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Synthesis and Characteristics Of Graphene Oxide (GO) from the Skin Shell: Candlenut, Palm Oil and Coconut Hybrids

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Abstract: In this work, graphene oxide from the skin shell, such as candlenut, palm oil, and coconut hybrids, was successfully synthesized over hydrothermal technique and utilized as employing them as a potent adsorbent for removing synthetic methylene blue (MB) dye. Moreover, the adsorption experiments were performed with the initial MB dye concentration and contact time variation. The investigation revealed a maximum adsorption efficiency of 99.5 with an adsorption capacity of 6,220 mg/g at a contact time of 45 min. A second-order kinetic equation best represented the kinetics of the sorption process. Thermodynamic analyses provided evidence that the adsorption of methylene blue onto the material was thermodynamically viable and occurred spontaneously. The scientific examination of plausible adsorption mechanisms for methylene blue onto GO surfaces involved a thorough analysis utilizing Fourier transform infrared spectroscopy, X-ray diffraction spectroscopy, and scanning electron microscopy. Our finding exhibited that the GO from palm shell had the potential as a cost-efficient and promising sorbent for treating methylene blue-contaminated wastewater; this is attributed to their remarkable efficiency.

Keywords: Palm shell; coconut shell hybrids; candlenut shell; graphene oxide; adsorption; methylene blue.

1. Introduction

Biomass waste derived from agricultural activities, such as plant waste products, is promising for graphene oxide production. Candlenut shells, palm oil shells, and coconut hybrid shells all contain high carbon composition and include agro-industrial waste, an agricultural product [1–3]. Due to this, the agro-industrial product contains high shell waste, has a hard structure, and is difficult to decompose, so they have the potential to become waste if not further processed [4,5]. It is known that the waste contains carbon content for candlenut shells at 53.6%, palm oil at 20.5%, and hybrid coconut at 18.29% [6]. In addition, the high carbon content makes the shell potentially used as a precursor for graphene oxide production.

Graphene is a two-dimensional material that has gained significant attention due to its remarkable properties, such as high mechanical strength, excellent thermal and electrical conductivity, and large surface area [7,8]. It is a form of carbon arranged in a honeycomb lattice structure, making it highly desirable for various applications in electronics, energy, sensors, supercapacitors, and biomedical fields [9–11]. In recent years, researchers have been exploring sustainable and cost-effective methods to produce graphene. One such approach involves utilizing biomass waste materials as a precursor for graphene synthesis [12,13]. Biomass waste, such as agricultural residues and forestry waste, is a rich carbon source and can be easily converted into graphene through a simple and green process. Using biomass waste as a source of graphene addresses environmental sustainability issues and provides an economical solution for producing graphene [14–16]. Moreover, using biomass waste for graphene production also has the potential to reduce the amount of waste generated by the agriculture and forestry industries.

The utilization of graphene derived from biomass waste for dye adsorption not only offers a sustainable and cost-effective solution for wastewater treatment but also contributes to the circular economy by valorizing biomass waste [17–19]. Furthermore, this research has the potential to reduce the environmental impact of dye pollutants and promote cleaner and healthier ecosystems. The exploration of graphene derived from biomass waste as an adsorbent for dye removal represents a promising approach in the field of environmental remediation [20]. By leveraging the unique properties of graphene and addressing the challenges associated with dye pollutants and waste management, this research can pave the way for sustainable and efficient wastewater treatment processes [21].

This study focused on synthesizing graphene oxide from agro-industrial waste such as candlenut shells, palm oil shells, and coconut hybrid shells to remove methylene blue. The first step was to prepare the graphene oxide from agro-industrial waste for adsorbent production and evaluate the generated absorbents' morphological structural, crystal phase, and functional group characteristics. Moreover, in this work, we studied the effect of contact time adsorbent over methylene blue.

