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Catalytic Graphitization of Biomass as a Potential Method Produce Graphite In The Future : A Review

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Abstract: The traditional graphitization process involves the use of non-renewable carbon sources and high temperatures, which are time-consuming and expensive. Biomass has been proposed as an alternative renewable source of carbon, which can be graphitized at lower temperatures using transition metal catalysts. The article highlights successful research on graphitization of various biomass carbon sources, such as coconut coir, whey protein, pine wood sawdust, mangosteen peel, miscanthus grass, and palm kernel shell waste, using metals as a catalyst. The graphitization process using catalysts derived from transition metals has been shown to reduce the graphitization temperature, shorten the graphitization time and improve the physicochemical properties of the resulting graphite material.

Keywords: Graphite; Biomass; Catalytsts; Graphitization; Metals

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1. Introduction

Graphite is an allotrope of carbon which has a unique layered structure. In a layered graphite crystal structure, carbon atoms form a hexagonal network with sp2 hybrid orbitals that contribute to strong covalent bonds in the graphite layers, while between graphite layers are connected by weak van der walls forces [1]. Graphite carbon has been widely used in energy storage systems since the early 1990s because this material has high electrical and thermal conductivity, graphite crystal structure, good physicochemical stability, making it suitable for use in ion diffusion processes in lithium battery systems [2].

Graphite is a potential material for anodes in lithium ion batteries (LIB) which has the advantage of incorporating Li+ ions at low voltage, safety and durability [3]. Graphite represents an advanced material for anodes as it offers medium gravimetric capacity (372 mAh/g, LiC6 stoichiometry), long cycle life, low working potential (0.2 V vs Li/Li+), and low voltage, indicating high efficiency energy [4]. Graphite is an isomer of the element carbon, and consists of a hexagonal layered structure of crystalline carbon. Its unique structure contributes to graphite's excellent electrical conductivity and thermal and chemical stability [5]. Therefore, graphite is widely used in the production of refractory materials, automobiles, batteries, supercapacitors and graphene [6].

Graphite generally comes from non-renewable carbon which is processed through carbon treatment at very high temperatures around 2000-3500 C and takes several weeks to transform from amorphous carbon to crystalline carbon (graphite).[7].

The calcination process at high temperatures (1800 C-3500 C) of carbon materials causes the rearrangement of carbon atoms and increases the degree of graphitization of the carbon material. [8]. Sources of graphite from mining materials are generally petroleum coke, asphalt, coal and other carbon sources which are treated by means of: steam deposition or carbon precursors which are treated at very high temperatures in the temperature range of 1800–3500 C and require a long time and cost expensive [9]. Therefore, a renewable graphite source and a faster and simple graphitization process at low temperatures are needed to produce graphite.[8].

Biomass has been widely used as an alternative source of carbon to obtain graphite as a transitional form of graphite sources from earth-based carbon to natural material-based carbon with renewable properties [10]. Biomass is also a cheap source of carbon and the graphitization process can be carried out at low temperatures using metal as a catalyst for graphitization process [11]. In addition, it has a natural tubular structure that can increase the diversity of pores in artificial carbon materials and thereby increase the migration rate of ions in the electrodes in electrochemical applications [12]. The graphitization process with a biomass carbon source is considered "carbon-neutral" because the emissions released during pyrolysis are absorbed from the atmosphere. Biocarbon is a porous material with a high surface area, which can be increased by physical or chemical processes using steam activation, milling, or potassium hydroxide (KOH) to increase the surface area [13].

The use of catalysts derived from transition metals such as Fe, Co, Ni has been proven successful in reducing the graphitization temperature for carbon materials from biomass to less than 1000 C.[9]. Research related to the graphitization method using a catalyst has been successfully carried out by several researchers to reduce the graphitization temperature and shorten the graphitization time. The graphitization catalytic process using Ni catalyst derived from NiCl2 solution succeeded in lowering the graphitization temperature to form graphite from coconut coir carbon at 1300 C for 3 hours[14]. Another catalytic graphitization process that has been successfully carried out by several researchers is the graphitization of carbon derived from whey protein with a Ni catalyst derived from Ni(NO3)2 solution at a temperature of 700 C for 1 hour. [15]; Carbon derived from sawdust of pine wood has also been successfully carried out by catalytic graphitization process using Fe catalyst derived from Fe(NO3)3 solution with temperature variations of 700 C – 1000 C within 1 to 4 hours. [3]. The use of a Co catalyst for the catalytic graphitization process has also been carried out to form graphite from a carbon source of mangosteen peel and a catalyst derived from a solution of CoCl2 at 800 C for 2 hours [16]. The next development of the graphitization catalytic method is the use of a bimetallic catalyst in the graphitization process, sawdust has been successfully graphitized using a Ni-Mo catalyst derived from a solution of NiCl2-(NH4)6Mo7O26 at a temperature of 750 C for 2 hours [8]. Another catalytic graphitization process using biomass as a source of carbon are miscanthus grass with iron and cobalt as a catalyst [17], and palm kernel shell waste as a source of carbon with iron and nickel as a catalyst [18]

