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# *Regulation of Oxygen-Containing Functional Groups in Biochar and Optimization of Supercapacitor Performance via Heteroatom Doping*

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***Abstract.*** The development of the digital industry and the growing embrace of green development have driven the advancement of high-performance, low-cost green batteries. As an electrode material, biochar boasts diverse raw sources, excellent stability and low cost, endowing it with significant application value in both electric double-layer and pseudocapacitive energy storage systems. Oxygen-containing functional groups in biochar serve as a key factor in tuning the chemical properties, safety and cycling stability of capacitors. Their content, type and distribution directly determine the performance of the devices. This paper systematically reviews the origin, regulation strategies and structure-activity relationships of oxygen-containing functional groups in biochar, with a focus on the effects of inherent feedstock components, pyrolysis conditions and oxidative modification on such groups. It clarifies the dual regulation and critical points of oxygen-containing functional groups in capacitors. By comparing the impacts of various oxygen-containing functional groups (OFGs), the mechanism and contradictory effects governing capacitor performance are revealed. The conflict between capacitance enhancement and compromised cycling stability is addressed through a B/N/P ternary co-doping strategy, aiming to explore future development pathways and achieve a balanced progress toward high capacity, high efficiency and long cycle life.This work systematically summarizes the origin, regulatory mechanisms and dual-regulation behavior of oxygen-containing functional groups, and outlines future development directions consistent with the green development philosophy. It holds important research significance for realizing low-cost, high-performance energy storage.

***Keywords:*** Biochar, supercapacitor, oxygen-containing functional group, composite doping method, specific capacitance

## 1. Introduction

With the rapid evolution of information technology and the ongoing energy revolution, the global demand for green, sustainable energy keeps growing. A study covering 285 cities in China shows that urban digital transformation has pushed up electricity consumption by 7% to 20% [1]. As power use rises and the shift to green energy accelerates, there's an urgent need for supercapacitors with top-tier performance.

Supercapacitors fall roughly into two categories: double-layer capacitors and hybrid pseudocapacitors. Both of them enjoy outstanding performance, like exceptional stability, long cycle life, and high charge-discharge efficiency [2]. As a carbon material, biochar which has a huge specific surface area, excellent electrial conductivity, and low cost. It is often used as an electrode material to improve energy density and lifespan. Yet there are many problems exiting: while many studies have explored how oxygened functional groups affect electrode properties, quantifying the role of each individual group and achieving synergistic performance gains are still challenging for me. After all, only focusing on one group alone can't reflect the real situation.

Only by having a systematic research on how multiple groups work together and resolve the contradiction between efficiency and the number of cycles, can we design and develop electrode materials effectively which have better performance .

By analyzing the influence and mechanism of oxygen-containing functional groups on the performance of capacitors. This paper will propose more ideas for enhancing the performance of the electrodes and resolving the contradiction between performance and cycle efficiency.Additionally,it can provide theoretical and technical support for future research.

## 2. Source of oxygen-containing functional groups

Biochar is a porous carbon material produced under limited oxygen or anoxic conditions, using methods such as high-temperature pyrolysis, hydrothermal carbonization, and gasification [3]. The oxygened functional groups in biochar primarily originate from the material itself, the pyrolysis process, and intentional introduction. Performance optimization is often achieved through the regulation of these three aspects

### 2.1. Inherent oxygen-containing functional groups of raw materials

Biomass is composed of a large amount of cellulose, hemicellulose, lignin, etc. The inherent hydroxyl, carboxyl, ether bonds, etc. contained therein are the initial sources of oxygenated functional groups and also important factors affecting the content of oxygen-containing functional groups. Jiefeng Chen's team studied sawdust and soybean straw and found that the former has an oxygen content of 44.90 wt%, while soybean straw has an oxygen content of 43.33 wt%, making sawdust more suitable for preparing biochar electrodes with high oxygen content and high active sites [4].