2. Materials and Methods

2.1. Sample collection and preparation.

Sample preparation (candlenut shell, hybrid coconut, and oil palm) was performed in several stages: washing, drying, burning, and grinding. Further samples were cleaned and dried in the sun for 3-4 days. The sample was then put into the media for the combustion process. This burning turns the shells into charcoal and removes the water content. The combustion process was carried out until all samples stopped emitting smoke. This indicates that the sample mentioned has become charcoal. The resulting charcoal was then pounded and mashed to a size of 200 mesh.

2.2. Preparation of graphene oxide.

The GO was synthesized using the modified Hummers methods [22]. Summarily, powder of candlenut shell, hybrid coconut, and oil palm of 2.0 g and 4.0 g $NaNO_3$ were added into a sulfuric acid (H_2SO_4) solution of 100 mL for each glass beaker while stirring in an ice bath for 4 h within high speed. Further, 8.0 g of potassium permanganate ($KMnO_4$) was slowly appended to each glass beaker while stirring in an ice bath at a temperature of 25 C for 20 h.

Then 200 mL of distilled water was added gradually to the graphite solution of candlenut shells, hybrid coconut, and oil palm and stirred for a few minutes until each solution was homogeneous. Moreover, 15 mL of hydrogen peroxide (H_2O_2) was added gradually while stirring, and a concentrated hydrochloric acid (HCl) solution of 10 mL was added. Subsequently, each graphite oxide solution was washed with distilled water repeatedly until the pH was neutral. In the last step, the sample was dried in an oven at 105°C for 12 h to remove the water content.

2.3. Structural characterization.

GO from each sample was characterized using a scanning electron microscope (SEM; ZEISS EVO MA 10, Germany) to notice morphological structural properties. X-ray diffractometer (XRD; X Pert Pro, PANalytical, Almelo, The Netherlands) was performed to sight the crystal phase of each sample. Besides, a Fourier transform infrared spectrometer (FTIR; JascoFTIR-6300, JASCO, Japan) was performed to confirm the functional group.

2.4. Methylene blue adsorption experiments.

The performance test of GO from the palm shell was conducted in a reactor system. Previously, the three adsorbents, namely candlenut shell, palm oil shell, and coconut hybrid shell, were first tested for their adsorption capacity in methylene blue solution. The mass of adsorben used during the test was 0.10 g. After that, the absorbance was measured at a maximum wavelength of 665 nm. Moreover, the parameters examined include contact times. In the contact time test, the time used was 15, 30, 45, and 60 min with the same composition.

3. Results and Discussion

3.1. XDR analysis.

Figure 1 exhibits the X-ray diffraction (XRD) pattern of rGO from candlenut, palm oil, and coconut hybrid shells. The diffraction pattern obtained from XRD analysis of the produced three samples reveals a distinct and tightly defined diffraction peak corresponding to the (002) crystallographic plane [23,24].

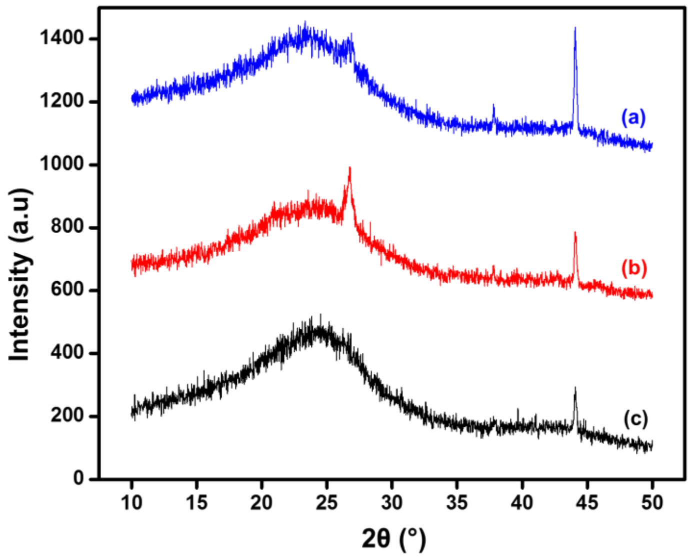

Figure 1. XRD pattern of the GO from **(a)** candlenut shell; **(b)** palm shell; **(c)** coconut hybrid shell.

