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EFFECTS OF 4M NAOH OVER-ACTIVATION ON COCONUT SHELL CARBON AND ITS IMPACT ON THE ELECTRICAL CONDUCTIVITY OF REDUCED GRAPHENE OXIDE

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ABSTRACT

This study investigates the effect of excessive NaOH activation (4M) on coconut shell-derived carbon and its impact on the structural characteristics and electrical conductivity of reduced graphene oxide (RGO). Coconut shell waste was carbonized at 350°C for 2 h and activated using 4M NaOH for 24 h, followed by oxidation via the modified Marcano method and chemical reduction using 1.5 mL hydrazine hydrate assisted by microwave heating. Structural properties were analyzed using X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR), while electrical properties were measured with an LCR meter. The activated carbon exhibited an anomalously high mass yield of 125.30%, indicating severe over-activation and particle agglomeration. XRD analysis revealed an expanded interlayer spacing (d_{002} = 3.496 Å), larger than standard graphite (3.248 Å), confirming significant structural disorder. FTIR results showed persistent O–H stretching (~3330 cm^{-1}) and residual C–O vibrations (~544 cm^{-1}) in RGO even after reduction, indicating incomplete restoration of the sp^2 carbon network. The resulting RGO exhibited a very low electrical conductivity of 2.11×10^{-6} S/cm, which is approximately 5–7 orders of magnitude lower than typical RGO values reported in the literature (10^{-1}–10^2 S/cm). This severe degradation is attributed to disruption of sp^2 carbon domains and persistent oxygen functional groups that impair electron transport. These findings confirm that 4M NaOH induces over-activation that is detrimental to the electrical performance of biomass-derived RGO.

Keywords : reduced graphene oxide, coconut shell, NaOH activation, over-activation, electrical conductivity

I. INTRODUCTION

The increasing global demand for energy and the depletion of fossil fuel resources have accelerated the development of sustainable energy storage technologies, particularly lithium-ion batteries (LIBs) for electric vehicles and portable electronics [1], [2]. The performance of LIBs is strongly influenced by electrode materials, where electrical conductivity is a key factor determining charge transfer efficiency and overall electrochemical performance [3]. Graphite is commonly used as a commercial anode material, however, its limited theoretical capacity, high production cost, and energy-intensive manufacturing processes have driven the exploration of alternative carbon-based materials [4]. Reduced graphene oxide (RGO) has emerged as a promising candidate due to its relatively high electrical conductivity, tunable surface chemistry, and potential for low-cost synthesis from biomass-derived carbon sources[5].

Among various biomass precursors, coconut shell offers significant advantages due to its high fixed carbon content, low ash content, and renewable availability. Through carbonization and chemical activation, coconut shell can be converted into activated carbon, which serves as a precursor for graphene oxide (GO) and RGO synthesis [6], [7]. Chemical activation using sodium hydroxide (NaOH) is widely employed to enhance pore development and surface reactivity. At appropriate concentrations, typically 1–2 M, NaOH promotes controlled pore formation and facilitates the formation of oxygen functional groups that support effective GO synthesis and subsequent

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reduction [8]. However, excessive NaOH concentration can induce over-activation, characterized by severe chemical etching, structural fragmentation, pore collapse, and disruption of sp^2 carbon domains [9], [10].

Over-activation is particularly detrimental for graphene-based materials, as excessive defect formation and destruction of conjugated π-electron networks significantly reduce electrical conductivity even after reduction. Despite its critical impact, the over-activation effect caused by high NaOH concentration, especially at 4M, on biomass-derived carbon and its implications for the electrical conductivity of RGO remain insufficiently explored. Therefore, this study investigates the over-activation effects induced by 4M NaOH on coconut shell-derived carbon and examines its impact on the electrical conductivity of reduced graphene oxide. Structural and chemical characteristics were analyzed using X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR), while the electrical properties of RGO were evaluated using an LCR meter. This work provides insight into the limitations of excessive chemical activation and highlights the importance of controlling activator concentration in the development of sustainable graphene-based materials for energy storage applications.

II. METHOD

Coconut shell (CS) waste was washed and dried at 110°C, then carbonized at 350°C for 2 h in a furnace under limited-air conditions to produce coconut shell carbon [11]. The carbon was chemically activated by immersion in a 4M NaOH solution prepared by dissolving 16 g NaOH in 100 mL deionized water, yielding an impregnation ratio of 1:12.5 (w/v). Activation was carried out under constant stirring at room temperature for 24 h. The activated samples were washed with deionized water until a neutral pH was reached and dried at 105°C [12].