### 2.2. Oxygen-containing functional groups generated during pyrolysis

Controlling the types and amounts of oxygenated surface functional groups through regulating the pyrolysis process is an important means in modern production. For example, Han Ping's team adjusted the pyrolysis temperature and discovered through infrared spectroscopy that biomass tends to retain relatively stable -OH groups. Through processes such as dehydration, decarboxylation, bond breaking, cyclization, and rearrangement, carboxyl, phenolic hydroxyl, carbon-carbon double bonds, and carbon-oxygen double bonds can be eliminated, while aromatic hydrocarbon functional groups can be increased. However, as the temperature further increases, if it reaches 900 degrees Celsius or above, it will decompose into gases such as CO or $CO_2$ and be released [5].

In addition, the type of functional groups present is affected by the pyrolysis atmosphere. Li Hui and Xin Xing's team noted the presence of oxygen and its impact on -OH and -CH. With the increase of oxygen, the absorption peaks of the two groups become less. This is potentially due to the facilitative role of oxygen in the cleavage of -OH and -CH. The free hydrogen ions formed will combine with oxygen in the pyrolysis atmosphere to become water. On the other hand, CO2 is in the pyrolysis intermediate isomerization and therefore isomerization of the intermediate, which in turn reduces the formation of phenolic and aromatic compounds, increases the formation of ketones and heterocycles and even in an N2 atmosphere, phenols with branched methoxy groups are produced [6,7]

### 2.3. Directional introduction of oxygenated functional groups

Directional introduction is currently the most commonly used method for introducing oxygenated functional group., beacuse of its specific characteristics. For example it was controllable, engineering-valuable, and performance-optimizing. And chemical oxidation and high-energy field-assisted are the two most commly used methods among it. .

#### 2.3.1. Chemical oxidation method

One such modification method is chemical oxidation, which involves oxidizing and etching the surface of biochar using strong chemical oxidants to produce functional groups containing oxygen. Depending on the medium, it may be classified into liquid-phase oxidation and gas-phase oxidation. Oxidizing solutions, commonly including nitric acid, sulfuric acid and hydrogen peroxide, are typically used as modifiers in the liquid-phase oxidation procedure to treat biochar in mild conditions to enable the introduction of a large number of carboxyl, hydroxyl, lactone and other groups. In an experiment by the team of Fan (2018) it was discovered that when 5g of wheat straw biochar (prepared at 450 o C) was oxidized in 400mL of concentrated HNO3/H2SO4 solution at 70 C over 6 hours, the oxygen content had significantly risen to 20.09% compared to 1.32% before ox [8]. Moreover, the technique demonstrates significant strengths of relatively gentle etching, high degrees of controllability, and controlled proportion of carboxyl and hydroxyl groups. Conversely, the oxidation approach that operates in the gaseous phase typically employs oxygen, carbon dioxide, and ozone as oxidizers, and typically forms weakly polar oxygen-containing functional groups, which include carbon-oxygen double bonds and ether bonds. This is an operation that runs at a higher temperature and hence can be carried out continuously because there is no waste liquid and is also cost effective and flexible to large scale production. Nevertheless, it also possesses shortcomings like over oxidation causing the overflow of oxygenated surface functional groups, including CO/CO 2, which causes a reduction in O/C ratio, low selectivity, and poor controllability. The liquid-phase oxidation method is the most common method being used in laboratories today [9].

