

Correlation of surface area and I_D/I_G ratio of coconut shell graphene carbon synthesized using Hummers method: A review

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ABSTRACT

Graphene from coconut shells has been widely developed by previous studies, graphene has excellent conductivity properties that are widely applied in electronics to sensors. Coconut shells are chosen as raw materials for making graphene because of their high carbon content, and are also environmentally friendly. Characteristics can be identified by the Raman curve to determine the peak values of D and G where Peak D is the defective part of the graphene structure and Peak G is the inner part of graphene in crystalline form. This article discusses the I_D/I_G values synthesized from coconut shell materials. Based on the last 5 years of journals, this review discusses the surface area and I_D/I_G values of the 25 journals that I have read, the most widely used journal is the Hummers method, because this method is easy to understand to determine the comparative value of I_D/I_G , surface area, and correlation between the two parameters. The resulting nanocomposites were analyzed using Raman Spectroscopy to determine the I_D/I_G value and BET Analysis to measure its surface area. The results obtained showed that the I_D/I_G value was in the range of 0.85 to 1.15, while the measured surface area ranged from 40 to 120 m²/g. Statistical analysis shows that the Pearson correlation value between the surface area and I_D/I_G value is -0.028, indicating a very weak relationship and almost no linear correlation between these two variables. The p-value of 0.932 also shows that this correlation is not statistically significant. This indicates that increasing or decreasing the surface area of graphene-magnetite nanocomposites has no direct effect on the I_D/I_G ratio.

Keywords: Coconut shell; graphene; Hummers method; Raman spectroscopy; surface area

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INTRODUCTION

Graphene group is a two-dimensional carbon structure consisting of carbon atoms arranged in a hexagonal honeycomb pattern [1, 2]. Graphene is a material that has a two-dimensional (2D) crystal structure with a hexagonal lattice resembling a honeycomb [3]. The two-dimensional structure of graphene and its derivatives, such as Graphene Oxide (GO) and reduced Graphene Oxide (rGO), provides a high surface area, flexibility, excellent conductivity, as well as good thermal stability and dispersion [4]. The structure of graphene is planar, while graphene oxide is curved due to the presence of oxygen groups in the form of carboxyl and carbonyl [5]. There are several methods for synthesizing graphene oxide, one

of which is the Hummer method, or chemical oxidation [6].

There are two methods for synthesizing graphene: top-down and bottom-up [7–9]. Four well-known graphene synthesis methods are chemically derived, graphite exfoliation, chemical vapor deposition, and organic synthesis [10]. Graphene oxide is synthesized using the Hummer method [4, 11, 12]. Lignin-based materials can be precursors to various carbon materials, including carbon fiber, activated carbon, graphite, and graphene. The resulting carbon materials are primarily used as capacitors and electrocatalyst supports [13]. Synthesis of graphene oxide from coconut shells [6]. Synthesis and Characterization of Graphene Oxide (GO) from Natural Coconut Shell Materials [3].

Graphene and graphene oxide are characterized using FTIR spectroscopy [14]. Characterizing graphene aims to understand its size, number of layers, defects, and associated functional groups [15]. Graphene characterization involves understanding its structure, properties, and quality through various techniques. Raman spectroscopy is commonly used to assess physical and chemical properties, providing insight into the quality of graphene [16]. There are 2 ways they use characterization, namely Characterization Using XRD and Characterization Using Raman Spectroscopy [17]. Characterization Using XRD is a technique used to determine the crystal structure of a material [18]. Raman spectroscopy is a technique that is very suitable for use in research and its workings are very easy to understand [19]. Graphene is commonly used in electrode applications in electrical and optical devices [20].

To observe the relationship between the quality or level of irregularity of graphene material and its surface area. This research is limited only to the process of synthesizing graphene from coconut shell charcoal using the modified Hummers method. Focusing on the study of the structure and physical properties of graphene, such as electrical conductivity, mechanical strength, and interaction with the environment [9]. Focusing on graphene applications in energy storage (supercapacitors, batteries) and electronics (transistors, sensors) [21]. The weaknesses of graphene include defects that hinder its performance in electronic applications [15].

LITERATURE REVIEW

Characteristics of Coconut Shell Graphene

Coconut shells are a widely available agricultural waste [22]. Coconut shells are known for their hardness, which is due to their high lignin and cellulose content [23]. Their porous structure and energy storage capacity contribute to activated carbon electrodes [24]. Coconut shells are composed of cellulose,

lignin, pentosans, and ash, making them an ideal material for various applications ranging from fuels to electronic materials [25]. Physical properties such as pH, apparent density, bulk density, and particle size of carbonized and uncarbonized coconut shell nanoparticles [26, 27] have been studied.