This peak is observed at an approximate angle of 44.04 degrees. Such a sharp and narrow diffraction peak suggests that the rGO from three samples possesses a remarkably high degree of crystallinity. Moreover, two other characteristic diffraction peaks for each sample can be observed at 11.89 and 27.02 (Figure 1a), at 12.39 and 26.58 (Figure 1b), and 11.37 and 26.80 (Figure 1c) arising from the (100), (101), and (004) planes of graphene sheets, respectively. Sharp peaks at 26.60 and 44.04 in spectrum B (Figure 1b) described typical peaks of GO as reported in previous research (Banerjee et al., 2016) and thereby pointed to the successful synthesis of rGO from the sample and absence of the typical peaks at 2θ = 26.6°of GO in spectra A and C (Figures 1a,c). The modified Hummer method, employed for synthesizing graphene oxide (GO), involves incorporating hydroxyl groups between individual graphite layers, resulting in the formation of GO.

3.2. SEM analysis.

In this SEM discussion, we examine the morphological characteristics of graphene oxide derived from three different precursors: candlenut shell, palm oil shell, and coconut hybrid shell (Figure 2). SEM images recorded at high resolution unmistakably reveal the planar characteristics of each sample [23,25].

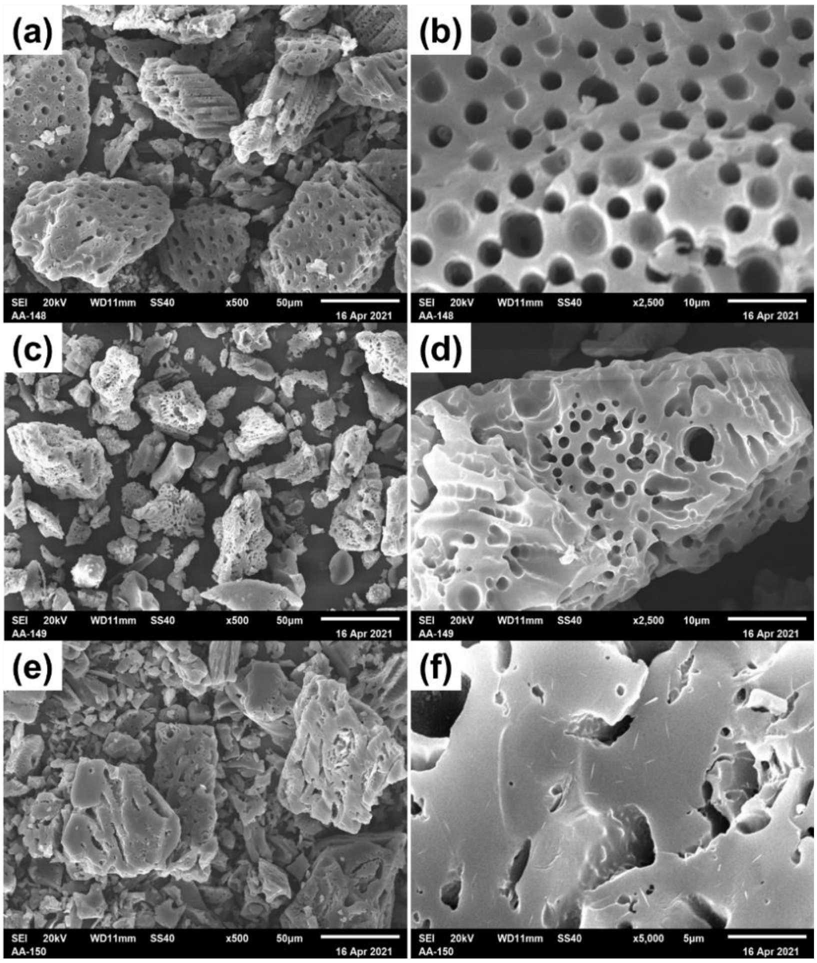

Figure 2. SEM image from **(a,b)** candlenut shell; **(c,d)** palm shell; **(e,f)** coconut hybrids shell.

