Perspective on Carbon Anode Materials for K$^+$ Storage: Balancing the Intercalation-Controlled and Surface-Driven Behavior

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2. K+ Storage Mechanism in Carbon Anode

The operation mechanism of PIBs is similar to that of LIBs, in which K+ travels between the anode and cathode in a rocking-chair fashion during the charge/discharge process (Figure 1a). Unlike the major intercalation/deintercalation mechanism in the cathode, various K+ storage processes are involved in the anode. Depending on the microstructure and discharge behavior of carbons, several charge storage mechanisms have been proposed including intercalation, adsorption, pore filling, etc.[8] Generally, two main mechanisms have been identified: diffusion-controlled intercalation process and surface-driven capacitive process (Figure 1b).[2,27] In addition, K+ plating is also proposed to form metallic state in the nanopore/voids at the low potential, similar to Na+ plating.[29,30] The intercalation process is based on K+ intercalation/deintercalation into/from interlayers of graphitic carbon layers, and thus the storage capacity relies on ionic state, interlayer structure, and so on. The surface-driven capacitive K+ storage mainly happens at the surface or near surface region and does not pose damage to the electrode. Thus, the storage capacity is related to specific surface area, intrinsic carbon defect/edges, and heteroatom-doped functional sites within carbon materials.

2.1. Graphitic Carbon with Intercalation Involved K+ Storage

Due to the remarkable layer stacking with certain interlayer spacing, K+ storage in graphite materials mainly comes from its intercalation into graphitic layers.[11,13] Such intercalation process adopts a similar staging mechanism as Li-intercalation of graphite layer, in which the K+ completely intercalates into graphite layers until occupying neighboring graphite layers.[12] More specifically, this stage was divided into pseudocapacitive intercalation behavior (C to KC24) and diffusion-limited intercalation (KC24 to KC8).[12] The intercalation process occurs at lower working potential (e.g., <0.5 V vs K+/K), as confirmed by Ji et al. in 2015, delivering a high reversible capacity of 273 mAh g\(^{-1}\), approaching the theoretical capacity of a stage-one KC8 formation (279 mAh g\(^{-1}\)). Meanwhile, sequential formation of KC36, KC34, and KC8 were identified during the intercalation/deintercalation process, as revealed by X-ray diffraction (XRD) patterns (Figure 2).[11] Afterward, the intercalation mechanism was thoroughly studied with operando characterizations and theoretical simulations, revealing the reversible staging transition: C-KC60-KC48-KC36-KC24/KC36-KC24/KC8.[13]

Despite the intercalation mechanism is under development, graphite anode is still considered as a promising candidate in consideration of the low-potential plateau curve for obtaining higher full cell voltage and energy density. However, diffusion of K+ into graphite layers is sluggish and leads to as high as 58% volume expansion with the graphitic structure collapse, thus causing low-rate performance and serious capacity fading during repeated cycling.[27] Designing an appropriate structure of graphite materials is promising to tolerate large volumetric deformation. For example, a new carbon-polyonancrystalline graphite with hollow shape and porous walls was prepared by chemical vapor deposition, which exhibited disorder at
nanometric scales but strict order at atomic scales. This novel structure buffers volumetric expansion during intercalation process, showing 50% capacity retention over 240 cycles. In addition, expanding the layer spacing of graphite materials is also an effective strategy for promoting rapid K\(^+\) intercalation/deintercalation. An et al. presented an expanded graphite with wider interlayer spacing as anode materials for PIBs, which enhanced K\(^+\) diffusion coefficient and delivered a capacity of 263 mAh g\(^{-1}\) at the rate of 10 mA g\(^{-1}\) and managed to improve the cyclability to 500 cycles. Despite these efforts, there is still enough room for further enhancement of both cyclic stability and rate capability.

