PRODUCTION OF GRAPHENE BY COCONUT SHELL AS AN ELECTRODE PRIMARY BATTERY CELL

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Abstract: Coconut shells are a natural resource that contains a lot of carbon (C). The pyrolysis process can be used to create coconut shells. A single layer of carbon atoms that have undergone sp^2 hybridization to form a hexagonal, two-dimensional structure is known as Graphene. Graphene has excellent potential for battery manufacturing applications, supercapacitors, etc. Activated carbon and the pyrolisis method of producing Graphene were combined and heated to 600 ^oC for one hour. The graphene generated is assessed using an XRD, SEM-EDX, TEM, Raman, and conductometer. The results of the X-ray diffractogram analysis revealed that the peaks at $2\theta = 23{,}87^o$ and $44{,}5^o$ are not particularly sharp and slightly broadened. It means Graphene are well formed. SEM-EDX investigation reveals that the surface size and shape structure is smaller and thinner, a flat pile dominated by carbon atoms. The result of conductometer analysis shows the electrical conductivity of Graphene is quite good, but Graphene can still not control the movement of electrons. Graphene has layer distances between Graphene and Graphene layers are 3.3 Å (TEM data), with many Graphene layers being 0.85 (multi-layer) (Raman data).
Keywords: Coconut shells; Graphene; Characterization

Abstrak: Batok kelapa merupakan salah satu sumber daya alam yang mengandung banyak karbon (C). Proses pirolisis dapat digunakan untuk membuat batok kelapa. Satu lapisan atom karbon yang telah mengalami hibridisasi sp^2 untuk membentuk struktur dua dimensi heksagonal dikenal sebagai Grafena. Grafena memiliki potensi besar untuk aplikasi pembuatan baterai, superkapitasitor, dan sebagainya. Karbon aktif dengan metode pirolisis untuk memproduksi Grafena dipanaskan hingga 600 ^oC selama satu jam. Grafena yang dihasilkan dikarakterisasi menggunakan XRD, SEM-EDX, TEM, Raman, and Konductometer. Hasil analisis difraktogram sinar-X mengungkapkan bahwa puncak pada $2\theta = 24{,}22^o$ dan 44^o tidak terlalu tajam dan sedikit melebar. Ini berarti Grafena terbentuk dengan baik. Analisis SEM-EDX mengungkapkan bahwa ukuran permukaan dan struktur bentuk lebih kecil dan lebih tipis dan itu adalah tumpukan datar yang didominasi oleh atom karbon. Hasil analisis konduktometer menunjukkan konduktivitas listrik Grafena cukup baik, namun Grafena masih belum

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mampu mengontrol pergerakan elektron. Grafena memiliki jarak lapisan antara lapisan Grafena dan Grafena adalah 3,3 Å (data TEM) dengan jumlah lapisan Grafena adalah 0,85 (multilapisan) (data Raman).

Kata Kunci: Tempurung Kelapa; Grafena; Karakterisasi

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Introduction

Researchers today have created new processes for making carbon-based nanomaterials like Graphene by utilizing biomass resources that are environmentally benign (Mubarik et al., 2021). Creating Graphene from carbon waste recycling might have a significant positive impact on the environment and the economy. Graphene can be utilized as electrode material in batteries, sensors, transistors, solar cells, and conducting polymer composites because of its enormous surface area. Additionally, it is employed in various disciplines, including photodegradation, biodegradation, and clean energy technologies for water purification and sterilization (Somanathan et al., 2015). One of the semiconductor carbon materials, Graphene has a two-dimensional hexagonal structure made of a thin layer of sp^2 carbon joined by the van der walls force (Tiwari et al., 2016). As an active component of energy-storing processes, Graphene may be thought of as a single layer of carbon atoms that have undergone sp^2 hybridization to produce a two-dimensional hexagonal structure is called Graphene. Supercapacitors, battery production, and other uses for Graphene have a lot of potentials (Zhu et al., 2014). There may be new potential for manufacturing Graphene-based nanomaterials due to this straightforward, affordable, and renewable approach to producing Graphene (Singh, Bahadur, & Pal, 2017) to encourage the development of more competitive carbon-based products made from agricultural wastes and renewable resources.

