

Utilization of Biomass as a Carbon Source for The Synthesis of Graphene as a Sustainable Materials Innovation

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Abstract

One of the alternative ways to reduce the use of fossil fuels is the advancement of energy storage devices and energy conversion devices for renewable energy. The electrode is an essential part of its electrochemical performance in energy storage. The specific surface area of the electrode will affect the energy density, lifetime, charging, and discharging of the energy storage devices. Graphene has been widely used for energy storage, such as batteries and supercapacitors. Generally, graphene is synthesized from graphite as a carbon source which some researchers have reported. However, they are still fighting against simple synthesis methods, low-cost raw materials, eco-friendly co-product, and large-scale production. Therefore, advanced research is required to bring graphene from laboratory-scale projects to industry. One of the best solutions is biomass as a raw material to replace graphite in synthesizing graphene. In this review, some methods for synthesizing graphene from biomass will explain briefly, following with their strength and weakness.

Keywords:

graphene; biomass; carbon; sustainable materials

Introduction

Biomass has been used for fuel sources, perfume, cosmetics, food additives, nutritional elements, cleaning products, detergent, and plastic because of its abundance, carbon sources, and safety. Lignocellulosic biomass, composed of cellulose, hemicellulose, and lignin, was used to produce biodiesel using oleaginous microbe to collect lipid content of plant (Chintagunta et al. 2021). That could enhance the economic value of agricultural residues as the largest source of lignocellulosic (Yousuf 2012). In addition, the biomass utilization for essential oil is gradually increasing since Lummiss et al. (2012) found a method to assemble complex structures from a molecular structure in plant materials for the first time. The essential oil was widely used for perfume, cosmetics, and aromatherapy (Lummiss et al. 2012). The use of microalgae biomass (*Chlorella sorokiniana*) as a food additive in pasta recipes could exchange



synthetic dye. The addition of microalgae to pasta could enhance polyunsaturated fatty acid, chlorophyll, and carotenoid content, potentially preventing foodborne diseases (Bazarnova et al. 2021). Besides, household organic waste like fruit peels and vegetable dregs could be chemically treated to form eco-enzyme for cleaning products such as detergent, kitchen cleaner, and floor cleaner (Mavani et al. 2020). Moreover, biodegradable plastic (bioplastic) could be made from biomass such as algae and rice straw as a carbon source, potentially decreasing environmental pollutants and opening the opportunity to advance sustainable packaging products (Coppola et al. 2021).

Graphene is a two-dimensional (2D) carbon allotrope that possesses sp^2 carbon hybridization (Novoselov et al. 2004) with a hexagonal lattice and has a thickness of one atom (Novoselov et al. 2012). Graphene is the basic structure that forms other allotropes of carbon, such as fullerenes, nanotubes, and graphite. Zero dimension (0D) fullerene is a structure formed when graphene is rolled into a ball, 2D nanotubes are produced by rolling graphene into a tube, and 3D graphite is formed from graphene stacks or arrangements to produce a 3-dimensional shape. Graphene material has attracted many researchers' attention because of its fascinating electronic, thermal, and mechanical properties (Mu et al. 2014). Graphene has great potential for various applications such as transparent electrodes, flexible displays, energy storages (batteries, capacitors, and fuel cells), sensors, biosensors, biomedical, and energy generation. Among the superior properties of graphene include excellent thermal conductivity (5 x 10³ W.m⁻1.K⁻¹), high electrical conductivity reaching (6 x 10³ S.cm⁻¹), high charge carrier mobility at room temperature (2.5 x 10⁵ cm².V⁻¹.s⁻¹), and large theoretical surface area (2.63 x 10³ m².g⁻¹) (Troncoso and Torres 2020).

In general, graphene is synthesized from graphite as a carbon source. Various methods of synthesizing graphene from graphite include the mechanical exfoliation graphene (Scotch-tape) method (Novoselov et al. 2012), chemical vapor decomposition (CVD) (M. Wang et al. 2015), epitaxial growth on SiC substrate (Whitener and Sheehan 2014), and reduction graphene oxide (RGO) (Husnah et al. 2017) (Dreyer et al. 2010). The mechanical exfoliation method was used to synthesize graphene by Geim and Novoselov. This method is carried out by peeling off crystalline graphite or carbon layers to the micrometer scale using a stylus. One graphene sheet was observed hanging on a silicon oxide substrate with an optical microscope, this method can



produce graphene with good quality, but the results obtained have a low quantity (Novoselov et al. 2004) (Novoselov et al. 2012). The CVD method is a graphene synthesis method using SiO₂ substrate as a medium for the growth of carbon atoms into graphene. This method can produce graphene in large quantities but requires relatively expensive costs due to it is needed supporting equipment with high technology. Epitaxial growth method growth on SiC substrate also requires relatively expensive fabrication costs because this method requires high vacuum conditions (Berger et al. 2004).

