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Energy Storage Mechanisms and Recent Advances in Iodine-Modified Carbon-Based Supercapacitors

Yuluo Qin

School of Chemistry, Sun Yat-sen University, Guangzhou, China
qinyluo@mail2.sysu.edu.cn

Abstract. Porous carbon materials are an important series of materials for the electric double layer supercapacitors (EDLCs). However, they have a limited energy storage performance due to non-Faradaic charge storage. The incorporation of iodine in the porous carbon structure offers an interesting pathway to bring both electric double-layer capacitance and extra Faradaic charge storage. The review details three key mechanisms in iodine modified carbon-based supercapacitors, namely: iodine redox reaction to enhance the charge storage capacity, interfacial interaction between iodine and carbon to control electronic transport to the carbon surface and the retention of iodine by nanoscale confinement to stabilize iodine species, and to inhibit the migration of polyiodide. It is proposed that the iodine redox chemistry and iodine-assisted pore-structure optimization primarily improve the charge-storage ability and the interfacial regulation could improve the rate capability and iodine utilization. The suppression of the migration of polyiodide and the improvement of cycling stability is especially effective at the nanoscale, because of the nanoscale confinement and interfacial stabilization. In conclusion, the ability to optimize the iodine utilisation and pore accessibility and reduce the polyiodide migration is the key to high-performance iodine-based supercapacitor. The present review offers a guideline to assess these features based on the criteria of energy density and long cycle life.

Keywords: supercapacitors, porous carbon materials, iodine modification, pseudocapacitance, nanoscale confinement

1. Introduction

Supercapacitors have attracted a great deal of research interest as energy storage devices because of their high-power density, fast charge-discharge rates and long cycling stability. Among the different types of electrode materials utilized in such devices, porous carbons are of particular interest because they possess high accessible surface area, high electrical conductivity and high chemical stability [1]. In traditional EDLCs, the storage of charge is mainly due to reversible, electrostatic adsorption of electrolyte ions at the interface between the electrode and the electrolyte. This process of charge transfer is however inherently non-Faradaic, meaning that the charge capacity to be stored is limited and so is the energy density to be achieved [2, 3].

The addition of pseudocapacitive reactions is a useful way to overcome the intrinsic drawbacks of charge storing in an electric double layer and to enhance the energy storing capability of carbon

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based supercapacitors [4]. Pseudocapacitive materials are metal oxides, conducting polymers and redox active species [5-8]. Of them, iodine is appealing because of its easily redox reaction and fast reaction rate that can offer extra Faradaic charge storage when combined with porous carbons [9-11]. However, the previous works have largely targeted individual iodine-related pathways like redox reactions, iodine-carbon interactions or iodine nanoscale confinement without systematic comparisons on their respective roles in the achievement of charge-storage, the rate capability and the cycling stability. Furthermore, differences in iodine loading, mass-normalization approaches, electrode architectures and iodine retention make it difficult to make a direct comparison of the performance reported. Therefore, there is a need to categorize and compare such iodine related mechanisms and challenges for evaluation systematically.

First, this review discusses the fundamental energy storage mechanism and limitation of performance of porous carbon-based supercapacitors. The literature mostly deals with aqueous redox systems with iodide; studies related to iodine loaded porous carbon or iodine pre-adsorbed porous carbon, studies of iodine doped carbon materials, and studies dealing with iodine-assisted pore-structure engineering are also discussed to broaden the picture of the roles of iodine in carbon-based energy storage. The studies reviewed are classified according to the main purpose of iodine modification which includes the enhancement of charge-storage capacity, the improvement of the rate capability and the regulation of iodine-species migration and cycling stability. Iodine redox reactions, iodine-carbon interfacial interactions and nanoscale confinement are further discussed to explain the underlying mechanisms. Finally, the synergistic regulation strategies of porous-carbon structure and interfacial properties focusing on the balance of iodine loading, pore accessibility, electronic transport and iodine retention are discussed.