The SEM images reveal intriguing differences in the morphologies of the graphene oxide samples obtained from these three precursors. Graphene oxide derived from candlenut shell (Figures 2a,b) exhibits a relatively uniform and smooth surface with minimal defects, indicating a successful conversion process and a high degree of graphene oxide exfoliation. On the other hand, graphene oxide derived from palm oil shells (Figures 2c,d) displays a more irregular surface with some aggregated structures. This observation suggests that the synthesis method or precursor composition might have influenced the exfoliation efficiency and overall quality of the graphene oxide obtained from palm oil shells. Interestingly, graphene oxide from coconut hybrid shell (Figures 2e,f) exhibits a distinct surface morphology characterized by a combination of layered and crumpled structures [26]. The presence of these unique features suggests that the coconut hybrid shell's composition and structure have a significant impact on the resulting graphene oxide's microstructure.

Furthermore, the SEM analysis also allows us to estimate the average particle sizes of the graphene oxide sheets derived from each precursor. Candlenut shell-derived graphene oxide shows a relatively uniform size distribution [27]. In contrast, palm oil shell-derived graphene oxide exhibits a wider range of particle sizes due to the presence of aggregates. Coconut hybrid shell-derived graphene oxide displays a mixture of large and small particles, indicating a more complex exfoliation process.

3.3. FTIR analysis.

Fourier transform infrared spectroscopy (FTIR) analysis was conducted on three different samples of graphene oxide derived from a candlenut shell, palm oil shell, and coconut hybrid shell. The FTIR spectrum for each sample revealed distinct characteristic peaks, providing valuable insights into their chemical composition and functional groups. The FTIR spectrum of graphene oxide from the candlenut shell (Figure 3a) exhibited prominent peaks at approximately 3425 cm^{-1}, indicating the presence of hydroxyl groups (-OH) associated with water molecules and residual alcohol groups from the oxidation process [28]. The peak observed at 1710 cm^{-1} corresponds to carbonyl groups (C=O), which can be attributed to carboxylic acid functionalities formed during the oxidation of the carbonaceous material. Additionally, the peak at 1050 cm^{-1} indicates the presence of epoxy groups (C-O-C), further confirming the successful formation of graphene oxide from the candlenut shell.

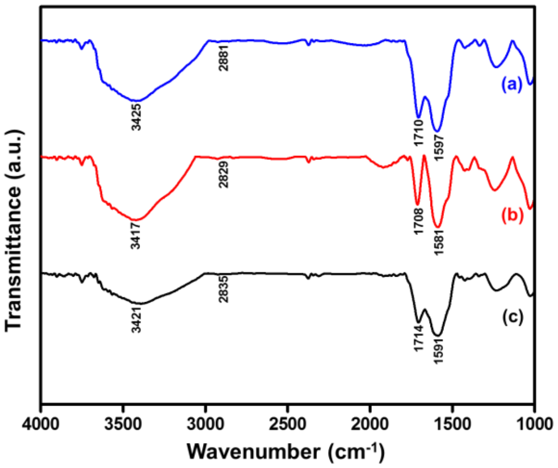

Figure 3. FTIR spectrum of **(a)** candlenut shell; **(b)** palm shell; **(c)** coconut hybrid shell.

In the FTIR spectrum of graphene oxide from palm oil shell (Figure 3b), a similar peak at 3417 cm^{-1} was observed, suggesting the presence of hydroxyl groups, much like in the previous sample. The intense peak at 1708 cm^{-1} corresponds to the stretching vibration of conjugated carbonyl groups (C=O), indicating the formation of carbonyl functionalities during the oxidation process [29]. Moreover, the peak at 1200 cm^{-1} confirms the existence of ether groups (C-O-C), signifying successful graphene oxide synthesis from palm oil shells [30]. In the case of graphene oxide from a coconut hybrid shell (Figure 3c), the FTIR spectrum exhibited peaks at 3421 cm^{-1} and 1714 cm^{-1}, corresponding to hydroxyl and carbonyl groups, respectively, similar to the other two samples. Notably, a peak at 1100 cm^{-1} indicates the presence of ester groups (C-O-C), characteristic of the graphene oxide formed from coconut hybrids' shells [31,32].