The Hummer method is used to create Graphene, a naturally occurring, non-renewable material from mined graphite. The Hummer method for synthesizing Graphene based on graphite produces waste and only produces small amounts of the desired product (Siburian & Nakamura, 2012). The process of making Graphene from raw materials like coconut shells (Cocos nucifera L.) is undergoing extensive development (Siburian et al., 2018). Lignin, cellulose, and hemicellulose are its key components, and their thickness ranges from 3 to 8 mm. One adamant part of the fruit is the coconut shell. The carbonization method may turn coconut shells into charcoal or activated carbon. Graphene may be produced using coconut shell as a carbon source (Liyanage & Pieris, 2017). Supeno's (2019) research in carbon shell charcoal, carbon (C) makes up 87.1% of the total, making it the most common element. Therefore, it is hypothesized that coconut shells can produce carbon molecules, particularly "green carbon" products.

The goal of the current research was to create Graphene using coconut shells. The fundamental reason for using these sources is to offer environmentally friendly, cost-effective options for the mass manufacturing of carbon-based Graphene. XRD, SEM-EDX, TEM, Raman, and electrical conductivity were used to characterize synthetic Graphene.

Method

Materials

Coconut fruits of the *Cocos nucifera L.* kind were provided by a local farmer, and aquadest and ammonia were bought from the E-Merck Company. The tools used in this experiment include an analytical balance from Mettler with a 0.0001 g accuracy, a 200 mL porcelain dish, Pyrex glassware, a Cimarron hot plate, aluminium foil, a Furnace glass funnel, Whatman N0.42 filter paper, an oven, an XRD instrument from PaAlytical, a Phenom SEM-EDX, and a conductometer.

Charcoal Making

The primary material for generating Graphene on a big scale is coconut shell. The coconut shell is first dried in the sun before being pyrolyzed in the furnace for 5 hours at 600 ^{o}C in an oxygen-free environment to turn it into charcoal. The charcoal is then pulverized in a mortar when it has cooled. Then, it is sieved through a sieve with a mesh size of 100.

Synthesis of Graphene

The powdered activated carbon is added to the chip-shaped coconut shell charcoal. It is then ground for one hour at a temperature of 600 ^{o}C. Additionally, to differentiate between activated carbon and coconut shell charcoal, it is filtered using a sieve with a mesh size of 150. Distilled water is used to adequately clean coconut shells, which are then dried in an oven that is preheated to 70 °C. XRD, SEM-EDX, TEM, Raman and Conductometer are employed to provide further details about the item.

Result and Discussion

Synthesis of Graphene

In this work, the main battery electrodes were made using Graphene as their starting material by pyrolyzing coconut shell raw materials. The first step in the synthesis of Graphene is the sun-dried coconut shell, which is then subjected to pyrolisis at 600 ^{o}C for five hours to generate chip-shaped charcoal. The final step involves mixing charcoal chips with activated carbon, re-pyrolyzing them for a further hour, mashing them using a mortar, and sifting them through a 150 mesh screen to produce tiny sheets of Graphene known as nanosheets (Siburian et al., 2018; Siburian et al., 2021; Supeno et al., 2020).

Coconut Shell Amendment

The coconut (Cocos nucifera L.) is heated in the coconut shell (warmer) before being cracked till it breaks. Next, the water and coconut fruit is removed from the coconut shell. Once dried in the oven, the coconut fruit turns into a coconut shell. The dried coconut shell is then pyrolyzed at a temperature of 600 ^{o}C for 5 hours to acquire the changes in coconut weight, as shown below.

Table 1. Coconut Shells Amendment

A Total Coconut Fruits	Activity			Coconut Amendment (%)
	Before pyrolysis (g)	After pyrolysis		
		Charcoal Weight (g)	Ash Weight (g)	
8	196,4565 125,1140	40,37	3,51	12.55
	175,0489 141,7129	33,69	1,01	10.64

From Table 1, it can be seen that the result of the percentage change in the average coconut weight of 8 pyrolyzed coconut shells was 12.55% and 10.64%. This is because, at the time before pyrolysis, it still contains a lot of moisture content in it, so after the pyrolysis process, the coconut shell experiences a decrease in its scope and weight of the coconut shell.