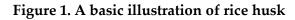
Another synthesis method developed is RGO (Stankovich et al. 2007). This method synthesizes graphite into graphite oxide by involving several chemicals, namely concentrated acids such as sulfuric acid, nitric acid, and phosphoric acid, then oxidation of graphite to graphene oxide (GO) using powerful oxidizing reagents such as potassium permanganate and potassium perchlorate. The final step is that the oxide bond in GO is reduced by using chemical or thermal reducing agents (Nekahi, Marashi, and Haghshenas 2014) (Chua and Pumera 2014). Chemical reducing agents involve chemicals such as hydrazine, sodium borohydride, hydroxylamine, glucose, hydroquinone, and L-ascorbic acid. Thermal reducing agents involve heating processes such as the furnace, microwave, or hydrothermal. The graphene oxide reduction method based on graphite requires several complicated steps, a variety of chemicals, and a long synthesis time.

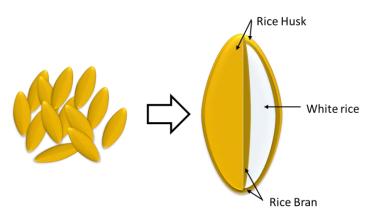
Therefore, a simple method made from cheap, environmentally friendly raw materials can be produced on a large scale, and sustainable development is still a challenge. Recently, activated carbon from biomass is one of the materials with high potential as a carbon source to replace graphite in graphene synthesis. Biomass is organic material that usually comes from plants or their derivatives, such as wood, wood waste, crops, agricultural production waste, etc. The use of biomass as a carbon source has several advantages, including cost-effectiveness because it is cheap and abundant, environmentally friendly, can be produced on a large scale, and is a sustainable and renewable material (Ouyang et al. 2021) (Saha and Dutta 2021) (Raghavan, Thangavel, and Venugopal 2017) (Yang Liu et al. 2018). This brief review presents several preparation processes for synthesizing graphene from biomass, including mechanisms, methods, and applications.



Graphene from rice husk

Rice husk is a by-product of rice generated during milling, the outer hard protective layer of rice grain (shown in Figure 1). About 20% of the rice weight is received as a husk during rice milling. The world annual production of rice husk is about 120 million tons (Tuck et al. 2012), and it is estimated to increase each year along with the increasing quantities of rice processing, impacting environmental problems (Safian, Umar, and Mohamad Ibrahim 2021). Recently, many researchers have developed the use of rice husks as a carbon source for the synthesis of graphene (Muramatsu et al. 2014) (Seitzhanova et al. 2018) (Sankar et al. 2017) to reduce environmental problems and increase the economic value of rice husks. Rice husk contains chemical compounds that can be used as a carbon source to synthesize graphene. Recently, rice husk has become the interest of many researchers because it contains chemical compounds that can be used as a carbon source to synthesize graphene. Recently, rice husk consist of 50% cellulose, 30% lignin, and 20% silica (Seitzhanova et al. 2018). Cellulose and hemicellulose or lignin content are the main compounds for activating carbon, while the silica content is a constituent of impurities in the synthesis of graphene; to remove it requires pretreatment using an alkaline solution.





In general, the synthesis of graphene from biomass sources is carried out in several processes, specifically carbonization, graphitization, and activation. Carbonization is the process of increasing the carbon content through thermal treatment to remove low molecular weight compounds. In other terms, carbonization is often associated with adjusting the carbon



structure. Graphitization is related to arranging the structure of carbon into a graphite-like structure, usually employing further thermal treatment after carbonization. Activation is a process to remove impurity compounds that coat the carbon surface to open or increase the porosity of activated carbon and increase the diameter of the pores that have been formed in the carbonization process. The activation process can be done chemically, physically, or by combining the two. Chemical activation is carried out using chemical materials such as KOH, H₂SO₄, NaOH, NaCl, and others (Stojanovska et al. 2019) (Yan Liu et al. 2012), while the physical activation method can be through heat treatment.

