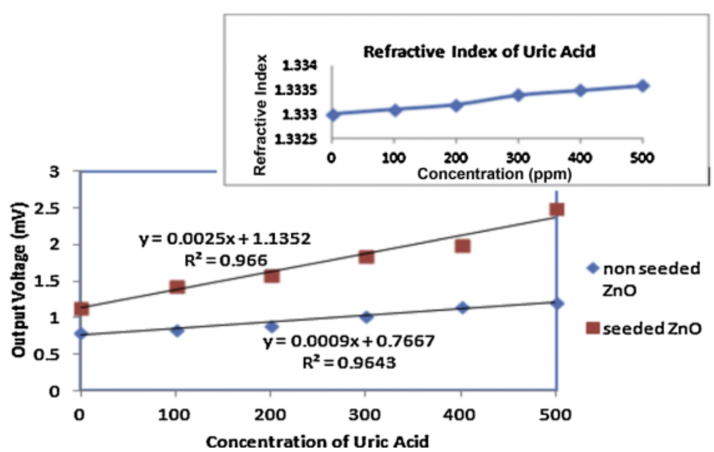


Fig. 5. Output voltage against uric acid concentrations for the proposed tapered POF with ZnO nanostructure. Inset shows the measured refractive index at different composite solution concentrations.

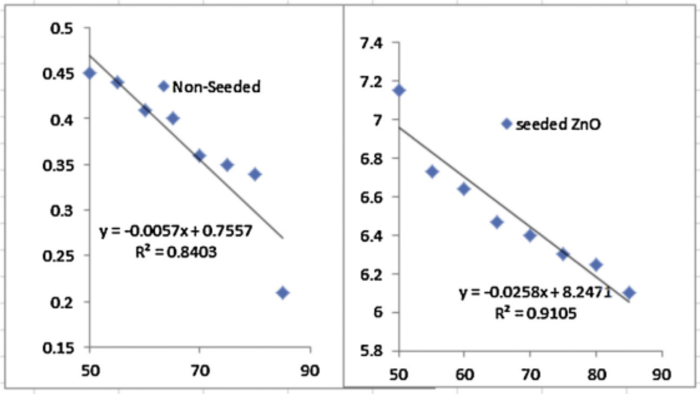


Fig. 6. Sensing results for the proposed relative humidity sensor using a tapered POF coated with ZnO nanostructure.

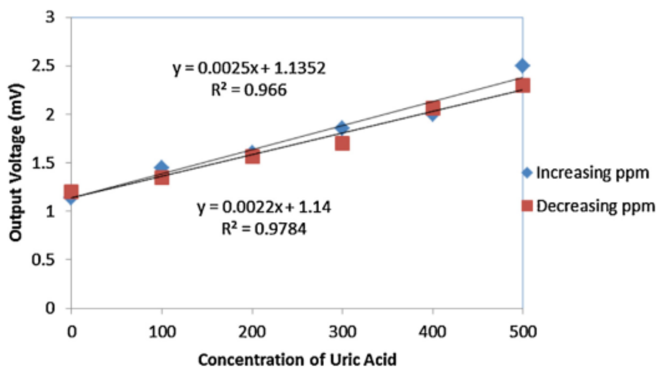


Fig. 7. The reversibility of the results obtained for two different runs (concentration of uric acid).

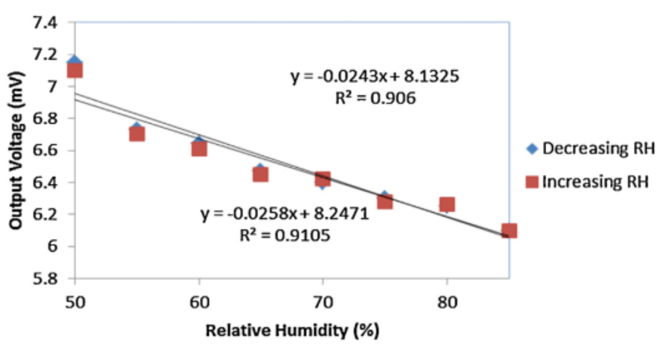


Fig. 8. The reversibility of the results obtained for two different runs (relative humidity).

Table 1
The performance of the proposed uric acid detection sensor.

Performances	ZnO coating	ZnO grown
Sensitivity	0.0009 mV/ppm	0.0025 mV/ppm
Linearity	98.19%	98.28%
Standard deviation	0.017 mV	0.014 mV
Limit of detection	18.9 ppm	5.6 ppm

Table 2
The performance of the proposed RH sensor.

Performances	ZnO coating	ZnO grown
Sensitivity	0.0057 mV/%	0.0258 mV/%
Linearity	91.67%	95.42%
Standard deviation	0.0789 mV	0.0037 mV
Limit of detection	13.84%	0.143%

time duration of 500 s for non seeded and seeded, respectively. The sensor is observed to be sufficiently stable for relative humidity changes with standard deviations of 0.0789 mV and 0.0037 mV for tapered POF coated with ZnO nanostructure as recorded in time duration of 100 s for non seeded and seeded, respectively. These results show that the proposed sensor coated with ZnO nanostructures grown on seeded fiber is suitable and showed better performance in measuring both uric acid and relative humidity in real time because the lower value for stability shows that the system is more stable.

4. Conclusion

Simple sensors are proposed and demonstrated using a tapered POF coated with ZnO nanostructures grown using the seeded and non seeded techniques for measurement of different concentrations of uric acid in de-ionized water and changes in relative humidity. The tapered POF was fabricated by etching method using acetone, sand paper and de-ionized water to achieve a waist diameter of 0.45 mm and tapering length of 10 mm. The tapered fiber were then coated with ZnO nanostructures using sol–gel immersion method on seeded and non-seeded fiber. As the solution concentration of the uric acid varies from 0 ppm to 500 ppm, the output voltage of the sensor using tapered POF with seeded ZnO nanostructure increases linearly with better sensitivity of 0.0025 mV/ppm and a linearity of more than 98.28% and limit of detection of 5.6 ppm. The tapered POF with seeded and non-seeded ZnO nanostructure interact with uric acid due to strong electrostatic interaction and response with the increasing concentration. However the seeded ZnO nanostructure could significantly enhance the transmission of the sensor that is immersed in solutions of higher concentration. On the other hand, the change in the intensity of the transmitted light of the tapered POF with seeded and non-seeded ZnO nanostructure decreases linearly with relative humidity. The tapered POF with seeded ZnO nanostructure provides better sensitivity at 0.0258 mV/% with a slope linearity of more than 95.48% and limit of detection of 0.143%. The ZnO nanostructure are exposed to an environment of humidity causes rapid surface adsorption of water molecules. The optical properties of ZnO nanostructure surfaces are modulated by the surface adsorption of water molecules and causes an increase in both effective RI of surrounding medium and absorption coefficient of the ZnO nanostructure surfaces and leads to larger leakage of light. Results shows that tapered POF with seeded ZnO nanostructure enable to increase the conductance of fiber for uric acid detection and also applicable as relative humidity sensor.

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