Table 1. Coconut shell composition [11, 22].

Analysis	Coconut Shell
C	41.8 – 42.3
O	51.5 – 52.0
H	4.1 – 4.6
S	0.5 – 0.4
N	2.1 – 0.6

Table 2. Physical and mechanical properties of coconut shell [28, 29].

Parameter	Value
Maximum size (mm)	12.5
Moisture content (%)	4.20
Water absorption capacity (24 hours)	24.0
Crushing value (%)	2.58
Mass density (kg/m ³)	650
Fineness modulus	6.26
Shell thickness (mm)	2 – 8

Parameters include density, color, electrical conductivity, optical transparency, tensile modulus, and lattice parameters [30–37]. Its density has a value of 62 (kg/m³) [30]. Graphene is dark brown in color [36]. It has a thermal conductivity of 15 (mg/cm³) and an electrical conductivity of 2.833 (S/cm) [35, 37]. In addition, graphene also has an optical transparency of 97.7 (%) [32]. The tensile modulus value is 1.001 (Tpa) and the tensile strain value is ~1.3 (%), the compression strain is ~0.7 [31, 33]. It has a lattice parameter value of 2.450 (a) [34].

RESEARCH METHODS

Surface Area Determination The specific fiber surface area and fiber porosity volume were tested using the BET (Brunauer-Emmet-Teller) surface area test [38–40]. This tool works by utilizing the adsorption mechanism of gases, typically nitrogen, argon, and helium, on the surface of a solid material to be

characterized at a constant temperature, usually the boiling point of the gas. The physical characteristics of graphene, in the form of specific surface area (m^2/g), can be determined

through nitrogen adsorption-desorption isotherm analysis at the nitrogen boiling point (77K) using a BET tool [10].

Table 3. Physical and mechanical properties of graphene.

Parameter	Value	Ref.
Density (kg/m^3)	62	[30]
Color	Dark brown	[36]
Thermal conductivity (mg/cm^3)	15	[37]
Electrical conductivity (S/cm)	2.833	[35]
Optical transparency (%)	97.7	[32]
Tensile modulus (Tpa)	1.001	[31]
Tensile strain (%)	~1.3	[33]
Lattice parameter, a	2.45	[34]

BET is very effective for measuring the specific surface area of various types of materials, including catalysts, metal powders, clay, activated carbon, and other porous materials [41]. BET provides information on the total surface area available in a material, including both the external surface and the internal surface within the pores [38]. The BET method can be used to analyze small samples, making it suitable for rare or expensive materials [38]. BET is more accurate for monolayer adsorption and less accurate for materials with complex multilayer adsorption [42].

Raman Characterization

Raman spectroscopy is a spectroscopic technique used to observe vibrational, rotational, and other low-frequency modes in a system [43]. Spectroscopic methods can be based on emission, absorption, fluorescence, or scattering phenomena [44]. This technique can also be applied to detect organized and disordered crystal structures and identify the monolayer or multilayer nature of GO samples [45]. The G peak has almost the same intensity as the D peak and is larger than the D peak, indicating high material quality because the D peak can represent defects in the material [3]. There is a Raman spectrophotometer with an ID/IG ratio that does not show defects at 700°C [46].

RESULTS AND DISCUSSION

From these data (Figure 1 and Table 1), it can be concluded that the surface area is higher than the I_D/I_G value. In fact, the surface area value can be directly proportional to the I_D/I_G ratio.

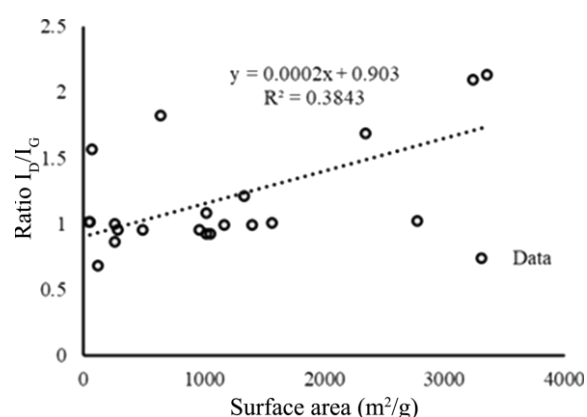


Figure 1. Raman spectrum curve.