Muramatsu et al. (2014) have first reported the synthesis of graphene from rice husk as a carbon source using potassium hydroxide (KOH). The main processes used in this experiment are carbonization for conversion of rice husk (biomass source) to rice husk ash (RHA), chemical activation using KOH to transform RHA into graphene-based structures. Then the mixture of RHA with KOH was heat treated. During the synthesis process, the mixture of RHA with KOH is entered in a container coated with carbon black to protect against oxidation against air. Activation of RHA using KOH can result in high-purity graphene formation, producing graphene with an enriched edge structure, clean, stable, and atomically smooth edges. The resulting graphene has the potential as a high-performance carbon-based energy storage fabricating and conversion device (e.g., supercapacitors and hydrogen storage systems), next-generation water filters, and various nanocomposites.

Rhee et al. (2015) also used KOH as a chemical activation reagent to synthesize graphene from rice husks. The use of KOH as the activation reagent involves a redox reaction between carbon (C) and KOH, which influences the morphology of the generated graphene, expands the lattice spacing, increases the specific surface area of graphene, and promotes the formation of a porous structure. However, the interaction of C with KOH can produce inorganic impurities, accordingly that further purification is needed to remove potassium metal from graphene (Liou and Wang 2020). The purification process can be carried out by washing the activated carbon using hydrogen peroxide (H₂O₂) and deionized (DI) water (Rhee et al. 2015). Singh et al. (2017) have modified the synthesis process Muramatsu et al. (2014). The modification was carried out by replacing carbon black with rice husk (Singh, Bahadur, and Pal 2017). Modifications have been reported to replace carbon black with rice husk itself, making



graphene synthesis more cost-effective.

As shown in some of the literature above, it can be concluded that the synthesis of graphene using rice husks as raw material is possible. In addition, because it involves a simple process and is cost-effective, this approach can be applied in synthesizing graphene in large quantities. Moreover, the synthesis of graphene from rice husks can be applied in various applications in the field of civil engineering (such as cementitious construction materials) (Rhee et al. 2015), transistors, lithium batteries (Liang et al. 2017; Xue et al. 2021), and supercapacitors (Liang et al. 2017). Among the applications mentioned, supercapacitors are considered to be one of the most appropriate uses for graphene derived from rice husks. Sankar et al. (2017) have succeeded in synthesizing graphene nanosheets using the carbonization method followed by an activation process using one-stage KOH for supercapacitor electrodes (Sankar et al. 2017). The resulting graphene nanosheet has a higher surface area than the previous literature.

Graphene from rice straw

The Rice straw is a by-product of rice produced during the harvesting process. About 50 - 60% of the rice itself is straw. The amount of rice straw will increase along with the increase in rice production (Rezania et al. 2017). In general, rice straw is used as animal feed, fertilizer, compost, packaging material, and paper material, but most of it is burned in open fields for quick disposal purposes (Nam et al. 2018). This contributes to environmental problems and global warming (Jorn-am et al. 2021). Various strategies continue to be developed to increase the utilization of this type of biomass. Like rice husks, rice straw contains various compounds including cellulose, hemicellulose, and lignin which can also be used as a carbon source for the synthesis of graphene (Xu et al. 2020). The utilization of rice straw as a carbon source has been developed by many researchers (Sudhan et al. 2017). Suzuki et al. (2007) studied the use of rice straw as a carbon source. The results confirm that rice straw is a very potential material economically (Jin et al. 2018; Suzuki et al. 2007; Zhu et al. 2017).

Adinaveen et al. (2015) have reported the results of the synthesis of activated carbon from rice straw. A two-stage process namely carbonization followed by activation using phosphoric acid (H3PO4), generates morphological characteristics that lead to porous activated carbon, high surface area, and good electrochemical properties. These results are promising for high-performance supercapacitor applications (Adinaveen et al. 2015). Saad et al. (2019), have



produced carbon from rice straw by carbonization and KOH activation. Variations of heat treatment produce activated carbon properties similar to graphite in graphene sheets. The resulting surface area increases with the increase in activation temperature (Saad et al. 2019). Recently, Charoensook et al. (2021) have produced activated carbon from rice straw with a specific surface area and high specific capacity for energy storage applications. Activated carbon with these specifications is produced through a process of carbonization, activation using KOH and nitrogen doping treatment (Charoensook et al. 2021). Based on the literature review, it is evident that rice straw has great potential for various applications, especially in supercapacitor applications. Further development of methods is still needed to obtain high quality carbon from rice straw.