2.2. Amorphous Carbon with Surface-Driven Capacitive K\(^+\) storage

Amorphous carbons include hard and soft carbons, which are different from graphite with long-range order in crystallographic structure. Hard carbon consists of highly disordered carbon layers and is nongraphitizable, while soft carbon is partially disordered with short-range order and less defects, and can be graphitized at higher temperatures. Therefore, the microstructure (such as defects, edges, pores, functional groups, and graphitization degree) directly affects K\(^+\) storage performances. In addition to the partial intercalation of K\(^+\), surface-driven capacitive K\(^+\) storage is predominated in amorphous carbons. This capacitive process is crucial to facilitate rate capability and cycle performance because the pseudocapacitive behavior mainly takes place at surface or near surface region with fast kinetics and little damage to the bulk electrode. In this context, potassiation/depotassiation curves appear as the sloping characteristics. The notable capacitive contribution can be estimated by cyclic voltammetry (CV) profiles with different scan rates. As can be seen from Figure 3c, the surface-driven capacitive contribution is 71.4% at 0.2 mV s\(^{-1}\). With increasing the scan rate, the surface-driven contribution gradually increases and a value as high as 86.5% can be achieved at 1.2 mV s\(^{-1}\) (Figure 3d). Relatively massive investigations have been conducted to improve the capacitive contribution for pursuing high-rate and ultralong stability. The strategies include: 1) enhancing specific surface area by introducing nanopores or designing various nanostructures, 2) producing tremendous intrinsic carbon layer defects, and 3) incorporating doped heteroatom functional groups. Some representative electrochemical results in terms of capacity, cycle stability, and rate performances are summarized in Figure 3e.

As an example, the construction of carbon materials with developed pore structure is helpful to reduce K\(^+\) diffusion distance and increase the K-storage capacity. For instance,
Li et al. synthesized a series of carbon materials with different pore structures and found that the carbon anode synthesized at 900 °C showed the best performance due to its appropriate surface area (2205 m² g⁻¹), high mesoporous volume (1.07 cm³ g⁻¹) and large number of active sites for K⁺ adsorption. A capacity of 284.8 mAh g⁻¹ was obtained after 200 cycles at 0.1 A g⁻¹. Moreover, defects can be created in carbon materials by adjusting the carbonization temperature. Low temperature induces more abundant defects, which is beneficial to the effective adsorption of K⁺ for capacitive charge storage. For example, polyaniline-co-polypyrrole derived carbon at 500 °C shows a high capacity of 305 mAh g⁻¹ after 660 cycles at 0.2 A g⁻¹. However, defects can be created in carbon materials by adjusting the carbonization temperature. Low temperature induces more abundant defects, which is beneficial to the effective adsorption of K⁺ for capacitive charge storage. For example, polyaniline-co-polypyrrole derived carbon at 500 °C shows a high capacity of 305 mAh g⁻¹ after 660 cycles at 0.2 A g⁻¹. Moreover, defects can be created in carbon materials by adjusting the carbonization temperature. Low temperature induces more abundant defects, which is beneficial to the effective adsorption of K⁺ for capacitive charge storage. For example, polyaniline-co-polypyrrole derived carbon at 500 °C shows a high capacity of 305 mAh g⁻¹ after 660 cycles at 0.2 A g⁻¹. Moreover, defects can be created in carbon materials by adjusting the carbonization temperature. Low temperature induces more abundant defects, which is beneficial to the effective adsorption of K⁺ for capacitive charge storage. For example, polyaniline-co-polypyrrole derived carbon at 