2. Fundamentals of porous carbon-based supercapacitors

2.1. Electric double-layer energy storage

The electrolyte ions reorder at the electrode/electrolyte interface creating an electric double layer (EDL) when a potential is applied. The charge storage mechanism of porous carbon electrodes is largely based on non-Faradaic electrostatic adsorption and desorption of electrolyte ions resulting in fast charge/discharge, high power density and excellent cycling stability. But the attainable storage of charge is significantly affected by accessible interfacial area and accumulation of ions. In practice, porous carbon electrodes, and EDL formation and ion transport, can be influenced further by ion solvation, pore-size matching, surface functional groups and electrolyte composition especially in the nanoscale-confined regime [12, 13]. Unlike ideal models of EDL with planar interfaces, in real porous carbon electrodes, the impact of ion size, diffusion, specific adsorption and interactions with pore walls on the spatial distribution of ions in the confined pores cause the behavior to deviate [13]. Understanding of such interfacial effects is of special importance in understanding the ion storage and the subsequent redox behavior of iodine species in the iodine modified porous carbon electrodes.

2.2. Structural characteristics and energy storage of carbon-based electrode materials

The accessible surface area and pore-size distribution of the porous carbons have been shown to significantly affect their electrochemical properties, and they are usually adjusted by carbonization and activation processes [14-16]. A high specific surface area provides a high density of sites for interfacial adsorption of ions, and is not alone sufficient for charge-storage performance. Ion

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transport and accessibility of the pore network and therefore the formation and response of the electric double layer is also controlled by the pore size and connectivity.

Furthermore, recent investigations of nanoconfined carbon materials have indicated that there is a strong dependence of energy-storage behavior on the relation between pore size and the size of the electrolyte ions. When the pores are comparable to the effective size of the solvated ions, the desolvation and rearrangement of ions around the pores may be the case depending on the pore size, electrolyte composition and ion-pore-wall interactions [17, 18]. These effects may result in a different charge distribution at the interface and accumulation of ions, and thus electrochemical responses different from those of the macroscopic planar interfaces.

2.3. Performance limitations of EDLCs and the need for pseudocapacitive enhancement

Porous carbon materials have good electrical conductivity, hierarchical pore structures and chemical stability and have been widely used in electric double layer supercapacitor (EDLC). The structural and physicochemical properties of carbon based EDLCs can enable them to have a high-power density, a high charge-discharge rate and a high cycling stability. However, in EDLCs, the charge storage is predominantly due to reversible adsorption and desorption of ions in the electrolyte and, consequently, the charge-storage capacity of such devices is inherently limited by the available interfacial area and the amount of adsorption of ions.

The optimal EDLCs have quasi-rectangular CV curves and near linear GCD curves, which means that the charge is primarily stored by reversible ion adsorption rather than Faradaic reactions (Figure 1). This non-Faradaic mechanism provides for very good rate capability and cycling stability; however, the charge amount that can be stored is restricted and as a result the energy density of purely carbon-based supercapacitors is limited. This is a drawback that restricts their use in areas where continuous energy supply is required [19].