Graphene from coconut shell

The coconut shell is part of the coconut fruit covered by coconut husk, which is endocarpshaped and hard. The coconut shell is a protective layer between the coconut flesh and coconut husk (Sahat 2017). Coconut shell is considered a waste with low economic value, and their utilization is not yet optimal. Generally, coconut shell is used as a single-use fuel. Therefore, it is necessary to increase the utilization of coconut shell waste to increase its economic value. Coconut shell contains chemical compounds including cellulose (34%), hemicellulose (21%), and lignin (27%) which are composed of carbon elements (74.3%) (Bledzki, Mamun, and Volk 2010; Liyanage and Pieris 2015; Sekhon, Kaur, and Park 2021).

Cellulose is an organic compound with the formula (C6H10O5)n, which is found in cell walls and strengthens the structure. Hemicellulose is a heterogeneous polysaccharide polymer that fills the space between cellulose fibers in plant cell walls, a filler matrix for cellulose fibers, or a filler matrix for cellulose fibers. Another component is lignin, which binds to other cells and provides strength. Cellulose, hemicellulose, and lignin significantly affect the content of the carbon phase formed (Bledzki, Mamun, and Volk 2010; Z. Wang et al. 2018). Sources of biomass containing these compounds can be used as a carbon source in the synthesis of graphene.

Carbon materials from coconut shells have been studied for their capabilities as electrodes for supercapacitor applications. Yan et al. (2010) showed that activated carbon from coconut shells



has a high surface area and pore surface (Geng et al. 2013; Yang et al. 2010). Many researchers have carried out the synthesis of graphene from coconut shells (Baqiya et al. 2020). Wachid et al. 2014 used a simple, easy and inexpensive method. The method used is carbonization and activation. Dehydration, cellulose evaporation, lignin evaporation, and carbon purification were employed in the coconut shell during the carbonization process (Wachid et al. 2014). Coconut shells that have become charcoal will produce carbon by 57.11%, oxygen by 42.67%, and other materials by 0.23% (Mozammel, Masahiro, and Bhattacharya 2002). In another study, coconut shell was used as a carbon source to synthesize reduced graphene oxide (rGO) (Asih et al. 2019; Darminto et al. 2018; Kurniasari et al. 2017; Prasetya et al. 2015; Yang et al. 2010). Graphite can be classified into natural graphite and synthetic graphite, produced from the graphitization process (Simón et al. 2018). In general, rGO produced from coconut shell combustion has characteristics equivalent to natural graphite rGO (Somanathan et al. 2015). Nugraheni (2017) has reported the results of the characterization showing that coconut shell that has become charcoal will produce a reduced graphene oxide phase (Nugraheni et al. 2017). Sujiono et al. (2020) have reported the synthesis of graphene from coconut shells as a carbon source to replace natural graphite (Sujiono et al. 2020). Graphite powder from coconut shell was converted to graphene oxide (GO) using the Hummers method, and the resulting GO tends to form an rGO phase. This method involves several materials such as NaNO₃, H₂SO₄, KMNO4 (Sujiono et al. 2020). However, NaNO3 will produce toxic gases NO2 and N2O4, released during the oxidation process. Therefore, the opportunity to develop a coconut shellbased graphene synthesis method is still very much needed.

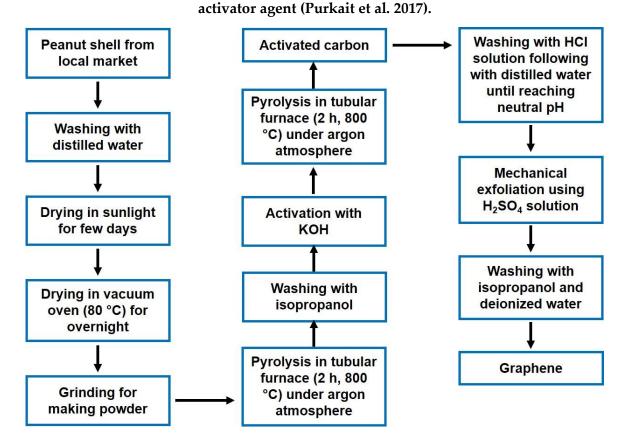
Graphene from peanut shell

Generally, peanut shell is only used for animal feed and building material. The addition of peanut shells to the plaster could reduce thermal conductivity, which enabled the mixture to be an insulator (Lamrani et al. 2017). Recently, peanut shells have significantly increased as a raw material in synthesizing carbon-based materials. Bay et al. (2020) prepared porous biomass charcoal from peanut shells without using chemical activator and protective gases during the pyrolysis process in the furnace. The as-produced porous biomass charcoal could adsorb Pb²⁺ and methylene blue in wastewater (Bai et al. 2020). Peanut shell was used as a raw material to