500 °C shows a high capacity of 305 mAh g⁻¹ after 660 cycles at 0.2 A g⁻¹. Moreover, defects can be created in carbon materials by adjusting the carbonization temperature. Low temperature induces more abundant defects, which is beneficial to the effective adsorption of K⁺ for capacitive charge storage. For example, polyaniline-co-polypyrrole derived carbon at 500 °C shows a high capacity of 305 mAh g⁻¹ after 660 cycles at 0.2 A g⁻¹. Moreover, defects can be created in carbon materials by adjusting the carbonization temperature. Low temperature induces more abundant defects, which is beneficial to the effective adsorption of K⁺ for capacitive charge storage. For example, polyaniline-co-polypyrrole derived carbon at 500 °C shows a high capacity of 305 mAh g⁻¹ after 660 cycles at 0.2 A g⁻¹. Moreover, defects can be created in carbon materials by adjusting the carbonization temperature. Low temperature induces more abundant defects, which is beneficial to the effective adsorption of K⁺ for capacitive charge storage. For example, polyaniline-co-polypyrrole derived carbon at 500 °C shows a high capacity of 305 mAh g⁻¹ after 660 cycles at 0.2 A g⁻¹. Research on carbon materials with heteroatoms has shown that doping with nitrogen can effectively promote K⁺ adsorption with fast reaction kinetics. Based on this, the material 9.34 at% of edge-N shows impressive K-storage stability of 252 mAh g⁻¹ at 1 A g⁻¹ after 6000 cycles. Although the introduction of heteroatoms can improve the performances, their optimum content and configuration remains elusive. For example, some carbons have a nitrogen content as high as 22.7 at%, but their performances are moderate. Therefore, clarifying the optimized heteroatom content along with their suitable chemical configuration is worthy of further investigation. Besides, incorporation of multiple-element doping is found to work synergistically, such as N/B, N/P, S/N, N/O, S/O, and O/F. Despite the enhancement in rate and cycling performances, these capacitive-dominated carbons generally face the following challenges. First, the ICE is low, normally in the
range of 10–50% (Figure 3f). The large irreversible capacity loss in the first cycle (Figure 3a) is mainly originated from the high specific surface area with the irreversible electrolyte decomposition, instability of formed solid electrolyte interface (SEI) and the irreversible adsorption of ions at defect sites, etc. Recently, the ICE can be raised to 60% by regulating the heteroatom-doped carbon structure.\(^{36,82}\) For example, Alshareef et al. designed a defect-rich, edge-nitrogen doped carbons with a high ICE of 65%.\(^{36}\) In fact, there is no need to pursue too high specific surface area. The material with a specific surface area as high as 2372.5 m\(^2\) g\(^{-1}\) has an ICE of 52.3%.\(^{32}\) Second, the sloping discharge/charge profile from such surface-driven behavior would decrease the average voltage and energy density of the full cell when paired with a cathode. Cathode materials are classified into four categories in PIBs, including Prussian blue and its analogs, layered metal oxides, polyanion compounds, and organic cathode materials.\(^{98–100}\) The redox potential depends on the types of cathode. For instance, KVP\(_2\)O\(_3\) deliver higher potential up to 4.2 V.\(^{101}\) When using highly porous carbons as cathode materials, potassium ion hybrid capacitors can be developed,\(^{102–105}\) which exhibit lower energy density but higher power density than PIBs. Finally, unsatisfactory volumetric capacity is another problem of amorphous carbon material, which is due to the lower packing density with massive void space (such as hollow carbon of 0.23 g cm\(^{-3}\)) and activated carbon frequently below 0.5 g cm\(^{-3}\)).\(^{106,107}\) as compared to that of graphite (e.g., 2.3 g cm\(^{-3}\)).