Various attempts were made by researchers to reduce the graphitization temperature and shorten the graphitization time of biomass as a renewable source carbon using metals as a catalyst. This article, thus, focuses on review the graphitization of biomass using metals as a catalyst. The work provides an overview of the effect of using metals as a catalyst for the graphitization of biomass to produce high value graphitic materials. Existing state-of-the-art graphite characterization are also discussed

2. The Effect of Using Metals As A Catalyst For The Graphitization Biomass

Destyorini et al. investigated the potential of coconut fiber as a carbon source for the catalytic graphitization process using Ni catalysts from NiCl2 solution. The process began with the carbonization of coconut fiber at 500°C for 1 hour in a furnace with N2 gas flow to obtain coal-rich carbon. The carbon was then impregnated with NiCl2 solution, followed by pyrolysis at 1300°C with N2 gas flow. The removal of Ni metal after the catalytic graphitization process was carried out using hydrochloric

acid solution. The results showed that the temperature of coconut fiber graphitization process decreased to 1300°C, producing graphite material with a graphitization degree of 88.4%, lower than that of commercial graphite (90.23%). XRD characterization showed that the broad peak at 2 theta 20-30 degrees (amorphous carbon) began to disappear at a temperature of 1200°C, at which a sharp peak with high intensity appeared at 2 theta 26-27 degrees, indicating that graphite began to form at 1200°C. [7, 14].

The potential of sucrose as a carbon source for graphite materials has been investigated by Liu et al using a catalytic graphitization process with Ni metal originating from Ni(CH3COO)2 solution which produces graphite material with an IG/ID value from the raman characterization results of 1.38 and a surface area of based on the characterization of S BET of 691 m2/g. The catalytic graphitization process takes place at a temperature of 900 C with a rate of 5 C/min [19].

Variations of Ni catalyst from various solutions, Ni(NO3)2, Ni(CH3COO)2, and NiCl2 in the catalytic graphitization process using coconut shell carbon sources with temperature variations of 900 C – 1400 C using a microwave have been carried out by Khoshk Rish, Tahmasebi, Wang, Dou, & Yu. The results showed that the Ni source solution which produced the best graphitization process was Ni(NO3)2 and Ni(CH3COO)2 at a temperature of 1400 C for 30 minutes with an IG/ID value of 4.16 and a surface area of 148 m2/g [19].

Wang et al investigated the potential of a carbon source for graphite from whey protein with a catalytic graphitization process using a Nickel catalyst. This process begins with the carbonization of whey protein which has been doped with nitrogen at a temperature of 400 C. After that, the carbon is activated using KOH and impregnated with Ni catalyst using Ni(NO3)2 solution. The catalytic graphitization process took place at 700 C for 1 hour. The results of this study indicate that catalytic graphitization with a whey protein carbon source using a Ni catalyst produces graphite material with a good level of graphitization with an IG/ID value of 1.2 and a surface area of 2536 m2/g[15].

Choi, Choi, & Seo have investigated the carbon source for graphite material from carbon fiber-based PAN (polyacrylonitrile) by catalytic graphitization process using Ni catalyst derived from NiCl2 solution. This research shows that the temperature of the graphitization process takes place at 600 C for 1 hour to produce graphite material with a high level of graphitization, namely with an IG/ID value of 1.16 [20].