#### 2.3.2. High-energy field-assisted oxidation

In addition to traditional chemical oxidation methods, the use of high-energy fields such as plasma, microwave, and ultraviolet radiation for assisted oxidation is emerging as a significant research direction for regulating the functional groups on the surface of biochar, owing to its advantages of low reaction temperature, broad spectrum, and high efficiency. For instance, the team led by Istiaq Jamil Siddique discovered through research on microwave oxidation that microwave heating can process large-particle-size raw materials, reduce crushing costs, and achieve extremely rapid temperature rise.With a sample mass of approximately 30 kg, the heating efficiency of microwave and traditional heating processes is about 3 o C/min and 1 o C/min respectively, and the internal temperature gradient of the heated object is less than that of traditional heating processes. Nevertheless, the application of microwaves to treat biomass involves in-depth study of the dielectric constant, loss factor and loss tangent of biomass which has not been forthcoming in previous studies. Moreover, because of the extreme variation in the dielectric properties of the biomass, as a function of temperature, power in microwaves must be dynamically adjusted to achieve efficacy. Hotspot effect, commercial microwave absorbers, and the suitable penetration depth are just some of the problems preventing a practical use of this technology [10]. Surface etching and photo-oxidative degradation that destroy carbonyl groups are challenges to plasma oxidation and ultraviolet radiation oxidation, respectively [11,12]. All in all, these processes, involving the use of high-energy physical fields to produce reactive oxygen species, are progressing in the face of disappointments, and slowly in the replacement of standard processes, which are highly polluting and energy-intensive. Both of which can be effective in the introduction of assorted oxygen-bearing functional groups, and are clean, efficient, and low-cost, the high-energy field-assisted oxidation approach even yields no waste liquid and secondary pollution, they nonetheless require intense oxidation to obtain functional group grafting. This predisposes them to over etching of the carbon skeleton of biochar in the oxidation process resulting in the structure of the pore collapsing and the specific surface area being reduced hence not reaching the performance upper limit of single doping. Thus, the mechanism of oxygen-containing functional groups on the surface of biochar to electrochemical performance and safety of supercapacitors needs to be systematically investigated, the role of oxygen-containing functional groups in ion transport needs to be clarified, and a method of introducing oxygen-containing functional groups to the surface of biochar to achieve synergistic optimization of performance, stability, and safety needs to be explored.

## 3. The dual regulatory effect and mechanism of oxygen-containing functional groups

On the performance of supercapacitors. Oxygen-containing functional groups can influence the core characteristics of electrodes by altering the nature and quantity of functional groups, thereby exerting a significant dual effect on the performance of supercapacitors.

### 3.1. Influence of the amount of oxygen-containing functional groups

For the original activated carbon with 10.5 wt% oxygen-containing functional groups, it was converted into thermally oxidized activated carbon with an oxygen content of 14.8 wt% through activation using KOH and heat treatment in a quartz tube furnace with an air-to-nitrogen volume ratio of 1:1. This led to a significant increase in its specific surface area to 3260.4 $m^2/g$, and an increase in pore volume and pore count. The zeta potential also indicated better wettability of the material. The synergistic effect of these four factors significantly enhanced the specific capacitance of the capacitor, from 287.0 F/g to 420.0 F/g at 1.0 A/g. However, the oxygen mass fraction is not necessarily higher the better. For instance, when it reached 18.3 wt%, the specific surface area decreased significantly to 2318.8 $m^2/g$, and the specific capacitance dropped to 266.0 F/g. This is because excessive oxygen-containing functional groups occupy the pore channels, block micropores, disrupt the sp2 hybridization of the carbon framework, and increase internal resistance. Finally, thermal deoxygenation of the original activated carbon was found to reduce efficiency. Therefore, the optimal mass fraction range is determined to be between 10% and 15% [12,13].

## 3.2. Effect of the type of oxygen-containing functional groups on performance

Hsieh's team classified the hydroxyl, carbonyl, and quinone groups that desorb CO as one type, while the carboxyl, anhydride, and lactone groups that desorb $CO_2$ as another type, using temperature-programmed desorption.

