Li$_4$Ti$_5$O$_{12}$ Coating Layer as Li$^+$ Conductor and Cycle Stabilizer for SnO$_2$ Anode

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Li$_4$Ti$_5$O$_{12}$ as Li$^+$ conductor and cycle stabilizer was coated successfully on mesoporous SnO$_2$ spheres (M-SnO$_2$) via in-situ synthesis. The structure and morphology of as-prepared samples were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The results indicated that the SnO$_2$ spheres were coated fully by Li$_4$Ti$_5$O$_{12}$ layer, and the Li$_4$Ti$_5$O$_{12}$ layer did not change the structure or morphology of M-SnO$_2$. Electrochemical impedance spectra (EIS) and charge/discharge tests showed that the outer Li$_4$Ti$_5$O$_{12}$ coating layer enhanced Li$^+$ diffusion in SnO$_2$ anode. The Li$^+$ diffusion coefficient for S-1 and S-2 reached to 9.87×10$^{-13}$ cm$^2$ s$^{-1}$ and 1.82×10$^{-12}$ cm$^2$ s$^{-1}$, while the value of M-SnO$_2$ was only 6.36×10$^{-13}$ cm$^2$ s$^{-1}$. The cycle stability and rate cycle property of SnO$_2$-based anode were improved significantly due to promotional role of Li$_4$Ti$_5$O$_{12}$ coating layer.

Keywords: SnO$_2$, Li$_4$Ti$_5$O$_{12}$, Li$^+$ conductor, cycle stabilizer, Li$^+$ diffusion coefficient

1. INTRODUCTION

Compared to commercial graphite/carbon, SnO$_2$ has been considered as one of the candidate anode for lithium ion batteries due to its higher theoretical capacity (782 mAh g$^{-1}$) [1, 2]. However, on the one hand, large volume expansion/contraction occurs during Li$^+$ insertion/extraction, leading to the electrodes pulverization and cracking [3, 4]. On the other hand, Li$^+$ diffusion in SnO$_2$ is difficult, which results in its poor cyclability and rate performance [5]. Nowadays, there are some effective strategies to suppress the volume expansion/contraction, such as synthesizing nanoparticles or core-shell particles, or mixing with conductive carbon materials. Nanoparticles include nanospheres, nanotubes, nanosheets, nanobelts and nanofibers, etc. Core-shell particles include carbon coating and TiO$_2$ coating, etc. The composites include SnO$_2$ mixing with graphene, mesoporous carbon,
carbonaceous mesophase spherules, carbon tubes, or carbon fibers. [6-10]. Those strategies have improved the cyclability of SnO\textsubscript{2} anode, but the cyclability especially rate cycle performance can not be improved further due to poor Li\textsuperscript{+} diffusion. Therefore, how to construct SnO\textsubscript{2} anode with high rate cyclability becomes important.