AC_H coconut shell and AC_HR coconut shell [47]. AC_HR coconut shell is suitable as an electrode material for supercapacitors. Research shows that activated carbon from coconut shells processed using the standard Hummers method has a surface area of $496.03 \text{ m}^2/\text{g}$ and a density of $0.948 \text{ g}/\text{cm}^3$.

Graphite material produced from coconut shells has the following specifications: a specific capacity of $14.1 \text{ mAh}/\text{g}$ and an ID/IG value of 3.8 [4]. The surface area of $14.1 \text{ mAh}/\text{g}$ is relatively low compared to other energy storage materials [4]. This indicates that

graphite may have limitations in terms of energy storage capacity per unit mass. This may impact its use in applications requiring high capacity [4]. The ID/IG ratio of 3.8 does not seem to correspond to a commonly used value, as the ID/IG ratio is usually expressed in the

range of 0 to 1, indicating the ratio between stored and returned charges [4]. Although graphite from coconut shells exhibits a low specific capacity, it may still have benefits in applications requiring good electrical conductivity or low-cost materials [4].

Table 4. List of surface area data and ID/IG ratios in several journal references.

Material	Method	Surface area (m ² /g)	I _D /I _G	Structure	Ref
Coconut shell AC_H	Modified	496.03	0.948	Graphene	[47]
Coconut shell AC_HR	Hummers	642.50	1.817		
Coconut shell AC5	Hummers	14.1	3.8	Graphite	[4]
Coconut shell carbonization 700°C	Hummers	261.584	0.856	Reduced Graphene	[46]
Coconut shell carbonization 900°C		261.584	0.006	oxide (R-GO)	
Coconut shell CSK2	Hummers	1567	1.00	Graphene	[48]
Coconut shell EDAC 0.5	Chemical oxide	3359	2.13	Graphene	[49]
Coconut shell EDAC 1		2345	1.68		
Coconut shell GC400	Green synthesis	122.8	0.68	Graphene	[2]
Coconut shell GC1000		77.4	1.56		
Coconut shell TWA-HC	Green synthesis	~966.4	0.95	Graphene	[50]
Coconut shell TWA-WC		~1057.8	0.92		
Coconut shell PC	Electrocatalyst	1028.3	1.08	Oxygen reduction reaction (ORR)	[51]
Coconut shell NF-PC		1028.3	0.92		
Coconut shell ACCS	Activation	2782.97	1.02	Graphene	[52]
Coconut shell CAC		1402.08	0.99		
Coconut shell 1300-CC	Simple	5.71	0.46	Graphene	[53]
Coconut shell 1300-Ni-GCM		51.61	1.01		
Coconut shell ANi-GCM		59.02	1.01		

Characteristics of carbon materials produced from coconut shell carbonization at two different temperatures: 700°C and 900°C [46]. Both materials have the same specific capacity, namely 261,584 mAh/g, indicating that the energy storage capability of the materials remains consistent despite the different carbonization temperatures [46]. The material carbonized at 700°C has an I_D/I_G ratio of 0.856, while the material carbonized at 900°C increases to 0.996 [46]. This increase in I_D/I_G indicates that carbon material from coconut shells processed at higher temperatures has a better ability to store and return charges efficiently [46]. This material has the potential to be better in energy storage applications such as supercapacitors or batteries [46].

Graphene material produced from coconut shells through the Hummers process, [48]. a specific capacity of 1567 mAh/g and an I_D/I_G ratio of 1.00 [48]. The surface area of 1567 mAh/g indicates that this graphene material has a very high energy storage capacity per unit mass, making it very promising for energy storage applications, such as in batteries or supercapacitors [48]. The I_D/I_G ratio reaching a value of 1.00 indicates that almost all the charge is stored during charging graphene material produced from coconut shells through the Hummers process [48]. a specific capacity of 1567 mAh/g and an I_D/I_G ratio of 1.00 [48]. The surface area of 1567 mAh/g indicates that this graphene material has a very high energy storage capacity per unit mass, making it very

promising for energy storage applications, such as in batteries or supercapacitors [48]. The ID/IG ratio reaching a value of 1.00 indicates that almost all the charge stored during charging can be recovered during discharging, reflecting highly efficient performance in the energy storage cycle [48]. The Hummers process used to produce graphene from coconut shells appears to be successful in producing a material with high performance in terms of energy storage capacity and efficiency [48].