3. Design of Carbon Materials with Intercalation/ Capacitive Hybrid Storage Mechanisms

Rational balancing of surface-driven and intercalation mechanism is desirable to incorporate the merits of high ICE, low-potential plateau, remarkable cyclability, and rate capability (Figure 4). Currently, there are several strategies to construct carbon structure with balanced intercalation/capacitive processes to enhance these performances. The key issue is to rationally control the crystalline graphitic structure and disordered microcrystallite nanodomains within the carbons. Various approaches have been adopted including 1) adjusting carbonization temperature,\(^{108,109}\) 2) compositing different precursors with distinct properties,\(^{110–112}\) and 3) constructing pore and micro/nanostructures in graphitic materials.\(^{23,113–116}\)

The pyrolysis temperature plays a key role in determining the carbon microstructure. When using polycrylonitrile as the precursor, it was found that the resulting carbon materials transitioned from disordered structure, partially ordered structure, finally ordered structure when increasing the carbonization temperature from 650, 1250, to 2800 °C (Figure 5a).\(^{108}\) The variation of structure leads to distinct K\(^+\) storage mechanism, therefore reflecting different discharging/charging behaviors. Compared with disordered carbon (treated at 600 °C) and ordered graphitic carbon (treated at 2800 °C), partially ordered carbon at the mediate carbonization temperature of 1250 °C exhibited intercalation-induced faradaic reaction and surface capacitive behavior (Figure 5b), thus delivering an optimized capacity of 180 mAh g\(^{-1}\) at 1 A g\(^{-1}\). This strategy is also applicable to soft carbon materials. Liu et al. obtained pitch-derived soft carbon with different structures through a simple thermal control and found that the structure of materials changes from nearly amorphous to long-range ordered structures with the increase of temperature.\(^{109}\) And the materials with partially disordered specics managed to achieve a sloping/plateau profile, suggesting a high-energy-density anode material.\(^{109}\) Apart from carbonization temperature, direct mixing of distinct precursors, which are responsible for the formation of hard, soft or graphitic carbons, is another promising strategy to balance the sloping/plateau charge storage behavior. For example, by mixing graphite with pitch precursor in the desired mass ratio of 3:1, graphite-soft carbon composites with optimized performances were synthesized.\(^{111}\) In this respect, the material not only has the intercalation function of graphite structure, but also could inhibit the unstable SEI formation and provide structural protection for graphite layers with the existence of soft carbon structure. Thus, a high ICE of 67.3%, reversible capacities of 280.2 mAh g\(^{-1}\) and a plateau-dominated profile were obtained with the graphite-soft carbon composite.\(^{111}\) Meanwhile, compositing graphite with the hard carbon is also effective. By coating amorphous N-doped carbon nanosheets on multilayer graphite (random orientations of the flake graphene), the resulting composite exhibits a flat discharge plateau from graphite and enhanced cyclic stability (215.7 mAh g\(^{-1}\) after 1000 cycles at 0.2 A g\(^{-1}\)) and ICE (from uncoated graphene of 43.43% to 61.83%). This is due to the improved K diffusion coefficient and volume buffer ability.\(^{112}\)

Moreover, by using metal specie-containing precursor and special carbonization, graphitic structure can be designed with few layers graphene microspheres. Lu et al. reported a sulfur-assisted method (Figure 6a) that changed benzene rings of tetraphenyltin into 3D few layer graphene microspheres (FLGMs, Figure 6b).\(^{113}\) A dominated low-potential plateau curve was observed, and an ICE of 94% was achieved assisted by prepotassiation (Figure 6c). Unlike planar graphene and graphite, FLGMs was not layer-by-layer stacked compactly and thus was more suitable for facilitating intercalation kinetics and fast transport for K\(^+\), and exhibited an excellent cycling stability with capacity around 230 mAh g\(^{-1}\) after 1000 cycles at 200 mA g\(^{-1}\) (Figure 6d).\(^{113}\) Similarly, constructing nanostructure/pores/defect into graphitic carbons is beneficial for introducing certain surface-controlled capacitive charge storage to solve the sluggish kinetics and poor cyclability involved in K\(^+\) intercalation. As an example, Qian et al. utilized a hydrothermal treatment to produce a graphitic carbon with nanospring structure.\(^{114}\) The participation of water for the formation of nanospring structure is important to achieve excellent K-storage performances (Figure 7a,b). This not only induces a great amount of edge-plane active sites but also generates the mesoporous structure, which together make the material remain intact after potassiation compared with the graphite (Figure 7a). Such combination allows the obtained nanospring structure to integrate the intercalation-controlled and surface-driven K\(^+\) storage behavior, producing a sloping/plateau discharging characteristics (Figure 7c). Meanwhile, the cyclability was striking with an ultralong life span of 99.9 mAh g\(^{-1}\) after 10000 cycles at 2000 mA g\(^{-1}\) (Figure 7d). Dou et al. used potassium hydroxide as etching agent to adjust the graphite structure through high temperature annealing. Compared to the untreated graphite, the material has a larger layer.
spacing and forms a lot of carbon nanosheets, leading to rapid transport of \( K^+ \) and high reversible capacity of 100 mAh g\(^{-1}\) after 100 cycles at 0.2 A g\(^{-1}\).[115] Likewise, graphitic carbon with engineered defects can help to facilitate fast kinetics and obtain refined discharging–charging behavior.[23] It is worth noting that with excessive pore-making, cyclability and rate capability could be improved, but at the expense of adsorption-dominated sloping profile and packing density.[64] Evidently, designing special micro/nanostructures into graphitic carbons is essential for ensuring the low-potential plateau, high rate and superior cyclability, and even higher ICE.