In recent years, many Li\textsuperscript{+} conductors have been synthesized and studied. The Li\textsuperscript{+} conductors are usually inorganic lithium salt, such as Li\textsubscript{7}Al\textsubscript{5} (A=Sb, Bi, Ta, Zr), LiBiO\textsubscript{2}, Li\textsubscript{3}BiO\textsubscript{3}, Li\textsubscript{3}BiO\textsubscript{4}, Li\textsubscript{3}BiO\textsubscript{5}, Li\textsubscript{4}SiO\textsubscript{4}, Li\textsubscript{4}Ti\textsubscript{5}O\textsubscript{12}, etc [11-14]. However, not all those inorganic lithium salt are suitable using as Li\textsuperscript{+} conductor under room temperature condition, some lithium salt (such as Li\textsubscript{7}MO\textsubscript{6}) need high temperature to ensure the high Li\textsuperscript{+} conduction coefficient. Among the above-mentioned lithium salts, Li\textsubscript{4}Ti\textsubscript{5}O\textsubscript{12} is one of the most popular Li\textsuperscript{+} conductor due to its “zero strain” and high Li\textsuperscript{+} diffusion coefficient. The reported value of Li\textsuperscript{+} diffusion coefficient was various, such as Yu-Jun Bai et al. reported the value was 3.38\times10^{-15} \text{ cm}^2 \text{s}^{-1}[15] and 3.38\times10^{-15} \text{ cm}^2 \text{s}^{-1}[16], and some reports reported the value was \sim 2.0\times10^{-11} \text{ cm}^2 \text{s}^{-1} [17-18]. Our previous work studied the Li\textsuperscript{+} diffusion coefficient value of carbon coated Li\textsubscript{4}Ti\textsubscript{5}O\textsubscript{12} spheres was 8.53\times10^{-13} \text{ cm}^2 \text{s}^{-1} [19]. Although the Li\textsuperscript{+} diffusion coefficient value of Li\textsubscript{4}Ti\textsubscript{5}O\textsubscript{12} was various, but the various reported values were confirmed that Li\textsubscript{4}Ti\textsubscript{5}O\textsubscript{12} is a fast Li\textsuperscript{+} conductor. The values were various because the measure conditions were not same. From those reported values and the Li\textsuperscript{+} diffusion coefficient of pure SnO\textsubscript{2} (Ref. 19), a conclusion can be obtained that the Li\textsuperscript{+} diffusion coefficient of pure Li\textsubscript{4}Ti\textsubscript{5}O\textsubscript{12} was two orders of magnitude higher than that of pure SnO\textsubscript{2}. In recent years, although some works were reported SnO\textsubscript{2}-Li\textsubscript{4}Ti\textsubscript{5}O\textsubscript{12} composites, such Han et al. [18] synthesized the anode materials composed of 2D Li\textsubscript{4}Ti\textsubscript{5}O\textsubscript{12} nanosheets and 0D SnO\textsubscript{2} nanoparticles, however the composite delivered 140 mA h g\textsuperscript{-1} after 30 cycles at current density of 0.1 mA cm\textsuperscript{-2}. The capacity was low because the 2D Li\textsubscript{4}Ti\textsubscript{5}O\textsubscript{12} nanosheets were not coated on SnO\textsubscript{2} nanoparticles. Hao et al. [19] synthesized Li\textsubscript{4}Ti\textsubscript{5}O\textsubscript{12}-SnO\textsubscript{2} composite via sol-gel and high temperature solid-state methods. Its capacity was lower than 200 mA h g\textsuperscript{-1} at 0.5 mA cm\textsuperscript{-2} after 42 cycles because SnO\textsubscript{2} was simply mixed with Li\textsubscript{4}Ti\textsubscript{5}O\textsubscript{12}. Similarly, Wang et al. [20] also synthesized Li\textsubscript{4}Ti\textsubscript{5}O\textsubscript{12}-SnO\textsubscript{2} composite. However, its capacity was low because SnO\textsubscript{2} was simply mixed with Li\textsubscript{4}Ti\textsubscript{5}O\textsubscript{12}.

For improving the cyclability and rate property of SnO\textsubscript{2} anode, Li\textsubscript{4}Ti\textsubscript{5}O\textsubscript{12} coated M-SnO\textsubscript{2} were prepared via simple synthesis strategies. Here, Li\textsubscript{4}Ti\textsubscript{5}O\textsubscript{12} layer acts as volume expansion/contraction buffer layer and Li\textsuperscript{+} conductor to improve the cyclability and rate property of SnO\textsubscript{2} anode.

2. EXPERIMENTAL

2.1 Sample preparation

The synthesis process of Li\textsubscript{4}Ti\textsubscript{5}O\textsubscript{12} coated M-SnO\textsubscript{2} spheres were illustrated in Fig. 1. Firstly, 3.9630 g glucose and 2.8040 g SnCl\textsubscript{4}\cdot5H\textsubscript{2}O was dissolved into mixed solution of 10.0 mL deionized water and 60.0 mL absolute alcohol in a 100 mL Teflon-lined stainless steel autoclave and placed in an oven at 150 °C for 12 h. The precipitation was collected by centrifugation and washed with deionized water and ethanol several times after cooling down to room temperature. The slurry was dried in vacuum oven under 60 °C and heated at 500 °C under air atmospheres for obtaining M-SnO\textsubscript{2}. Next,
0.2958 g M-SnO₂ were dispersed into 15.0 mL absolute alcohol by strong stirring and ultrasonic, then 160 μL heptanoic acid, 300 μL ammonium hydroxide, 300 μL tetrabutyl titanate (S-1) or 700 μL tetrabutyl titanate (S-2) were dropped, and kept stirring. After 30 min, 7.0 mL deionized water was poured into above suspension quickly and kept stirring for another 2 h. The white precipitation was poured into a 100 mL Teflon-lined stainless steel autoclave and placed in an oven at 150 °C for 12 h. After cooling down, the precipitation was collected by centrifugation and washed with deionized water and ethanol several times, then re-dispersed into mixed solution of 15.0 mL absolute alcohol and 30.0 mL deionized water in a 100 mL Teflon-lined stainless steel autoclave and placed in 160 °C for 6 h. The white precipitation was collected by centrifugation and washed with deionized water and ethanol several times, and dried in 60 °C. Finally, the powder was heated in 500 °C under air atmospheres for final product. The sample of pure m-SnO₂ spheres named as S-0.