Analysis of XRD (X-Ray Diffraction)

In this experiment, By using activated carbon as a reducing agent and heating the old coconut shell to 600 ^{o}C for an hour in the furnace, graphene from coconut shell charcoal was created. Graphene from coconut shell charcoal was analyzed for structure and phase using XRD, SEM-EDX, TEM, Raman, and conductometer.

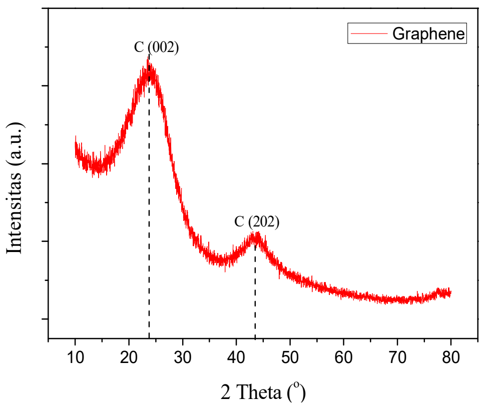

Figure. 1. XRD pattern of Graphene

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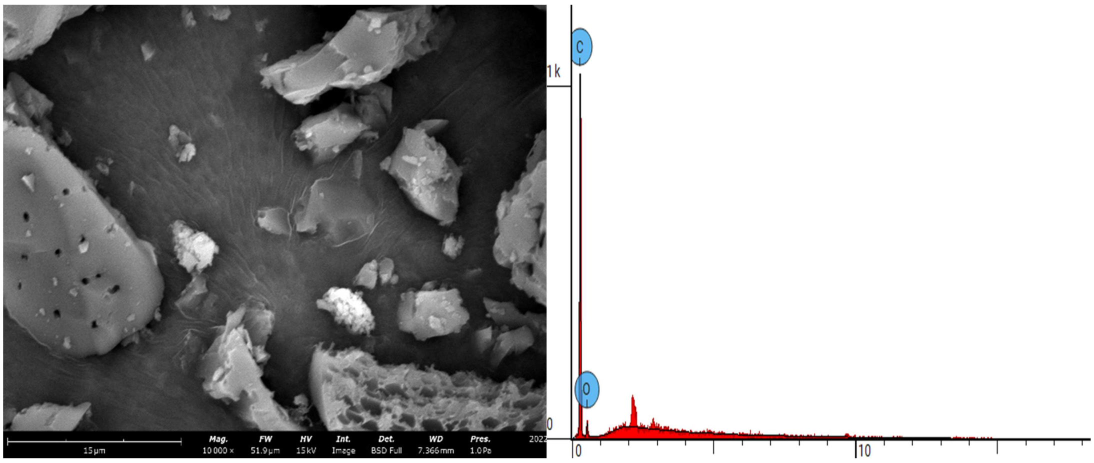
The pattern of diffraction from the characterization of the fraction and elemental content of the charcoal made from coconut shells is shown in **Figure 1**. Graphene's XRD diffractogram pattern reveals its amorphous nature, with two dominant peaks at $2\theta = 24.22^0$ and 44^0 (Kumar, Venkatesha, & Shabadi, 2018; Liu et al., 2013). These peaks typically indicate the presence of phase Graphene in the sample. Graphene's soft, dilated peaks show that the particles are nanoscale in size and layered atop the Graphene interlayer (Hu et al., 2017).

Analysis of SEM-EDX

Further analysis to prove that Graphene has been successfully synthesized, an SEM-EDX analysis was carried out. The image and EDX of the Graphene are shown in **Figure 2.**