4. Carbon Structure with Electrolyte Adaption

For carbon anode materials, electrolyte adaption is also important for enhancing the electrode stability and thus \( K^+ \) storage performance. According to the carbon structures with different \( K^+ \) storage mechanisms, several criteria should be considered for the electrolyte selection, including the solute, solvent, and concentration. For those intercalation-controlled graphitic carbons, large volume variation usually occurs during the intercalation/deintercalation process.[11,15] Thus, to mitigate the cracking of SEI under stress and impede further passivation and excessive side reactions, the selected electrolyte should help to build mechanically robust SEI layers or to reduce the volume variation.[117,118] The first is the selection of \( K^+ \) salt in electrolyte. It is reported that potassium bis(fluorosulfonyl)imide (KFSI)-based electrolyte can lead to more inorganic-rich SEI originating from the decomposition of FSI\(^{-}\) anions, which is more robust than KPF\(_6\)-derived organics/inorganics blended SEI.[119] The second is the choice of solvent, and it is demonstrated that the use of ether-based solvent can enable the co-intercalation of \( K^+ \) along with solvent molecules into graphitic layers, different from ester-based electrolyte.[118,120,121] Such co-intercalation leads to low volume variation and high \( K^+ \) diffusion coefficient, and therefore is responsible for high-rate and long cyclability performances. For example, Chou et al. studied the electrochemical performance of graphite in diethylene glycol dimethyl ether (DEGDME), and found that diffusion kinetics of \( K^+ \)-DEGDME

Figure 4. Schematic illustration of balancing the intercalation-controlled and surface-driven capacitive \( K^+ \) storage with their respective strengths and weaknesses.

Figure 5. a) Correlation between microstructure and annealing temperature of carbon materials, b) \( \frac{dQ}{dv} \) curves of CNF films at the oxidation. The inset shows the illustrations of main \( K^+ \) storage mechanism in different regions. Reproduced with permission.[108] Copyright 2019, Elsevier.
complex (about $10^{-9}$ cm$^2$ s$^{-1}$) is faster than K$^+$ (about $10^{-11}$ cm$^2$ s$^{-1}$). Therefore, the battery exhibited higher rate performance (77.8 mAh g$^{-1}$ at 10 Ag$^{-1}$) and cycling stability (capacity retention of 88.5% after 100 cycles). Besides, a superior ICE of 62.9% was achieved.\[123\] The third consideration is electrolyte concentration. Increasing the salt concentration can facilitate the decomposition of anion in salt, thus forming more inorganic-containing SEI with robust and dense structure.\[122,123\] Fan et al. revealed that an inorganic-rich SEI film was formed in the graphite electrode with an electrolyte of KFSI/ethyl methyl carbonate (molar ratio of 1: 2.5), and thus excellent cycling stability over 17 months was achieved.\[122\] Likewise, design of the localized high-concentration electrolyte is also beneficial to the formation of more inorganic-rich SEI layer.\[123\]

For surface-driven amorphous carbons, the capacitive K$^+$ storage induces little volume variation during cycling. Consequently, there is relatively less requirement on the electrolyte selection. The benchmark electrolyte is KPF$_6$ in carbonate solvent,\[30,36,39\] benefiting from the well-established knowledge of lithium-ion battery studies. Considering the existence of disordered graphitic microcrystallite in hard/soft carbons, partial intercalation/deintercalation behavior also presents in surface-driven amorphous carbons, and thus the electrolyte adaption follows similar rules. For example, Lu et al. reported

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**Figure 6.** a) Schematic illustration for the preparation of few layered graphene microspheres composite (FLGMs). b) TEM image of FLGM. c) The first discharge/charge curves of FLGMs electrode. d) Cycling performance of FLGM, commercial graphene, and graphite at a current density of 200 mA g$^{-1}$. Reproduced with permission.\[113\] Copyright 2021, RSC.