GO was synthesized from the activated carbon via the modified Marcano method using a mixed acid system of H_2SO_4 and H_3PO_4 (9:1 v/v), with $KMnO_4$ as the oxidizing agent. The resulting GO product was sequentially washed with a 5% HCl solution and deionized water to remove residual ions, then ultrasonicated for 2 h ensure uniform exfoliation, and oven-dried at 60°C to yield brownish-black GO powder [13].

The GO was reduced to RGO by dispersing the powder in ethylene glycol and adding 1.5 mL of pure hydrazine hydrate as the reducing agent. The reduction was performed using microwave heating, with cooling intervals between cycles to prevent overheating. The resulting RGO was washed thoroughly with deionized water and oven-dried to obtain dry RGO powder [14].

For electrical characterization, the dried RGO powder was compressed into pellets using a hydraulic press. The pellets were placed between conductive electrodes and measured using an LCR meter over a frequency range of 1 KHz to 10 KHz. The measured resistance values were used to calculate the electrical resistivity and conductivity of the RGO samples [15].

III. RESULTS AND DISCUSSION

The effect of high NaOH concentration on the activation behavior of coconut shell carbon is initially reflected in the mass yield. Activation using 4M NaOH resulted in an anomalously high yield of 125.30%, exceeding the initial carbon mass, as shown in Table 1.

Table 1. Mass yield of activated carbon at different NaOH concentrations

Sample Code	NaOH Conc. (M)	Initial Mass (g)	Final Mass (g)	Yield (%)	Mass Change (%)
CS *non-active*	-	8	-	-	-
CS1 *Active*	1	8.4	8.9	105.95	5.95
CS2 *Active*	2	8.3	8.2	98.80	-1.2
CS3 *Active*	3	8.4	9.2	109.52	9.52
CS4 *Active*	4	8.3	10.4	125.30	25.3

Such an abnormal yield is a strong indication of over-activation, where excessive alkaline interaction promotes the formation and entrapment of reaction by-products and moisture within the carbon matrix. Under highly concentrated NaOH conditions, aggressive chemical reactions can generate sodium-containing residues, such as Na_2CO_3, which may remain trapped within collapsed or partially blocked pore structures [9]. In addition, excessive activation may hinder effective washing, leading to incomplete removal of residual species [10]. Rather than representing improved carbon formation, this anomalous yield reflects structural instability and inefficient purification, which negatively affect subsequent oxidation and reduction processes.

The XRD diffractogram of the 4M NaOH–activated carbon shows a broad (002) reflection characteristic of turbostratic carbon (Figure 1). Structural parameters derived from the diffractogram are summarized in Table 2.

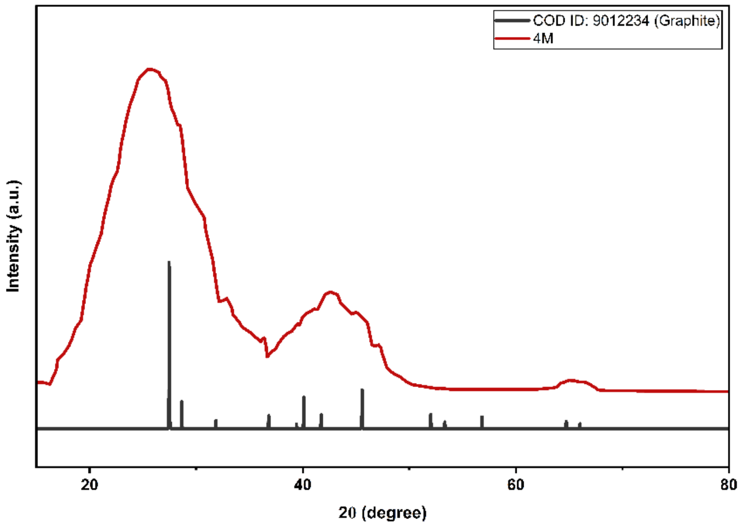

Fig 1. XRD diffractogram of activated carbon (4M NaOH) compared with graphite standard (COD ID: 9012234).

The (002) peak at 2θ = 25.48° yields d_{002} = 3.496 Å, which is notably larger than standard graphite (3.248 Å, COD ID: 9012234) and also exceeds the d-spacing typical of well-activated biomass carbons (~3.40–3.45 Å) reported in the literature [9]. This expansion indicates that 4M NaOH activation does not improve structural ordering but instead disrupts the graphene layer stacking, leading to misaligned layers and increased defect density. The large crystallite size values (Lc ≈ 51–53 nm) are notably higher than those typically reported for activated carbons (~2–10 nm), which is consistent with particle agglomeration rather than ordered crystalline growth a further consequence of overactivation. The disrupted sp^2 framework created under these conditions subsequently facilitates excessive penetration of oxidizing agents during GO synthesis, leading to uncontrolled oxidation and further degradation of the carbon network [16].