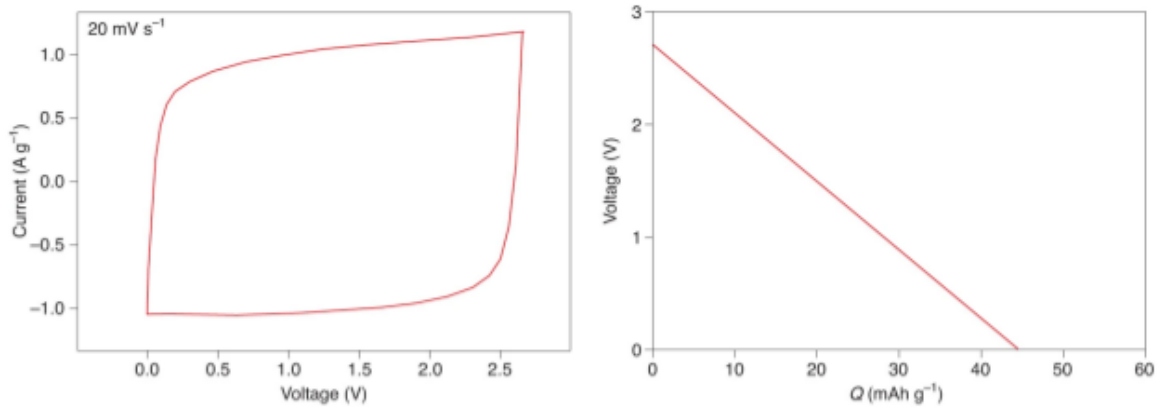

Figure 1. Typical CV and galvanostatic charge/discharge curves of an EDLC, adapted from [18]

There are trade-offs between charge-storage capability and reaction kinetics in electrochemical storage mechanisms as depicted in Figure 2. Pseudocapacitance is a promising compromise between rapid and reversible Faradaic charge storage, and relatively high charge transfer kinetics. Among redox materials, Iodine is particularly attractive because of its very flexible redox chemistry and relatively rapid reaction kinetics that enable the delivery of an extra Faradaic charge storage in conjunction with the porous carbon structure.

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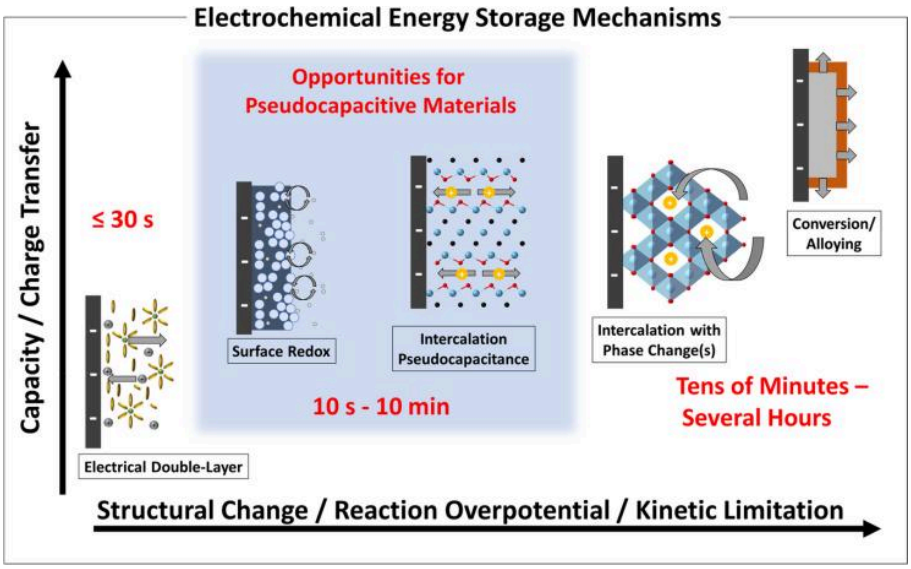

Figure 2. Classification of electrochemical energy storage mechanisms as a function of their characteristic capacity and the degree of structural changes of the electrodes, reaction overpotential, and kinetic limitations, adapted from [4]

3. Performance enhancement and recent advances in iodine-modified porous carbon materials

3.1. Iodine redox chemistry and iodine-assisted pore engineering

Iodine can enhance the performance of carbon-based supercapacitors in two fundamentally different ways: direct Faradaic charge storage due to iodine redox chemistry and indirectly by engineering the porous carbon structure in the presence of iodine. It is to be emphasized that these two mechanisms are different in that the charge storage in former is realized by the reversible electrochemical reactions of the iodine species, the charge storage in the latter is mainly realized by changing the structure and accessibility of the carbon electrode.

The reversible iodine redox reactions can add Faradaic charge to the charge storage in porous carbon electrodes by electric double-layer. Lota and Frąckowiak [10] investigated the carbon/iodide interface in 1 mol L aqueous KI electrolyte, and reported an electrode-level capacitance > 1840 F g^{-1} for the positive electrode in a three-electrode configuration in a small potential window. The value should not be compared directly with values calculated from capacitance of complete devices with two electrodes due to variations in electrode geometry and mass-normalization basis. The same carbon/iodide system in two-electrode configuration had a capacitance of 125 F g at a current density of 50 A g^{-1}, and was found to have stable electrochemical performance over 10000 cycles. It is found that under suitable electrochemical conditions, the iodide redox chemistry can store much more charge, however the apparent capacitance is highly dependent on the testing set-up and the way the capacitance is calculated.