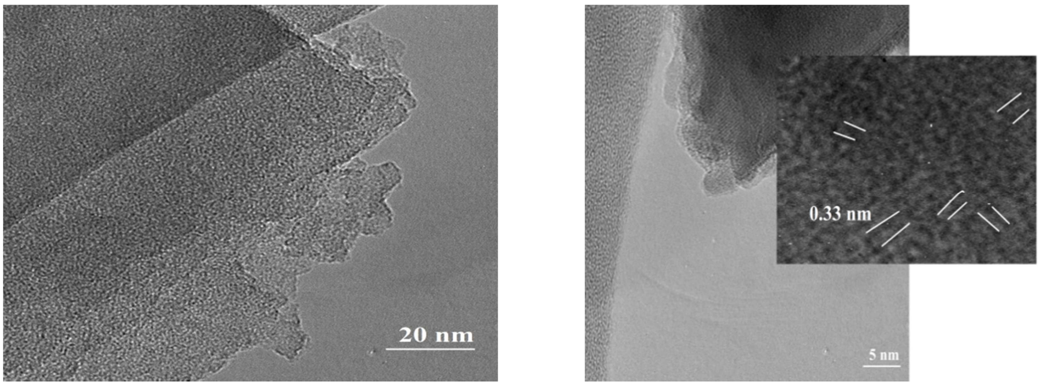
(a) (b)

Figure 2. (a) Morphology and (b) EDX of the Graphene by SEM-EDX

Morphology and element content between the layers of the Graphene coconut shell can be determined by using an SEM test coupled with EDX. The SEM figure in **Figure 2** shows Graphene's morphological surface and element. **Figure 2a** Graphene looks to have a thin, not thick, surface layer with tiny pores (Kucinkskis & Bajars, 2013). These graphene sheets are stacked on top of one another, creating a weak layer and uneven solids. This is consistent with the research of Qing (2020), which demonstrates that Graphene is a hexagonal pile with an extremely tight structure. The EDX spectra showed that the components contained in the Graphene of the coconut shell are in **Figure 2b. Figure 2b** shows that the Graphene of the coconut shell is dominated by the carbon atom (C). The intensity of the many C atoms (89.8 %). Graphene is not yet pure since it contains other elements besides carbon (C), such as oxygen (O). This is due to Graphene being composed of bonds of carbon atoms.

Analysis of TEM

More work has already been done to characterize the functionality, sustainability, and kinematics of various organic molecules on the Graphene sheets, for instance, carbon and other thin adatoms (Schäffel, Wilson, & Warner, 2011). Indeed, TEM of the dynamics of single-molecule adsorbent surface on Graphene has been researched extensively from the beginning of Graphene research, which is not surprising given that examining molecules does not require as high resolution as studying single atoms (Rummeli et al., 2019). One of the earliest studies described the structural rearrangement of hydrocarbon adsorbates on graphene to induce crystallization and produce layers of amorphous carbon by in situ heating beyond 1000 K (Westenfelder et al., 2011). **Figure 3** shows Graphene images from TEM analysis.



(a) (b)

Figure 3. (a) dan (b) Photo TEM of Graphene

Many organic compounds, including carbon and other light adatoms, have already been studied to define their usefulness, viability, and kinematics on graphene sheets. Indeed, TEM of the dynamics of single-molecule adsorbent surface on Graphene has been researched extensively from the beginning of Graphene research, which is not surprising given that examining molecules does not require as much high resolution as studying single atoms. Through in situ heating beyond 1000 K, one of the early investigations detailed the structural rearrangement of hydrocarbon adsorbates on Graphene to induce crystallization and generate amorphous carbon layers. **Figure 3b** depicts a thin layer and a flat sheet of Graphene, with a gap of 3.3 or 0.33 nm between the Graphene layers and may be characterized as a C (002). This is due to a Van Der Walls interlayer interaction inside the Graphene (Shen & Lua, 2013). The thick and dark component of the Graphene morphology on the nanoscale scale is an aggregation form of the Graphene sheet (multi-layer Graphene). However, the multi-layer structure is still between 5 and 20 nm (Meyer, 2019; Sahoo et al., 2018).

Analysis Raman Spectroscopy

Raman data were acquired after being shot with a laser, demonstrating the link between Raman intensity and wavelength displayed in Graphene form. The D band indicates disorder, which can be caused by flaws like voids or grain boundaries (Kaushal, Dhawan, & Singh, 2019; Surekha et al., 2020) and amorphous carbon species. The G band is slightly shifted from Graphene's (Tian & Zhang, 2016). **Figure 4** shows the D-band and G-band spectra of Graphene.