Table 2. XRD structural parameters of coconut shell activated carbon (4M NaOH)

Parameter	(002)	(100)
2θ (°)	25.4816	42.615
d-spacing (Å)	3.496	2.1216
Crystallite size (nm)	50.97	53.37

FTIR spectra of GO and RGO-TK4 derived from the 4M NaOH activated precursor compared in Figure 2. revealing distinct differences between the GO and RGO samples.

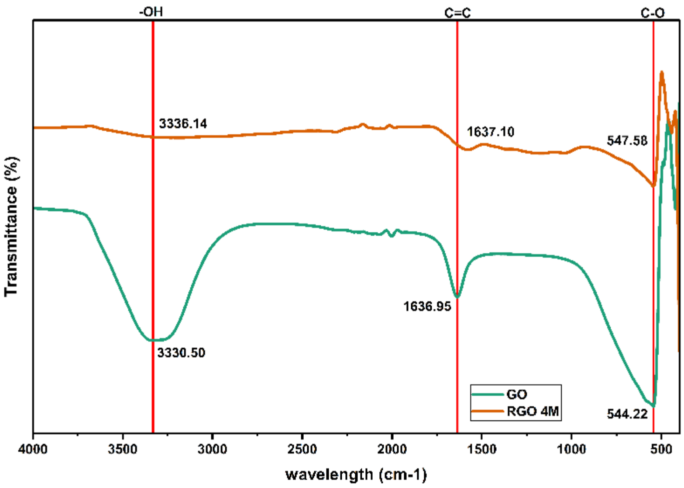

Fig 2. FTIR spectra comparison of GO and RGO derived from 4M NaOH-activated carbon

The GO spectrum shows a broad and intense O–H stretching band at 3330.50 cm^{-1}, indicating abundant hydroxyl functionalities resulting from the over-activated precursor. The C=C aromatic stretching at 1636.95 cm^{-1} reflects partial retention of the sp^2 carbon network, and the band at 544.22 cm^{-1} is associated with C–O or residual metal-oxide vibrations from the oxidation process. These features confirm that the structural disorder introduced during 4M NaOH activation created numerous reactive sites that were extensively oxidized during the Marcano process [17].

Following chemical reduction, the RGO-TK4 spectrum shows that the O–H band shifts slightly to 3336.14 cm^{-1} and remains broad and measurable, confirming that hydroxyl groups are not fully removed by hydrazine reduction. The C=C band at 1637.10 cm^{-1} is essentially unchanged compared to GO, indicating limited recovery of the aromatic sp^2 network. The low-wavenumber band shifts from 544.22 to 547.58 cm^{-1}, suggesting some structural rearrangement but continued presence of residual oxygen-related bonding. The persistence of these functional groups after reduction confirms that the oxidation damage introduced during over-activation cannot be fully reversed through chemical reduction alone [18]. These residual groups act as electron scattering centers that disrupt π-conjugated pathways essential for efficient electron transport.

The structural evolution from activated carbon through GO to RGO demonstrates a clear progression of degradation initiated by over-activation. The disrupted carbon framework in the activated carbon leads to uncontrolled oxidation in GO, which in turn results in incomplete structural restoration in RGO [19]. The inability to fully eliminate oxygen defects during reduction is a direct consequence of the severity of initial over-activation, confirming that damage introduced at the activation stage propagates irreversibly through the entire synthesis pathway. The electrical properties of RGO synthesized from the 4M NaOH–activated precursor are summarized in Table 3. The RGO exhibits a high resistivity of 4581.83 Ω·m and a low electrical conductivity of 2.11×10^{-6} S/cm. [20], [21].

Table 3. Electrical properties of RGO derived from 4M NaOH-activated carbon

Parameter	**Value**
Resistivity ρ (Ω·m)	4.58×10^3
Conductivity σ (S/cm)	2.11×10^{-6}
Typical RGO conductivity (S/cm)*	10^{-1}–10^2

* *Typical range compiled from* [20]–[22].

The RGO exhibits a high resistivity of 4.58×10^3 Ω·m and a very low conductivity of 2.11×10^{-6} S/cm. For comparison, well-prepared biomass-derived RGO typically achieves conductivities in the range of 10^{-1} to 10^2 S/cm [20], [22], meaning the present value is approximately 5–7 orders of magnitude lower. Previous studies have reported RGO conductivities of ~10^{-2} – 10^{-1} S/cm under comparable reduction conditions, further highlighting the severity of degradation observed here [21].