The use of a Fe catalyst derived from Fe(NO3)3 solution has also been investigated by Nakayasu et al in the catalytic graphitization process with a carbon source of pine sawdust with a ratio of Fe : pine sawdust of 4 : 10 at a temperature of 850 C. The graphite material produced in the process It has the characteristics of an average interlayer distance (d002) of 0.337 nm and a crystallite size of 35.8 nm for La and 56.1 nm for Lc which is comparable to commercial graphite [3]. According to Sun et al, who also studied pine wood sawdust using Fe catalysts derived from Fe(NO3)3, graphite was formed at a temperature of 700 C due to the formation of Fe nanoparticles caused by the reduction of Fe3+ to Fe at that temperature. Increasing the amount of catalyst can provide a contact-promoting reaction site between Fe and C, indicating that the amorphous carbon is dissolved on the Fe nanoparticles and precipitated into graphite carbon. Based on this, a "dissolving-deposition of carbon" mechanism is proposed to explain the formation of the graphite structure and the overall pyrolysis process. Increasing the temperature of the graphitization process to 800 C accelerates the process of reducing Fe3+ to Fe and accelerates the process of dissolving and depositing Fe-C, so as to accelerate the formation of graphite structures [6].

Utilization of mangosteen peel as a carbon source for the catalytic process of graphitization with a Co catalyst derived from a solution of CoCl2 which lasted at 800 C for 2 hours and produced graphite material with a high level of graphitization, namely an IG/ID value of 1.26 and a surface area of 1168 m2/g. In the catalytic graphitization process, Zha et al impregnated mangosteen peel with Co catalyst using CoCl2 solution, then activated using KOH solution, after that the catalytic graphitization process was carried out in a furnace with temperature variations of 600

C, 700 C, 800 C, and 850 C to observe the morphology, degree of crystallinity, and surface area of the resulting graphite material [16].

Dandelion flowers as a carbon source for the catalytic graphitization process using K2FeO4 have also been studied by Tan et al. 1 g dandelion stalks were soaked in 20 ml of K2FeO4 solution with various masses (respectively 0.05; 0.1; 0.15; 0.2 and 0.25 g) while stirring for 12 hours. Subsequently, the solvent was evaporated and dried at 70°C. Then, the samples were carbonized at 700°C, 800°C, 900°C for 2 hours. After that, the samples were soaked in 1M HCl for 6 hours and washed several times in water, followed by drying at 70°C for 1 night. The sample obtained is high porous biomass graphite carbon with an IG/ID value of 1.26 at a graphitization temperature of 800 C and a surface area of 1168 m2/g [9].

A recent graphitization catalytic process using a Ni-Mo bimetallic catalyst was investigated by Li et al with sawdust carbon as a source. In this study, activation of the carbon shaft from sawdust was carried out using a 3M H2SO4 solution for 6 hours in an 80°C water bath, after which the samples were rinsed with water and dried at 100°C. Furthermore, the samples were placed in a KOH solution with a ratio of 1:1. in a water bath at 60°C and dried with an evaporator at a temperature of 80°C. The sample was then placed in a furnace to be carbonized for 2 hours at a temperature of 750°C with a stream of N2 gas. Then the sample was washed again with 2M HCl. The sample is called activated carbon. The preparation of activated carbon impregnated with Ni and Mo catalysts was carried out by incorporating 1 g of activated carbon in a solution of NiCl2 and (NH4)6Mo7O24 at a mass ratio of 1 : 3 : 1, then the sample was treated with the same conditions as activated carbon using KOH, which was placed in furnace for carbonization for 2 hours at a temperature of 750 C with N2 gas flow. After the catalytic graphitization process, the samples were washed with 1M HCl and 30% H2O2 solution to remove Ni and Mo metals. The IG/ID value of the graphite material produced in this process is 1.05 with a surface area of 1366 m2/g [8].

The study emphasizes the collaborative catalytic effect of iron(III) and cobalt(II) nitrates on the pyrolysis of Miscanthus grass. The combined use of these metals has been found to be more effective in producing highly ordered graphitic carbon than when they are used separately. The catalytic effect of iron and cobalt on the degradation of ligno-cellulosic biomass led to the production of new metal species observed in situ through XRD analysis, which significantly impacted the level of biocarbon graphitization. Based on Raman and XRD data, it has been proposed that the formation of FeCo alloy nanoparticles is responsible for achieving the highest level of graphitization. The study suggests that increasing the pyrolysis temperature could improve the yield of graphite. Although the research primarily focused on temperatures below 1000°C for modeling purposes, the study's direct implications relate to heat treatment [17]