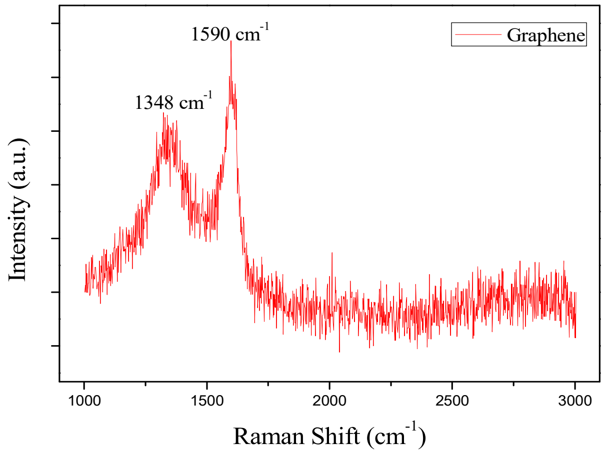


Figure 4. Graphene Raman spectra

Raman data were acquired after being shot with a laser, demonstrating the link between Raman intensity and wavelength displayed in Graphene form. The D band indicates disorder, which can be caused by flaws like voids or grain boundaries (Kaushal, Dhawan, & Singh, 2019; Surekha et al., 2020) and amorphous carbon species. The G band is slightly shifted from Graphene's (Tian & Zhang, 2016). There are two peaks on the Raman Graphene spectra at 1591 cm^{-1} and 1348 cm^{-1}, respectively. In other words, **Figure 4** demonstrates the material's presence of Graphene. The calculation findings suggest that Graphene contains 0.85 layers, respectively, indicating that the Graphene generated is multilayered. Uddin (2015) used the Hummer technique to synthesize Graphene and yield ID/IG ratios of 0.87, respectively.

Analysis of Electrical Conductivity

Analysis of the electrical conductivity of Graphene showed in **Figure 5.**

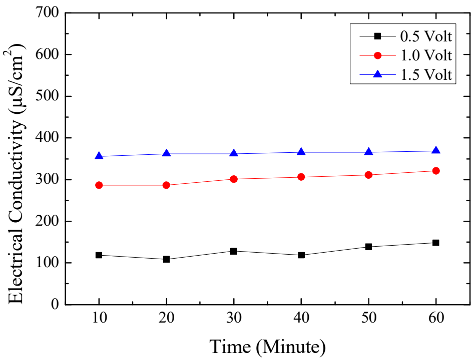


Figure 5. Electrical Conductivity of Graphene

The electrical conductivity analysis is carried out to determine the ability of Graphene materials to conduct electric current. Based on the results of electrical conductivity (**Figure 5**), the Graphene material has a relatively high electrical conductivity value. The value of conductivity that is up and down shows that the electron mobility possessed by Graphene is well controlled and has a rather good electrical conductivity value. Based on **Figure 5**, a graph of the electrical conductivity of Graphene can be obtained. This is because the Graphene has π electrons delocalized along the C = C bond that act as electric charge carriers. In addition, Graphene also has a larger surface area than graphite, so it can increase the electron mobility rate (Simanjuntak et al., 2021). In **Figure 5,** as Graphene is treated for longer and longer periods, the current produced by Graphene increases. This is due to Graphene's ability to manipulate electron mobility (Symons & Butler, 2018). Furthermore, Graphene cathode has better conductivity and a larger surface area. In other words, the Graphene cathode employed has better conductivity, a larger surface area, and a more stable structure than the graphite materials (Siburian et al., 2022).

Conclusion

The study's conclusions indicate that by employing activated carbon as a reducing agent and coconut shell charcoal as a starting material, Graphene can be produced. The XRD study findings for the characterization revealed that the diffraction peaks C (002) ($2\theta = 23.86^o$) and C ((200) ($2\theta = 44.5^o$) showed that Graphene had been successfully synthesized. SEM EDX revealed more minor results, thinner surface size and structural shape, reduced stacking in Graphene structure, and an abundance of Carbon C atoms predominating inside Graphene. When Graphene's electrical conductivity was analyzed, the results revealed an up-and-down graph brought on by unstable electron mobility. However, the material could still carry a respectable amount of current. Graphene can make electricity more stable and more significant in electrical conductivity.

Declaration of Competing Interest

The authors affirm that they have no known financial or interpersonal conflicts that would have seemed to have an impact on the research presented in this study.

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