**Figure 1.** Schematic illustration of the synthesis of Li₄Ti₅O₁₂ coated M-SnO₂ spheres. Red circles are cross-section view. From the cross-section view, the inner M-SnO₂ can be seen.

2.2 Characterizations

The crystal structure of the samples was studied by XRD (Shimadzu-XRD-7000S, Cu-Kα radiation, λ= 1.54056 Å, 40kV, 40 mA). The morphology was investigated via SEM (S4800) and TEM (FEI Tecnai G20).

2.3 Electrochemical testing

As-prepared samples (80 wt.%), conductive carbon black (10 wt.%) and polyvinylidene fluoride (10 wt.%) binder were mixed using N-methyl pyrrolidine as solvent for homogeneous slurry, then the slurry was pasted uniformly onto a copper foil and dried to give the electrodes with loading
materials of ~2.5 mg cm\(^{-2}\). The CR2032 coin cells were assembled in an argon-filled dry glove box using lithium metal foil as the counter electrode. The electrolyte was 1 mol·L\(^{-1}\) LiPF\(_6\)/\((\text{EC}+\text{DME}+\text{EMC})\) (1/1/1 by volume) solution. Celgard 2400 polypropylene membrane was used as the separator. Galvanostatic charge/discharge tests were performed on LAND battery tester with a cut-off voltage of 0.01~2.0 V. Electrochemical impedance spectra (EIS) in the frequency range 10\(^{-2}\) to 10\(^{5}\) Hz was performed by an Autolab PGSTAT 128 N electrochemical workstation. Cyclic voltammograms (CV) was acquired in same electrochemical workstation in the voltage range 0 to 3 V.

3. RESULT AND DISCUSSION

3.1 Physical properties

Fig. 2 shows XRD results of the samples. As shown in the figure, the diffraction peaks of S-1 and S-2 were consistent with standard SnO\(_2\) diffraction peaks (JCPDS No: 41-1445) and Li\(_4\)Ti\(_5\)O\(_{12}\) diffraction peaks (JCPDS No: 49-0207), indicating that there was no evidence of impurities. The intensity of Li\(_4\)Ti\(_5\)O\(_{12}\) peaks in S-1 was weaker than that of S-2 due to higher Li\(_4\)Ti\(_5\)O\(_{12}\) content in S-2.

![Figure 2. XRD results of S-1 and S-2. The green and blue bars on bottom are standard Li\(_4\)Ti\(_5\)O\(_{12}\) and SnO\(_2\) diffraction peaks](image)