The carbonization of palm kernel shell is currently being used in conjunction with ferum nitrate as a catalyst for graphitization. The degree of graphitization can be adjusted by altering the temperature, raw materials, and type of catalyst used. Higher heat treatment temperatures of up to 1000 ◦C lead to a higher degree of graphitization. XRD data analysis revealed that all samples showed a 2θ = 26◦ peak, but the samples prepared using an iron catalyst displayed higher and sharper peaks with a lower ID/IG ratio, indicating higher crystallinity and fewer defects [18]

Table 1 shows summary of the effect catalytic graphitization. In conclusion, the reviewed studies have shown that the catalytic graphitization process with different metal catalysts has the potential to produce graphite materials from various carbon sources. The temperature and duration of the process, the type of metal catalyst used, and the carbon source material all play important roles in determining the level of graphitization achieved. The results of these studies demonstrate that coconut fiber, sucrose, whey protein, carbon fiber-based PAN, pine sawdust, and mangosteen peel can all be used as carbon sources for the catalytic graphitization process. Additionally, Ni, Fe, and Co catalysts have been shown to be effective in promoting the graphitization process. Further studies are needed to optimize the process conditions and investigate the potential of other carbon sources and metal catalysts for the production of graphite materials.

Table 1. The Effect of Catalytic Graphitization

No	Author	Source of Carbon	Catalyst	Operation Conditions		IG/ID	S BET (m2/g)
				Temperature	Time		
1	Destyorini et al., 2021 [7, 14]	Coconut Coir	NiCl2	1300 C	3 hours	1.01 ± 0.04	51,61
2	(Liu et al., 2015)[19]	Sucrose	Ni (CH3COO)2	900 C	5 C/min	1.38	691
3	Khoshk Rish, Tahmasebi, Wang, Dou, & Yu, 2021 [2]	Coconut shell	Various Ni catalysts: **Ni(NO3)2; Ni(CH3CO2)2**; NiCl2	900 C; 1000 C; 1100 C; 1200 C; 1300 C; **1400** C	0, 15,**30**, and 60 min	4,16	148
4	Wang et al., 2016 [15]	Protein Whey	Ni(NO3)2	700 C	1 hours	1,20	2536
5	Choi, Choi, & Seo, 2022 [20]	PAN-based CF	NiCl2	**600 C** - 800 C	1 hours	1,16	
6	Nakayasu, Goto, Katsuyama, Itoh, & Watanabe, 2022 [3]	Pine sawdust	Fe(NO3)3	700 C - 1000 C	1 - 4 hours		
7	Sun et al., 2022 [6]	Pine sawdust	Fe(NO3)3	600 C - **800** C	5 C/menit		112,139
8	Li et al., 2022 [8]	Sawdust	NiCl2 - (NH4)6Mo7O26	750 C	2 hours	1,05	1366
9	Tan, Xu, He, & Li, 2022 [9]	Dandelion flower	K2FeO4	700 C	2 hours	1,103	780,94
10	Zha et al., 2021 [16]	Mangosteen peel	CoCl2	800 C	2 hours	1,26	1168
11	Major et al 2018 [17]	Miscanthus grass	iron(III) and cobalt(II) nitrates	500-900 C	10 C/min		
12	Jabarullah et al 2021 [18]	Palm kernel shell	iron(III) and Nickel (III) nitrates	1000 C			

3. Graphite Characterization

Fig 1 shows the results of SEM characterization of coconut shell charcoal and graphite powder. Morphological differences between coconut shell charcoal and graphite are shown from the sample surface where coconut shell charcoal has a porous structure and a flat surface with particles, while graphite has granular particles with different size distributions [21]

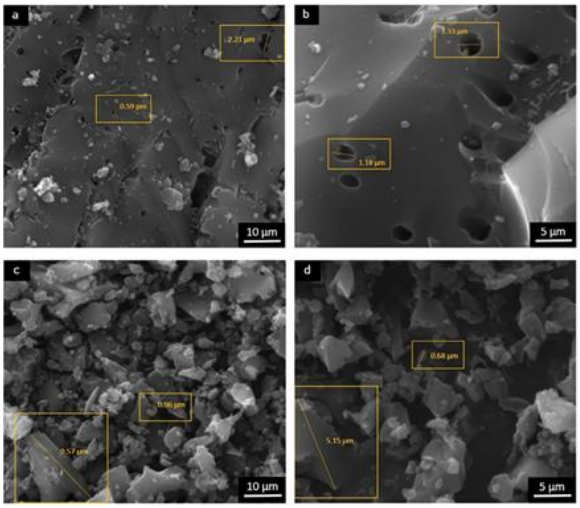

Figure 1. Coconut shell charcoal morphology (a-b); and graphite powder (c-d)[21]