Figure 3 indicates the Pourbaix diagram and indicates that the redox properties of species of iodine are greatly influenced by the electrode potential and electrolyte pH. I can be reversibly oxidized to molecular iodine in aqueous iodide electrolytes which can then equilibrate with excess I to form I. Other iodine species can be stable depending on the electrochemical environment [9]. Thus, the charge storage based on iodine is not determined by a universal reaction pathway but is dependent on the potential and the electrolyte conditions. In the aqueous iodide systems dealt with

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in this review the redox process may be broken down into a series of oxidation of I and equilibria between I and polyiodide species.

$$3I^- \rightleftharpoons I_3^- + 2e^- \quad (1)$$

$$2I_3^- \rightleftharpoons 3I_2 + 2e^- \quad (2)$$

$$5I^- \rightleftharpoons I_5^- + 4e^- \quad (3)$$

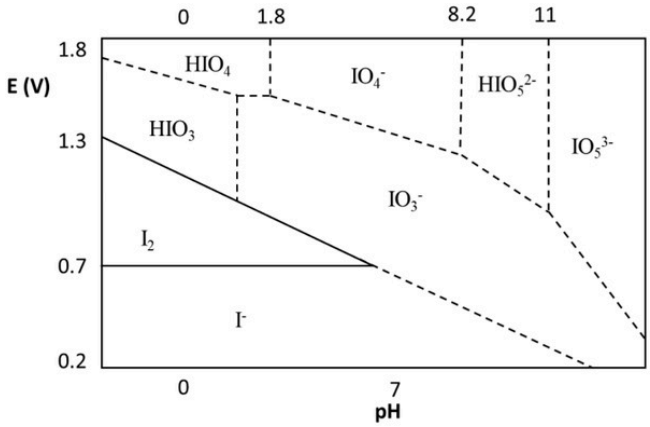

Figure 3. Pourbaix diagram showing chemical states of iodine species, adapted from [20]

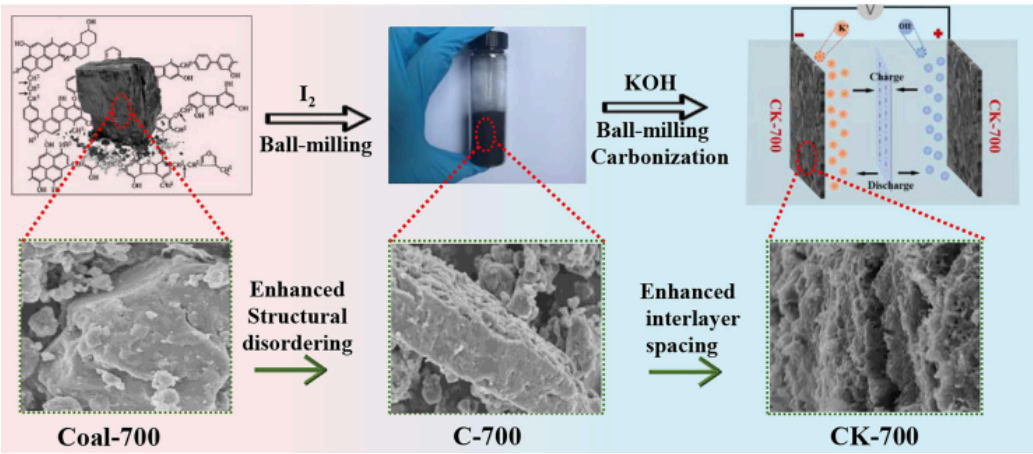

Figure 4. Schematic illustration of the iodine-intercalation-assisted synthesis of coal-based activated carbon, adapted from [21]