The morphology of the samples was observed via SEM and TEM as shown in Fig. 3. Fig. 3(a) shows that the surface of SnO\(_2\)/C spheres was smooth and the spheres particle size was ~1.5 μm. The SnO\(_2\)/C spheres were composed of SnO\(_2\) and pyrolytic carbon originated from glucose. Fig. 3(b) shows that there was no mesoporous in surface or inner of SnO\(_2\)/C spheres. After SnO\(_2\)/C spheres were heated at 500°C under air atmospheres, the carbon was oxidized and the mesoporous were formed on surface
and inner spheres, as shown in Fig. 3(c)-(d). The mesoporous on the surface was beneficial for adsorbing tetrabutyl titanate molecule due to large specific surface. Fig. 3(e) is the SEM image of S-2. The surface was coarser after Li$_4$Ti$_5$O$_{12}$ particles were coated on SnO$_2$. For unclosing more detail of the Li$_4$Ti$_5$O$_{12}$ layer, the sample was observed by TEM. The TEM result was shown in Fig. 3(f)-(i). Fig. 3(f) shows some Li$_4$Ti$_5$O$_{12}$ particles were coated on surface. Fig. 3(g) shows the inner mesoporous were not affected after Li$_4$Ti$_5$O$_{12}$ layer coated because the mesoporous on the SnO$_2$ surface has limited the tetrabutyl titanate adsorbed into inner mesoporous. Fig. 3(h) shows the thickness of Li$_4$Ti$_5$O$_{12}$ layer was 60~90 nm. Fig. 3(i) discloses the detail of the interface between Li$_4$Ti$_5$O$_{12}$ and SnO$_2$. The lattice fringe of Li$_4$Ti$_5$O$_{12}$ was estimated as 0.48 nm, and the lattice fringe of SnO$_2$ was estimated as 0.26 nm and 0.34 nm. The SEM and TEM results proved that the structure of M-SnO$_2$ was not affected after Li$_4$Ti$_5$O$_{12}$ coating and the structure of final samples was in accordance with the design idea.

![Figure 3. SEM image of SnO$_2$/C spheres (a, b), M-SnO$_2$ (c, d), S-2 (e), TEM images of S-2 (f-i)](image)

3.2 Electrochemical performances

The Li$^+$ diffusion coefficient of the samples was investigated using EIS. Besides S-0, S-1 and S-2, pure Li$_4$Ti$_5$O$_{12}$ was also investigated. Fig. 4(a) shows the EIS results. All the EIS curves were
made of a semicircle which located at high frequent and a line which located at low frequent. The larger the semicircle radius, the poorer the electronic conductivity of the samples [21, 22]. In high frequency, the radius of Li$_4$Ti$_5$O$_{12}$ was smallest, the radius of S-2 was smaller than that of S-1, and the radius of S-0 was largest, indicating that the electronic conductivity of Li$_4$Ti$_5$O$_{12}$ is obviously higher than that of SnO$_2$. Why? Because the band gap of Li$_4$Ti$_5$O$_{12}$ is 2.3 eV [23], while the value of SnO$_2$ is 3.5 eV [24]. The higher the band gap, the more difficult the electron transition between valence band and conduction band. It is well-known that the higher the band gap, the poorer the electronic conductivity of the materials. For calculating the Li$^+$ diffusion coefficient, $-Z'$ against $\omega^{1/2}$ was depicted in Fig. 4(b). Li$^+$ diffusion can be calculated from following equation [17, 25]:

$$D_{Li} = \frac{R^2T^2}{2A^2n^4F^2C_{Li+}^2\sigma^2}$$

where $R$ is the gas constant (8.3145 J mol$^{-1}$ K$^{-1}$), $T$ is the absolute temperature (298 K), $A$ is the area of electrode (1.539 cm$^2$), $n$ is the number of transferred electrons (4.4), $F$ is the Faraday constant (96485.6 C mol$^{-1}$), $C_{Li+}$ is the lithium-ion concentration (0.001 moL cm$^{-3}$), and $\sigma$ is the coefficient of Warburg impedance, which is the slope of $Z'/\omega^{1/2}$ line. $D_{Li+}$ for S-0, S-1 S-2 and pure Li$_4$Ti$_5$O$_{12}$ was calculated as $6.36\times10^{-13}$, $9.87\times10^{-13}$, $1.82\times10^{-12}$ and $8.89\times10^{-11}$ cm$^2$ s$^{-1}$. The Li$^+$ diffusion coefficient of pure Li$_4$Ti$_5$O$_{12}$ here was two orders of magnitude higher than that of M-SnO$_2$ spheres, which was consistent with the reported results [5, 15-17]. These results show $D_{Li+}$ of SnO$_2$ material can be increased substantially by Li$_4$Ti$_5$O$_{12}$ coating.