Subsequently, Qiu's team found that 0.58 $mmol \cdot g^{-1}$ of CO-producing groups could provide an additional capacitance of 7.032 $F \cdot g^{-1}$, while 0.08 $mmol \cdot g^{-1}$ of CO2-producing groups could produce a capacitance of 0.968 $F \cdot g^{-1}$. Therefore, the specific capacitances contributed by the two types of oxygen-containing groups were determined to be 12.12 $F \cdot mmol^{-1}$ for CO-producing groups and 12.10 $F \cdot mmol^{-1}$ for CO2-producing groups. It was believed that although CO-producing groups contributed more specific capacitance in acidic solutions, the difference was not significant [13]. However, in reality, due to the selection of too few CO2-producing groups, there was a significant error in calculating the specific capacitance [14]. Yitao discovered through the TPD method that the $CO_2$ desorption peak occurs below approximately 600°C, at which temperature free carboxyl, anhydride, and aliphatic groups undergo significant thermal decomposition [15]. In Yan's experiments, it was found that treating at 400°C led to an approximate 28% increase in capacitance [16]. This indicates that a large number of $CO^{2-}$producing groups can enhance capacitance by increasing wettability, reducing contact angles, and increasing active sites when present in small quantities. However, excessive amounts can create energy gaps, disrupt the conjugated system, inhibit Faraday reactions on the carbon surface, reduce capacitance, and decrease cycling stability [15]. Furthermore, capacitance represents the load capacity and is an important criterion for evaluating the performance of capacitors [17]. Total capacitance = double-layer capacitor capacitance + pseudocapacitor capacitance [18]. Therefore, the following analysis will focus on the performance of CO-producing groups in double-layer capacitors and pseudocapacitors.

For double-layer capacitor, in 2021, Wang's team discovered through quantitative calculations and CV, CS tests that carboxyl and phenolic hydroxyl groups contribute approximately 1.10 $\mu F\ cm^{-2} at.\%^{-1}$ to the additional capacitance in alkaline solutions, accounting for 30% of the total capacitance contribution. However, in double-layer battery systems, pore size and distribution still play a dominant role [19]. OFGS primarily enhances energy density by improving wettability, reducing ion transport resistance, increasing surface charge density, and boosting active site availability and surface area utilization—all without introducing additional capacitance. At the same time, excessive oxygen-containing functional groups can damage the electrochemical double layer, reduce additional capacitance, increase defects, and weaken conductivity [14,19].

For a pseudo-electric capacitor, as mentioned above, CO-producing groups such as hydroxyl, phenolic hydroxyl, carbonyl, ether, and quinone groups are often preferred for enhancing capacitance. However, regarding the gas produced by TPD of hydroxyl groups, Cheng's team attributed it to TPD of CO2-producing functional groups [20]. In fact, this is not absolutely accurate. For example, it may form cyclic lactones that can decompose into one molecule of CO2 or two molecules of CO, and these two reactions can occur simultaneously [21]. The following sections will focus on how these functional groups play different roles in different systems.

These groups mostly provide pseudocapacitance through Faraday reactions, which are rapid redox reactions often occurring on the electrode surface [22], and are more pronounced under acidic conditions. For example, a capacitor system composed of hydroquinone and benzoquinone can undergo a relatively stable reversible Faraday reaction : $HQ \rightleftharpoons BQ + 2H^+ + 2e^-$ under the catalysis of AC surface oxides, within a pH range of 2.2 to 7.2, thus providing a large amount of additional capacitance and exhibiting extremely high rate performance. Due to the reversibility of the reaction, after 1000 cycles, the capacitance retention rate remains above 82%, indicating good cycling stability [23]. Similarly, the resorcinol-formaldehyde system, as an electron acceptor, can contribute an additional capacitance of 196 F $g^{-1}$ in a 1 mol/L sulfuric acid solution, significantly higher than the 106 F $g^{-1}$ in 6 mol/L potassium hydroxide [24]. Ether bonds are more special, as they do not directly participate in Faraday reactions but promote pseudocapacitance by stabilizing the carbon framework and regulating the electronic structure, enhancing cycling stability. Especially, Ar-O-Ar groups, when used in electrochemical tests, showed that when the current density increased from 0.3 A $g^{-1}$ to 3.0 A $g^{-1}$, the capacitance only decreased by 18%. After 10,000 constant current charge and discharge cycles, the capacity of the capacitor only decreased by approximately 5%, which indicates that it played an extremely important role in enhancing the cycle stability [25].