In addition to charge storage by iodine redox, iodine also has the possibility to indirectly enhance the electrochemical properties of porous carbon by structural engineering. In the case of porous carbon prepared from coal, Pei et al. [21] used iodine intercalation in the preparation of porous carbon from coal to increase the interlayer spacing of the carbon precursor, which is favorable for more homogeneous KOH activation and the formation of an accessible pore network (Figure 4). The prepared CK-700 electrode showed a specific capacitance of 350 F g^{-1} at 0.5 A g^{-1} in 6 M KOH and 91.7% capacitance retention after 30000 cycles at 10 A g^{-1}. The enhancement of the performance was attributed primarily to the enhanced interlayer spacing of the precursor and the greater accessibility of the electrolyte and the desirable pore-size distribution achieved by the iodine-

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assisted activation. Instead of iodine species directly involved in the Faradaic charge storage in the iodine/iodide redox chemistry, iodine in this strategy mainly functions as an intercalation and activation facilitator to enhance the structural properties of the carbon electrode.

Therefore, the charge-storage performance enhancement from iodine can be direct involving iodine redox chemistry, or indirect involving iodine-assisted structural engineering of the porous carbon framework. The former can enhance the amount of charge stored, and the latter can regulate the porous structure and enhance the accessibility of ions and the transport. It is crucial to differentiate between these two pathways, then, when comparing the electrochemical performance of iodine modified carbon electrodes.

3.2. Electronic-structure modulation by iodine modification

The interaction of iodine species with the carbon framework can result in changes of electronic structure and transport properties of the carbon materials. A study by Zubair et al. [22] investigated the iodine-doped double-walled carbon nanotubes (DWCNTs) using Raman spectroscopy, transmission electron microscopy (TEM), X-ray diffraction (XRD) and first-principles calculations based on density functional theory (DFT). They found that the iodine to carbon-framework charge redistribution is consistent with an average transfer of 0.029e per carbon atom and an average shift of ~1 eV of the Fermi level. The average number of conductive channels also increased from 4/3 in pristine DWCNTs to 26/3 with iodine doping, which means that the electronic transport was improved. The doped nanotubes also exhibited a significant G-band shift and a significant decrease in relative resistivity. These experimental and theoretical results overall suggest the presence of iodine–carbon interaction to tune electronic properties of carbon materials (Figure 5). Similar charge transfer phenomenon triggered by iodine has also been observed in iodine-doped graphene [23, 24] and iodine-loaded porous carbon systems [10, 11].

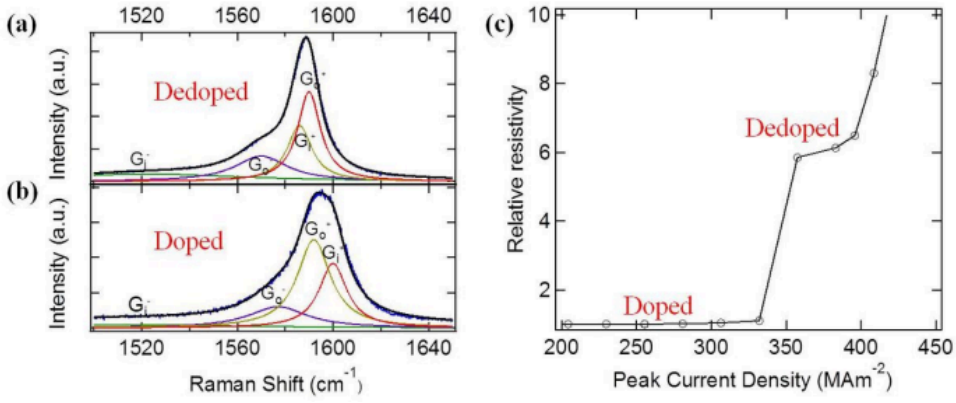

Figure 5. Effect of iodine doping on the electronic structure and electrical conductivity of double-walled carbon nanotube fibers, adapted from [22]. (a) Raman spectrum of the fiber after iodine removal; (b) Raman spectrum of the iodine-doped fiber, showing a shift in the G-band peak, indicating charge transfer between iodine and the carbon framework; (c) Variation in the relative resistivity of the fibers at different current densities, with the iodine-doped sample exhibiting lower resistivity and higher charge-carrying capability