The very high surface area of EDAC 0.5, namely 3359 mAh/g, indicates a very large energy storage capacity compared to other graphene materials [49]. The I_D/I_G ratio of 2.13 also indicates that this material is capable of storing energy not only with a high capacity [51]. EDAC 1 coconut shell, despite having a slightly lower surface area of 2345 mAh/g, also exhibits a good ID/IG ratio of 1.68 [51]. EDAC 0.5 shell offers a higher storage capacity, and EDAC 1 shell still provides excellent performance [51].

Characteristics of two types of graphene materials produced from coconut shells using a green synthesis method [2]. The surface area is 122.8 mAh/g with an I_D/I_G ratio of 0.68 [2]. For GC1000 coconut shell, the surface area is 77.4 mAh/g with an I_D/I_G ratio of 1.56 [2]. The GC400 graphene material exhibits a relatively high surface area of 122.8 mAh/g and an I_D/I_G ratio of 0.68. This surface area reflects its large energy storage capacity [2]. The GC1000 coconut shell has a lower Area capacity of 77.4 mAh/g, but shows a higher I_D/I_G Ratio of 1.56. This indicates that this material stores energy at a lower capacity [2]. GC400 may be more suitable for applications requiring higher storage capacity, while GC1000 may be preferred in applications where high charge return efficiency is more important [2].

Characteristics of two types of graphene materials produced from coconut shells with the following specifications: for TWA-HC coconut shells and TWA-WC coconut shells. [50]. TWA-HC coconut shells, the surface area is about 966.4 mAh/g with an I_D/I_G ratio of 0.95 [50]. For TWA-WC coconut shells, the surface

area is about 1057.8 mAh/g with an I_D/I_G ratio of 0.92 [50]. TWA-HC graphene material has an excellent surface area of about 966.4 mAh/g, indicating a fairly high energy storage capability [50]. Coconut shell TWA-WC has a slightly higher surface area of about 1057.8 mAh/g, and a slightly lower I_D/I_G ratio of 0.92 [50]. TWA-WC offers a slightly higher storage capacity than TWA-HC [50]. TWA-HC has a slightly better I_D/I_G ratio. The choice between the two will depend on the application priority [50].

Characteristics of two types of coconut shell materials for electrocatalyst applications [51]. Coconut shell PC, Surface area is 1028.3 mAh/g with an I_D/I_G ratio of 1.08 [51]. Coconut shell NF-PC has the same surface area of 1028.3 mAh/g, but with an I_D/I_G ratio of 0.92 [51]. Coconut shell PC material has an energy storage area of 1028.3 mAh/g and an I_D/I_G ratio of 1.08, indicating high performance in energy storage and efficient material degradation [51]. Despite having the same specific capacity, coconut shell NF-PC has an I_D/I_G ratio of 0.92, which is lower than PC, indicating a charge return efficiency that is not as good as PC [51].

Characteristics of two types of graphene materials produced from coconut shells through an activation process [54]. ACCS coconut shells have a high specific capacity of 2782.97 mAh/g, with an ID/IG ratio of 1.02 [55]. ACCS materials have a large energy storage capacity and a charge return efficiency above 100%, indicating superior performance in energy storage [52]. CAC coconut shells exhibit the characteristics of two types of graphene materials produced from coconut shells through an activation process [52]. ACCS coconut shells have a high specific capacity of 2782.97 mAh/g, with an ID/IG ratio of 1.02 [52]. ACCS materials have a large energy storage capacity and a charge return efficiency above 100%, indicating superior performance in energy storage [52]. Coconut shell CAC showed a lower surface area of 1402.08 mAh/g, and an ID/IG ratio of 0.99 [52]. The energy storage capacity of CAC was lower compared to ACCS [52].

CONCLUSION

Graphene is a two-dimensional substance consisting of carbon atoms arranged in a hexagonal pattern with various properties such as high electrical coefficient, high flexibility, and high stability of carbon atoms arranged in a hexagonal pattern with various properties such as high electrical coefficient, high flexibility, and high stability. Its derivatives are similar to graphene oxide (GO), can be synthesized using oxidation such as Hummer. Then characterized using methods such as FTIR, XRD, and Raman spectroscopy to understand its structure and quality. Such as the Hummer Method. Then characterized using techniques such as FTIR, XRD, and Raman spectroscopy to understand its structure and quality. Because of its hard and porous nature, coconut shells, rich in lignin, and cellulose, have been used as raw materials in the production of graphene, which has led to the development of high potential for efficient and large-scale energy harvesting applications at the edge of the earth. Utilizing BET technology and ID/IG analysis in Raman spectroscopy provides insight into graphene's capabilities in energy harvesting, making it a highly suitable material for high-tech devices such as supercapacitors and batteries.

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