synthesize mesoporous few-layer graphene nanosheets via mechanical exfoliation to apply for binder-free supercapacitor electrodes. The synthesis procedures are represented in Figure 2. The as-produced graphene provided a high specific surface area (2070 m²/g) with a large pore volume (1.33 cm³/g). The capacitor capacity reached 186 F/g with the highest energy density, and the highest power density is 58.125 Wh/kg and 37.5 W/kg, respectively (Purkait et al. 2017). Mechanical exfoliation is a top-down technique that provides advantages such as simple methods and producing high-purity graphene without defects. However, this method is unaffordable for large-scale graphene synthesis (Bhuyan et al. 2016). Another disadvantage of using the mechanical exfoliation method is reducing graphene lateral size, which decreases its surface area due to the exerted force during the exfoliation process (Yi and Shen 2015).

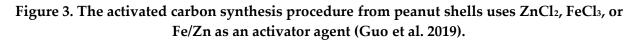
Figure 2. The activated carbon synthesis procedure from peanut shells with KOH as an



Activated carbon is a basis in graphene synthesizing, so that is important to observe its synthesis methods. Different activator agents would produce different structures of the as-produced activated carbon. The surface structure of activated carbon will affect the absorption capacity of the as-produced materials. Guo et al. (2018) has prepared activated carbon from



peanut shell using one-step synthesis with ZnCl₂, FeCl₃, and their mixture as the chemical activator. The synthesis procedures are explicitly described in Figure 4. The agent activator ratio of Fe/Zn was varied and contributed to the specific area surface of activated carbon. This material could be applied for electrodes because it has a large surface area (1482 m²/g) and abundant micropores (Guo et al. 2019). Zhang et al. (2015) has synthesized magnetic activated carbon from peanut shells via a one-step method using K₂CO₃ as activator agent and Fe₃O₄ as a magnetic additive agent, presented in Figure 5. The structures and the properties of the asproduced activated carbon depended on the activation temperature and activation time. The maximum specific surface area (1219 and 1236 m²/g) could be reached by the samples activated at 750 and 800 °C for 1.5 and 1 hour, respectively. Activation time facilitated the porosity development of the sample (micropore, mesopore, or macropore). (Zhang et al. 2015).



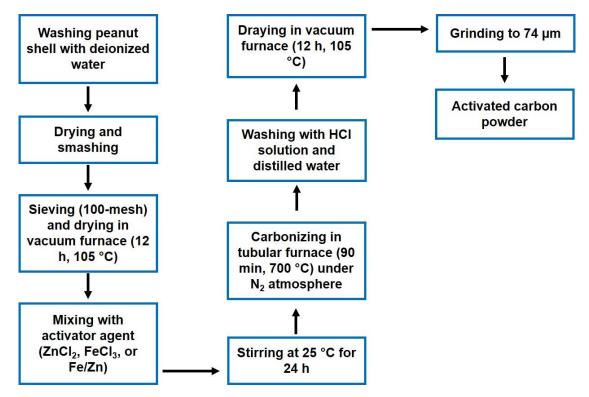
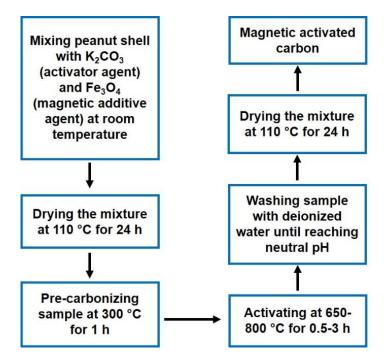




Figure 4. The magnetic-activated carbon synthesis procedure from peanut shells with K₂CO₃ as an activator agent (Zhang et al. 2015).



Conclusions

This brief review summarizes the utilization of biomass as a carbon source in graphene synthesis. The sources and synthesis methods of biomasses have been discussed for sustainable energy storages such as a capacitor, supercapacitor, and battery electrode. The as-produced graphene and its fullerenes depend on the biomass chemical and its synthesis method. All methods discussed above have several advantages, such as simple methods and low-cost production, enabling them to be used in large-scale production. However, advancement is still required in producing high-quality graphene.

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