This charge redistribution can lead to a change of the electronic states of the carbon material, and can help the carrier transport by the shift of the Fermi level and the change of the electronic states near the Fermi level [22-24]. In addition to direct doping of iodine, other than that, the surface

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chemical environment of porous carbon can also control the interaction between the carbon framework and iodine species. In particular, nitrogen doping can generate electron-rich sites to interact with the iodine species and allow tuning the redox behavior of the interfaces [9, 11]. This surface-chemical regulation is an alternative way to improve the iodine retention and the interfacial charge transfer.

3.3. Regulation of iodine-species migration and cycling stability

The main reason for the improvement in the cycling stability of the iodine modified is due to the reduction of migration and loss of the active iodine species. Soluble polyiodide ions can be formed in the charge and discharge processes in conventional iodine-based systems. The transfer of polyiodide species between the electrodes which take part in parasitic redox reactions is termed as the shuttle effect which causes self-discharge, loss of active material and capacity fading [10]. Thus, it is important to make sure that the active iodine species is stabilized with enough iodine content to make it stable for long-term cycling.

The iodine storage in nanoporous carbon was investigated using operando Raman spectroscopy and operando SAXS/WAXS by Prehal et al. [9]. They found that, under the conditions they studied, oxidized iodine species were primarily reversibly deposited as a solid-state iodine within nanopores, and not as soluble polyiodides. The results show that the nanoscale confinement can change the iodine storage pathway, and to suppress the formation and migration of soluble polyiodide species, alleviating the shuttle effect and self-discharge (Figure 6).

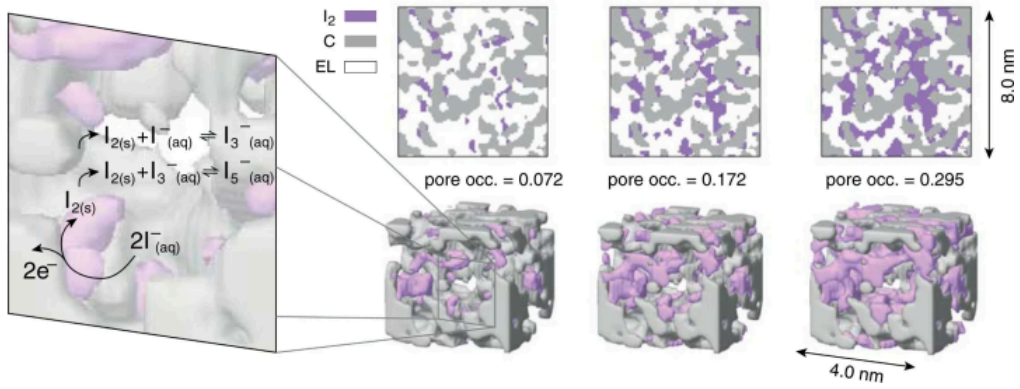

Figure 6. Schematic illustration of the confined deposition model of iodine within nanopores and the iodine redox reaction mechanism, adapted from [9]. Three-dimensional distribution models of solid-state iodine within carbon nanopores at different iodine pore-filling fractions, together with the iodine redox reaction pathway proposed based on operando Raman spectroscopy and operando SAXS/WAXS, reveal the role of nanoscale confinement in promoting the reversible deposition of solid-state iodine, suppressing polyiodide dissolution, and regulating the energy-storage mechanism

In this strategy, Larasati et al. [11] suggested a strategy called "pre-adsorbed iodine" (PAI) in which iodine is uniformly distributed in the nanopores in advance, which can effectively reduce the loss of the active species during charge/discharge cycle and improve the iodine utilization and cycling stability of the device. Furthermore, some strategies have also been developed to further inhibit the migration of iodine species and offer new ideas to design highly stable iodine-based supercapacitors including the regulation of the micropore size, enhancement of the interaction

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between iodine and carbon framework, and construction of hierarchical confinement structures [9, 11, 24].