Fig 2 shows the XPS results from graphite produced through a catalytic graphitization process using Ni-Mo with sawdust carbon as a source. The XPS results in Fig 2 show that the graphite produced from the graphite method with the Ni-Mo catalyst after treatment with HCl and H2O2, it can be seen that the graphite only consists of the elements C, N, and O. This means that the metals Ni and Mo have been successfully removed. The high-resolution XPS spectrum of the C1s can be divided into three peaks by deconvolution and integration peak processing, which are located at 284.6 eV (C-C/C=C), 285.4 eV (C-O/C-N), and 288.5 eV (C =O). The N 1 s spectrum can be deconvolved into two locked peaks at 398.7 eV (N-6) and 400.4 eV (N-5). According to the results of previous studies, N-5 and N-6 are located on the edge of the graphite layer, which can produce more active sites for storing charges, and their configuration significantly increases the pseudocapacitance of carbon materials.[8].

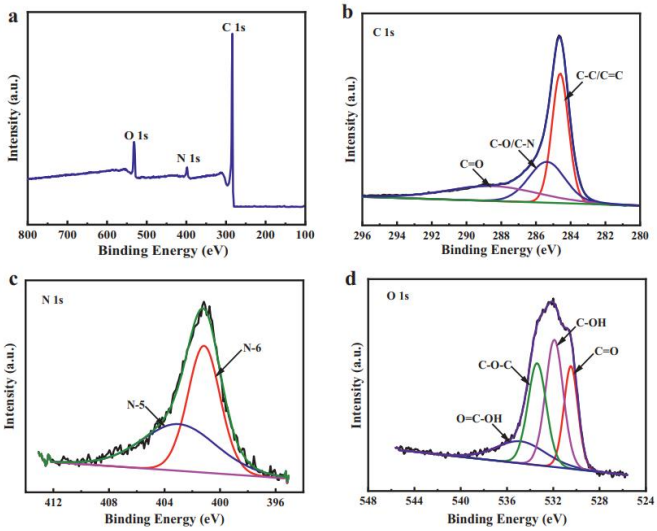

Figure 2. The XPS graph from PGC-Ni-Mo; high-resolution XPS spectra of (b) C 1s, (c) N 1s, and (d) C 1s for PGC-Ni-Mo [8]

Sample characterization using Raman spectroscopy is a technique to obtain information about the microstructure of various carbon materials. Carbon materials with a high degree of graphitization on the Raman characterization results show only 1 band in the area between 1100 cm-1 to 1700 cm-1 and show a second band between 2400 cm-1 and 3300 cm-1. The G band of the graphite material around 1580 cm-1 has E2g symmetry and is related to the relative motion of the carbon atoms with sp2 bonds. In microcrystalline carbon materials, a band appears next to the G band, called the D band, in the region around 1350 cm-1. The G band and D band are generally associated with the graphitic structure of sp2 carbon. The ratio of D bands and G bands (ID/IG) indicates the degree of graphitization of carbon. The lower the ID/IG value or the higher the IG/ID value indicates a good level of carbon graphitization [22].

Fig 3 shows the results of the characterization with the Raman instrument on graphite material with a dandelion flower carbon source by the graphitization process using K2FeO4. Highly porous graphite biomass carbon (HGPBC) from dandelion was prepared by one-step activation (carbonization with) K2FeO4. The two bands located at ~1328 cm− 1 and ~1588 cm− 1, correspond to the D band and G band, respectively. In general, the intensity comparison of the D-band and G-band (ID/IG) is to explain the degree of graphitization. It is noted that the ID/IG of HGPBC-700-K2FeO4 is 0.906 lower than PC-700-KOH (0.939) and DC-700-0 (0.954), indicating a higher degree of graphitization than HGPBC-700-K2FeO4 and implying electrical conductivity. higher. These results indicate that the activation of K2FeO4 can increase graphitization [9]