## 4. Breaking through performance constraints

The synergistic enhancement approach to supercapacitors is co-doping of different elements to produce multiple active sites that establish more sites on which Faraday reactions can take place, and the pore structure is enhanced to provide better interfacial stability. As an illustration, capacitance of a capacitor with the nitrogen/phosphorus ratio about 4/5 and a porous carbon structure covered with Mn3O4 nanoparticles can be as high as 384 F g -1 at 0.5 A/g, a much higher value than the 260 F/g of a single-element supercapacitor. It also has a high rate of cycling stability, and retention rate of capacitance of 98.2% following 5000 cycles is far superior to the retention rate of hydroquinone and benzoquinone system of 82.0% following 1000 cycles [23,26]. This clearly depicts the great relevance of the heteroatom doping in improving the capacitor performance. The elements in doping are typically classified into N-type doping of N, S and P, P-type doping of B and multi-doping according to the electron donor and acceptor. All these factors have the potential to improve the capacitor performance in many ways. An example is that B can modify the density of the electron cloud by chemical adsorption and the creation of a Π system because of the differences in electronegativity, which facilitates Faraday reactions and, therefore, enhances capacitance. It is especially good at improving conductivity [27]. Sulfur (S) has been used to distribute along the edges, and it is very essential in enhancing the stability of capacitors and minimizing self-discharge. It exhibits a high and stable electrochemical activity, especially in alkaline electrolytes, which is not the case with oxygen. Phosphorus (P) has the ability to increase capacitance more than S, and is only effective with acidic solutions. Depending on the properties of individual constituents, rational control of the mass content can serve various requirements. As an example, a charge transfer rate, surface activity, wettability and capacitance are greatly enhanced with 1.08 at% B, 0.34 at% P, and 6.94 at% N, leading to a supercapacitor with a capacitance of 450.86 F/g and a capacitance retention rate of 85% after 5000 cycles in practical [28].

However, the precise determination method for the specific mass fraction of each element has not yet been clarified, and the approach to achieving the ideal working state under actual operating conditions still remains to be explored. There is also a lack of systematic and feasible solutions for the promotion and application of relevant research results in the fields of lithium-ion batteries and sodium-ion batteries.

## 5. Conclusion

Given the driving forces behind the energy transition and the continuous rise in electricity demand, there is a demand for biochar-based supercapacitors that combine long lifespan, high power, and low cost. Oxygen-functionalized groups, as the core factors regulating the performance of biochar electrodes, directly determine the electrode wettability, conductivity, pore structure, and pseudocapacitive behavior based on their type and content. It was first discovered that oxygen-containing functional groups in biochar mainly originate from inherent components of biomass, in-situ generation during pyrolysis, and directional introduction through chemical/high-energy field oxidation. Precise construction of functional groups can be achieved by regulating raw materials, pyrolysis conditions, and modification processes. Oxygen-containing functional groups have a dual effect on the performance of supercapacitors: an appropriate amount of OFGS (oxygen content 10%–15%) can enhance wettability, increase active sites, and provide pseudocapacitance, significantly improving specific capacity; excessive oxygen-containing groups can block pores, damage the $sp^2$ carbon framework, and increase internal resistance, leading to a decrease in capacity and cycle stability. Among them, CO-producing groups such as hydroxyl and carbonyl groups are more conducive to pseudocapacitance and rate performance, while excessive $CO_2$-producing groups such as carboxyl and lactone groups tend to deteriorate electrochemical performance. Combining the regulation of surface oxygen functionalities with the co-doping of B, N, and P ternary heteroatoms can break through the limitations of single elements, synergistically optimize electronic conduction, ion transport, and structural stability, achieving a balance of high capacity, high rate, and long cycling. The following article describes the development processes and regulation methods and the structure-activity relationship of oxygenated functional groups in biochar. It also serves as a basis and explains the methods to design better and cheaper biochar supercapacitor electrode materials. In our further studies, we aim to better understand the synergistic effects among various functional groups, achieve an overall enhancement of capacitor performance by finely adjusting types and the amount of heteroatoms, and extend the use of biochar in combination with energy storage systems, especially in lithium and sodium batteries.

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