Besides the pore structure control, surface-chemical modification of particles can also stabilize the iodine species and adjust the interfacial redox behavior of iodine. With regard to aqueous iodide redox supercapacitor, Wang et al. [25] prepared N doped porous carbon nanosheets and discovered that the redox capability was enhanced as the amount of graphitic nitrogen increased. The optimized NC-900 electrode had a capacitance of 251 F g^{-1}, an energy density of 22.4 Wh kg^{-1} and retained 86.5% of the initial capacitance after 30,000 cycles at a voltage window of 1.6 V. The experimental and theoretical analyses revealed that the electron-donating function and adsorption capability of the iodine species on the graphitic N sites is responsible for the enhanced redox performance, implying that the surface-chemical regulation can enhance the iodine-carbon interactions, and thus the charge storage and cycling stability.

All the studies reviewed in Table 1 collectively indicate that various iodine related strategies have a preference for various performance needs. The iodine redox chemistry is mostly advantageous to the charge-storage capability, the iodine-assisted structural engineering provides advantages to pore accessibility and ion transport, and the regulation of the electronic structure and surface chemistry is advantageous to charge transport and iodine-carbon interaction. To suppress the migration of polyiodide and to improve the cycling stability, nanoscale confinement and interfacial stabilization are crucial. However, the reported electrochemical metrics cannot be directly compared across different studies due to large differences in the electrolyte composition, electrode configuration, voltage window and mass normalization basis, which can have a significant effect on the measured performance.

Table 1. Shows a comparison of representative Iodine related strategies, electrochemical performance and evaluation conditions on carbon-based supercapacitors

References	Iodine-relatedstrategy	Mainfunction	Electrolyte	Key performance	Mainevidence
[9]	Nanopore confinement	Polyiodide suppression /cycling stability	Aqueous iodide system	~0.81 nm pores; solid iodine occupancy up to ~30%	Confinement promotes solid iodine deposition and suppresses polyiodide migration
[10]	Iodide redox chemistry	Additional Faradai ccharge storage	1 mol L^{-1} KI	>1840 Fg^{-1};125 Fg^{-1} at 50 Ag^{-1}; >10,000 cycles	Iodide redox provides additional charge storage
[21]	Iodine intercalation-assisted activation	Porous-structure optimization	6 M KOH	350 Fg^{-1}; 91.7% after 30,000 cycles	Enlarged interlayer spacing and improved pore structure

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Table 1. (continued)

[22]	Iodine doping	Electronic-structure modulation	—	Fermi-level shift;charge transfer of0.029e/C	Iodine-induced charge transfer and enhanced electronic transport
[25]	N-doping/surface chemical regulation	Iodine–carbon interfacial interaction	Aqueous iodide	251 Fg^{-1}; 22.4 Wh kg^{-1};1.6 V	Graphitic N correlates with redox performance

4. Conclusion

Comparative representative studies demonstrate that iodine related strategies enhance the carbon-based supercapacitors in different and complementary ways, namely the iodide redox chemistry mainly enhances the charge storage capacity, while the pore confinement or the interfacial regulation mainly enhances the iodine retention and the cycling stability. But the performance indicators reported cannot be directly compared due to different iodine loadings, mass-normalization methods, electrode layouts and electrolytes. Thus, the problems are: high iodine utilization, good pore accessibility and effective suppression of polyiodide migration to be realized simultaneously.

Three priorities for future research should be identified. First, a standardised device level assessment should be used, and iodine loading, mass normalisation and two-electrode set-ups should be carefully reported. Second, the correlation between the iodine retention and the cycling stability should be quantitatively assessed through self-discharge, polyiodide crossover and iodine lost during long term cycling. Third, systematic studies are necessary to elucidate the correlation between pore size, iodine species and electrochemical performance under similar conditions. The efforts will lead to more reliable comparisons of iodine-based supercapacitors and will help to rationally design more higher-energy-density, lower self-discharge and longer cycle life supercapacitors.

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