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**Figure 7.** a) Schematic illustration of potassiation process of G (graphite, left) and OGCS (high-oriented mesoporous graphitic carbon nanospring, right) electrodes. b) HRTEM images of OGCS. c) The first discharge/charge curves of G, GC (graphitic carbon), and OGCS at 50 mAg$^{-1}$. d) Cycling performance at a high current rate of 2000 mAg$^{-1}$. Reproduced with permission.\[114\] Copyright 2019, Wiley-VCH.
that O-doped carbon anodes delivered long cycle performance (capacity retention of 91.5% after 3000 cycles at 0.5 A g\(^{-1}\)) in high concentration electrolyte of 3 M KFSI in 1,2-dimethoxyethane.\[126\] Despite the striking advantages of KFSI and ether-based electrolytes, there also exist some adverse effects. For example, FSI\(^-\) anion-based electrolytes could corrode Al foil during anodic polarization,\[123\] high salt concentration with denser inorganic-rich SEI probably suppress the intercalation in ether-based solvent,\[123,126\] and ether-based solvent is not tolerant to high-voltage cathode. Therefore, judicious electrolyte selection is necessary in consideration of not only performances but also carbon structures with different K storage mechanisms.

5. Areal Capacity and Cell Components

For the areal capacity, it is related to the carbon mass loadings in the electrode. Considering the areal capacity of 4 mAh cm\(^{-2}\) (the value of commercially available LIBs) and the anode capacity is 300 mAh g\(^{-1}\) for carbon in PIBs, a practical carbon anode mass loading of 13.4 mg cm\(^{-2}\) is required. However, we found that amorphous carbons with surface-driven capacitive storage generally show low mass loading less than 1.5 mg cm\(^{-2}\), corresponding to low areal capacity less than 0.4 mAh cm\(^{-2}\), which is far from practical requirements. This might be due to the low tap density with massive void space, which is also unfavorable for volumetric capacity. Recently, Lu et al. elevated mass loading of carbons with hollow architecture to produce high areal capacity.\[86\] The areal capacity of 0.47 mAh cm\(^{-2}\) is obtained when the mass loading is 5.65 mg cm\(^{-2}\) at the current density of 0.1 A g\(^{-1}\). While for intercalation-dominated graphitic carbons, the majority of literatures also adopt a low mass loading for better electrolyte infiltration. However, there are a few studies strived to increase the mass loading with areal capacity close to or even higher than commercialization requirement. For example, Fan et al. compared cycling performance of graphite electrode with different mass loadings.\[122\] As expected, with increasing mass loading to 28.56 mg cm\(^{-2}\), the battery delivered an areal capacity of 7.36 mAh cm\(^{-2}\). As for lab-level coin-cell assembling, excess of electrolyte dosage and large volume glass fibers separators are often used for evaluation, which are also unfavorable for large-scale applications. There are several reports on the investigation of full cells paired with the cathode, and the average discharge potential is in the range of 1.1–3.5 V.\[99\]

6. Conclusion

PIBs have recently attracted much attention due to resource-abundance of potassium, the low standard redox potential and high conductivity of K\(^+\)-based electrolyte. Particularly, considering the cost-effectiveness and environmental-friendliness, carbon materials stand out as the most promising candidates for PIBs anode. Carbon materials are available in various forms with different compositions and microstructures, which have a crucial influence on the performance of potassium storage. As for graphite materials, their K-storage originates from K\(^+\) intercalation in the graphite layer. However, diffusion of K\(^+\) into graphite layers is sluggish and leads to high volume expansion coupled with collapse of the graphitic structure, thus causing insufficient rate capability and poor cyclic stability. While amorphous porous carbons with expanded interlayer and tremendous defects/nanovoids can improved capacity, rate capability, and cyclability via surface-driven capacitive K storage. However, the low ICE, high-potential sloping curves and low material density are problematic for a practical application. Therefore, well balancing of both mechanisms is necessary, and further investigation should consider intercalation-dominated graphitic carbons with suitable micro/nanostructures and high tap density, which can deliver high ICE, low-potential plateau, enhanced cyclability and high energy density. Carbon model materials with well-defined pore size, doping, or microstructure may play an important role to achieve a better understanding of K-storage mechanisms. The compatibility of electrolyte with different carbon structures should be also considered. Other critical parameters and full cell considerations are also of importance. Besides, the development of novel analytical techniques has a great future for better understanding of SEI formation, K\(^+\) storage mechanism, specific electroadsorption sites, K\(^+\) mobility and anode degradation mechanisms. With the guidelines for rational design of these critical parameters, the K\(^+\)-based energy storage technology can be further enlightened and fostered into practical applications within decades.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

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