3.4. Adsorption study.

Before testing the parameters, the three samples, namely palm shell, candlenut shell, and hybrid coconut shells, underwent an adsorption capacity test against methylene blue. Table 1 reveals that all three samples significantly diminished the concentration of methylene blue in the solution, signifying their substantial adsorption capabilities for the dye. Notably, the palm kernel shell sample exhibited a higher adsorption capacity compared to the other samples. The disparities in adsorption capacities among the three samples may be attributed to their structure, morphology, and chemical composition differences. Therefore, based on these findings, the palm shell sample is chosen for further parameter testing.

Table 1. Adsorption capacity of the three samples.

GO sample	Abs	EA (%)	qe (mg/g)
candlenut shell	0.2408	97.01	6.06
palm shell	0.1424	99.88	6.24
coconut hybrids shell	0.5413	88.22	5.51

The adsorption process of dyes is greatly influenced by the contact time of the adsorbent against the MB solution, as it alters the surface charge of the adsorbent and the ionization behavior of both the adsorbent and the dye. The effects of contact time on the removal of MB using GO from the palm shell were also presented in Figure 4.

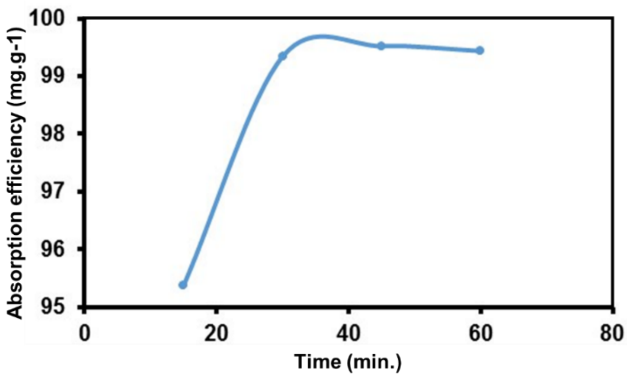

Figure 4. The contact time effect of GO from palm shell for removal of methylene blue.

Table 2. The isotherms and kinetic parameters for MB adsorption are used using GO from the palm shell.

		Sample	Graphene oxide
Adsorption isotherms	Freundlich	K_F (L/mg) n R^2	49.929 0.069 0.9248
	Langmuir	K_L (L/mg) q_{max} (mg/g) R^2	2.8×10^{-3} 0.3223 0.9727
Adsorption kinetics	Pseudo-first order	qe (mg/g) K_1(g/mg.min^{-1}) R^2	0.374 7.71×10^{-2} 0.7406
	Pseudo-second order	qe (mg/g) K_2(g/mg.min^{-1}) R^2	6.293 2.41×10^{-1} 0.9999

To evaluate the adsorption mechanisms of MB in advance, pseudo-first and pseudo-second-order models were employed to investigate the experimental data [33,34]. Table 1 shows that the correlation coefficients of both mentioned models from GO-derived palm shell adsorbent follow the second pseudo-order with a value of 0.9999 and near 1.0, meaning that 99% of the particle interactions follow the pseudo-second-order models. It can be deduced that the adsorption process likely includes both physical and chemical adsorption mechanisms.

4. Conclusions

In this work, we focused on examining the adsorption of methylene blue (MB) dye in water solutions by utilizing a GO from a palm shell. The GO from the palm shell was effectively produced through a hydrothermal technique. The GO derived from palm shell exhibited remarkable removal efficiency, achieving approximately 99.5% for MB. Our findings revealed that an optimum contact time of 45 min resulted in a higher removal rate of MB dye from the solution. Equilibrium analysis indicated a good fit with the Langmuir and Freundlich adsorption isotherm models, suggesting multilayer dye adsorption on the adsorbent's surface. Regarding adsorption kinetics, the GO from the palm shell adhered to the pseudo-second-order kinetics model with an R2 > 0.999.

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Conflicts of Interest

The authors declare that there are no conflicts of interest in this article.

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