![Figure 4](image_url)

**Figure 4.** EIS (a) and $Z'/\omega^{1/2}$ line (b) of S-0, S-1, S-2 and pure Li$_4$Ti$_5$O$_{12}$. Dots were experimental data, lines were fitting from dots

Another function of Li$_4$Ti$_5$O$_{12}$ layer is as volume expansion/contraction buffer layer to improve the cycle stability, which can be estimated by galvanostatic charge/discharge cycles. Fig. 5(a) shows the results. As shown in the figure, S-2 was more stable than S-1 and S-0 in 50 mA g$^{-1}$ or in 500 mA g$^{-1}$, indicating that Li$_4$Ti$_5$O$_{12}$ coating layer can improve the cyclability of SnO$_2$. The initial capacity of S-2, S-1 and S-0 at 50 mA g$^{-1}$ was 969.3 mAh g$^{-1}$, 919.6 mAh g$^{-1}$ and 841.4 mAh g$^{-1}$, respectively. When the current density was switched to 500 mA g$^{-1}$, their initial capacity were 731.4 mAh g$^{-1}$, 822.3 mAh g$^{-1}$ and 542.6 mAh g$^{-1}$, respectively. At 50 mA g$^{-1}$ after 20 cycles, S-2 and S-1 became stable, while S-0
was not yet. At 100th cycle, the capacity of S-2, S-1 and S-0 was 282.3, 193.3 and 123.9 mAh g⁻¹. The capacity of S-2 and S-1 was 127.8% and 56.1% higher than that of S-0. At 500 mA g⁻¹ after 15 cycles, the capacity of S-1 and S-0 still declined fast compared to that of S-2, suggesting that the volume expansion/contraction of S-1 and S-0 were severer than that of S-2. S-2 was more stable because the Li₄Ti₅O₁₂ layer was thicker and can relieve the stress of volume expansion/contraction more effectively. For coulombic efficiency, the initial cycle value for S-0 at 50 mA g⁻¹ and 500 mA g⁻¹ was 39.7% and 37.3%, and the value for S-1 was 46.1% and 47.9%, while the value for S-2 was 48.4% and 42.5%, respectively. Those values were lower than 50%. The reason why those values were lower than 50% was determined by the charge/discharge mechanism of SnO₂, which a conversion reaction was occurred in initial discharged. The initial discharge process of SnO₂ can be described by follow equation [26-29]:

SnO₂+8.4Li→Li₁₄.₄Sn+2Li₂O  (1)

After the initial discharged, the reaction was as follow:

Li₁₄.₄Sn⇌4.4Li+Sn                                  (2)

The equation (1) was irreversible, while equation (2) was reversible. From equation (1) and (2), there was 4.4 Li reversible, while there was 8.4 Li reacted with per SnO₂ in initial discharged. Thus, the theoretical coulombic efficiency of initial cycle was 52.38% (4.4/8.4×100%=52.38%) for pure SnO₂. The initial coulombic efficiency was lower than 50% was own to form a SEI film. After the initial cycle, the coulombic efficiency was increase gradually, indicating that the electrodes were become more reversible. However, the increase rate in first 20 cycles was different on S-0, S-1 and S-2 because their Li₄Ti₅O₁₂ content was different. It can see that more Li₄Ti₅O₁₂ content in electrodes, higher the increase rate of coulombic efficiency in first 20 cycles. The result was suggesting that Li₄Ti₅O₁₂ can increase the utilization rate of the SnO₂-based materials. Moreover, the rate cycles can prove the promotional effect of Li₄Ti₅O₁₂ layer on SnO₂ anode as well. Fig. 5(b) shows the rate cycles of the samples. At initial five cycles, the capacity of S-2, S-1 and S-0 was approximately equal. The capacity of S-0, S-1 and S-2 at 5th cycle was 706.5, 641.0 and 604.3 mAh g⁻¹, respectively. However, the capacity decline rate of S-1 and S-0 was more and more severe with the increase of the current density. At 10th (100 mA g⁻¹), 15th (200 mA g⁻¹), 20th (500 mA g⁻¹) and 25th (1000 mA g⁻¹) cycle, the discharge capacity of S-2 was 493.9, 351.9, 263.8 and 170.7 mAh g⁻¹, while the value of S-1 was 371.6, 289.6, 184.5 and 110.3 mAh g⁻¹, and the value of S-0 was only 314.5, 242.5, 109.4 and 35.0 mAh g⁻¹. The above values were indicating that S-1 and S-0 were less stable than S-2. Moreover, when the current density was switched from 500 mA g⁻¹ to 50 mA g⁻¹, the capacity recovery rate of S-2 was obviously higher than S-1 and S-0, which also proved that S-2 was more stable than S-1 and S-0. The above results proved that Li₄Ti₅O₁₂ coating layer played a promotional role on cyclability and rate cycle performance for SnO₂ anode.
Figure 5. Galvanostatic charge/discharge cycles (a) and rate cycles (b)