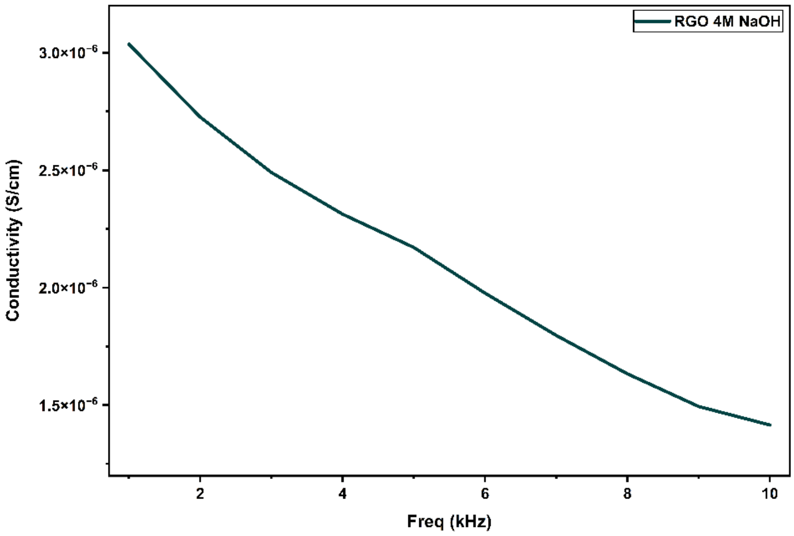

Fig. 3. Frequency dependent electrical conductivity of RGO derived from 4M NaOH-activated carbon.

The frequency-dependent conductivity profile in Figure 3 shows a gradual increase with increasing frequency, which is characteristic of defect-dominated, hopping-type charge transport. Under such conditions, electron transport occurs by hopping or tunneling between isolated conductive sp^2 regions rather than through continuous delocalized π-electron pathways, resulting in high resistance and very poor bulk conductivity [22]. This behavior results from the cascade of structural damage initiated at the over-activation stage: the disrupted sp^2 network in the activated carbon precursor leads to uncontrolled oxidation during GO synthesis, which in turn causes incomplete structural restoration during chemical reduction, as directly evidenced by the persistent FTIR bands. The resulting fragmented conductive pathways and abundant charge scattering centers collectively suppress electron mobility to semiconducting levels far below the requirements for high-performance energy storage applications [23].

Overall, the results demonstrate that activation using 4M NaOH induces severe over-activation, characterized by anomalous mass yield, structural disorder, excessive oxidation, and degradation of electrical conductivity. These findings confirm that excessively high NaOH concentrations are counterproductive for producing conductive graphene-based materials from biomass-derived carbon and highlight the necessity of controlling activation severity to preserve structural integrity and electrical performance.

IV. CONCLUSION

This study demonstrates that activation of coconut shell carbon using 4M NaOH leads to severe over-activation, which significantly degrades the structural integrity and electrical performance of reduced graphene oxide (RGO). The over-activation is evidenced by an anomalously high mass yield of 125.30%, indicative of residue entrapment, and an expanded interlayer spacing of $d_{002} = 3.496$ Å, which exceeds both the graphite standard (3.248 Å) and typical activated carbon values (~3.40 – 3.45 Å), as confirmed by XRD analysis.

FTIR analysis confirms that specific oxygen-containing functional groups O–H stretching at 3330.50/3336.14 cm^{-1} and C–O vibrations at 544.22/547.58 cm^{-1} persist in RGO-TK4 even after chemical reduction, indicating irreversible disruption of the conjugated sp^2 carbon network. Electrical characterization reveals that the resulting RGO exhibits a high resistivity of $4.58 \times 10^3\ \Omega\cdot m$ and a very low conductivity of 2.11×10^{-6} S/cm, representing a degradation of approximately 5 – 7 orders of magnitude compared to typical RGO values of 10^{-1} – 10^2 S/cm. This poor conductivity is governed by defect-assisted charge hopping rather than delocalized π-electron conduction.

These findings confirm that 4M NaOH over-activation is detrimental for producing electrically conductive RGO from biomass-derived carbon. Future research should (1) systematically investigate lower NaOH concentrations in the range of 0.5 – 2M at 0.5M intervals to identify the optimal activation threshold; (2) evaluate alternative activators such as KOH and $ZnCl_2$ for comparison; and (3) explore post-reduction thermal annealing at 300–500°C as a strategy to partially recover sp^2 domain continuity in over-activated precursors.

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