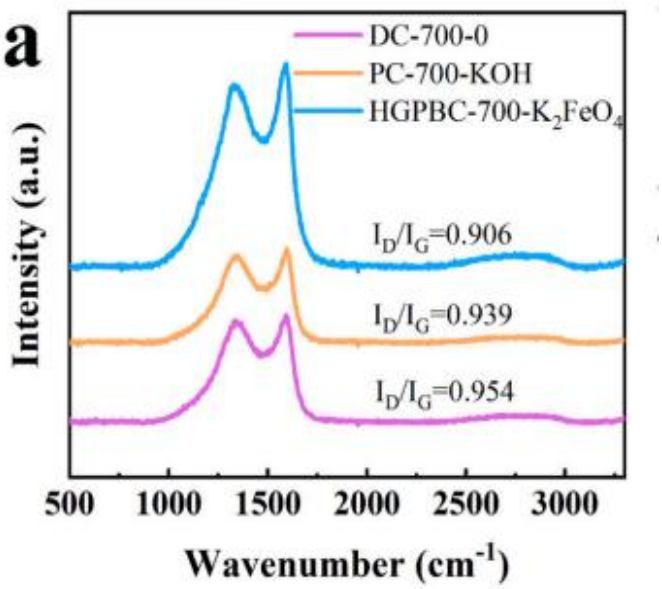

Figure 3. Raman Spectra of DC-700-0, PC-700-KOH dan HGPBC-700-K2FeO4 [9]

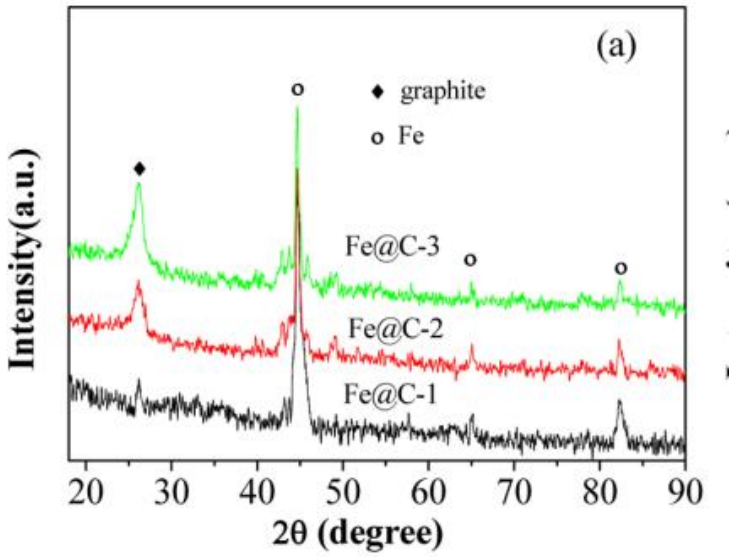

Figure 4. XRD pattern of Graphite material resulting from lignin graphitization process with Fe catalyst[23]

Further characterization of the graphite material resulting from the graphitic catalytic process using a metal catalyst and a biomass carbon source is by X-ray diffraction (XRD) which can see disturbances, but not defects in the basal plane [24]. Fig 4 shows the XRD pattern of graphite material from a lignin carbon source with a catalytic graphitization process using an Fe catalyst. The 2 theta peak shown in Figure 7 is around 26.3 which indicates the presence of a layer of graphite [23]

Overall, these characterization results provide valuable insights into the microstructure and properties of carbon materials produced through various graphitization processes, which are useful for developing high-performance carbon-based materials for various applications.

4. Conclusion

In summary, the catalytic graphitization process has the potential to produce high-quality graphite from biomass. By subjecting biomass to carbonization at lower temperatures followed by graphitization at relatively higher temperatures using a catalyst, the resulting graphitic chars can have various applications, such as in energy storage, water filtration, and electronics. Using metals as a catalyst increase the graphitization degree, and decrease the time and temperature for the graphitization process. This process is not only environmentally friendly but also economically viable since it utilizes renewable biomass resources. Therefore, it is a promising technology for producing high-quality graphite materials for various industrial applications. Meanwhile, Further studies are needed to optimize the process conditions and investigate the potential of other carbon sources and metal catalysts for the production of graphite materials. Typically, when carbon is heated at lower temperatures and then further heated at higher temperatures, it produces high-quality graphitic chars.

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