The lithiation/delithiation details of the samples were investigated by CVs. Fig. 6(a)~(c) show the CVs results. In first scan of S-2, four pairs peaks denoted as I / I’, II / II’, III/III’ and IV/IV’ were observed. The peaks of IV/IV’ were owing to lithiation/delithiation between Li⁺ and Li₄Ti₅O₁₂, and the peaks of I / I’, II / II’ and III/III’ were owing to lithiation/delithiation between Li⁺ and SnO₂. The reaction equations of those peaks were as follows [26-29]:

Peak IV/IV’: Li₄Ti₅O₁₂+3Li→Li₇Ti₅O₁₂  \( (3) \)

Peak III/III’: Li+Sn→LiSn  \( (4) \)

Peak II / II’: 2LiSn +5Li→Li₂Sn₂  \( (5) \)

Peak I / I’: Li₄Sn₂+1.8Li→2Li₄.₄Sn  \( (6) \)

Besides peaks of I / I’, II / II’, III/III’ and IV/IV’, two cathodic peaks denoted as A and B were also observed. The reaction of A peak was as follows [30]:

A: SnO₂+4Li→Sn+2Li₂O  \( (7) \)

While B peak was the result of forming a solid electrolyte interface (SEI) film on Li₄Ti₅O₁₂ (Formed from reaction IV/IV’) [31]. The reaction process of (7)→(4)→(5)→(6) was the decomposition of equation (1). The reactions of I / I’, II / II’, III/III’ and IV/IV’ were reversible, while reactions of A peak and the reaction of forming a SEI film (B peak) were irreversible. After initial scan, the peaks of A and B disappeared and the profile of third scan was similar with that of second scan, indicating that the reactions after first scan were reversible. Besides, the peaks of IV/IV’ fade gradually. Such a phenomenon was universal when Li₄Ti₅O₁₂ content was less in the composite [26-31]. The results
concluded from Fig. 6(b) were consistent with Fig. 6(a). In Fig. 6(c), no redox peaks of IV/IV' and no B peak were shown because there was only SnO₂ in S-0. The lithiation/delithiation details can be also observed from charged/discharge curves as shown in Fig. 6(d)~(f). In initial discharge, a short plateau of Li₄Ti₅O₁₂ was showed in Fig. 6(d) and (f), but no other Li₄Ti₅O₁₂ plateau in other curves.

Figure 6. CVs of S-2 (a), S-1 (b) and S-0 (c), charged/discharged curvy of S-2 (d), S-1 (e) and S-0 (f)

Furthermore, a plateau located at ~0.88V for S-2, ~0.75V for S-1 and ~0.57V for S-0 was also observed in initial discharge curve. Those plateaus were owing to form a SEI film. The results of initial charge-discharge curve were consistent with the results of initial CV scan.

4. CONCLUSIONS
Li$_4$Ti$_5$O$_{12}$ coated M-SnO$_2$ was synthesized via sol-gel, hydro-thermal method and high temperature solid-state methods. The particle size of M-SnO$_2$ was ~1.5 μm, and the thickness of Li$_4$Ti$_5$O$_{12}$ coating layer was 60~90 nm. The Li$_4$Ti$_5$O$_{12}$ layer acted as Li$^+$ conductor and volume expansion/contraction buffer layer for SnO$_2$ anode. The Li$^+$ diffusion coefficient of S-2 reached to 1.82×10$^{-12}$ cm$^2$ s$^{-1}$ compared to 6.36×10$^{-13}$ of S-0. At 50 mA g$^{-1}$, the capacity of S-2 and S-1 was 127.8% and 56.1% higher than that of S-0 after 100$^{th}$ cycle. Moreover, the cyclability and rate cycle performance of SnO$_2$ anode were improved obviously. The results of the research can be applied for preparing other lithium ion anodes, such as Li$_4$Ti$_5$O$_{12}$ coated Fe$_2$O$_3$, Co$_3$O$_4$, NiO, etc.

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