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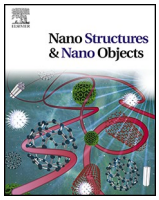

Synthesis and application of biomass-based graphene oxide using microwave-assisted pyrolysis method

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ABSTRACT

Graphene and related materials have excellent potential to revolutionise many emerging fields, but the large quantity synthesis using environmentally sustainable methods is still challenging. This work studies the synthesis of graphene oxide from food waste biochar with negatively charged properties, which is initially produced using microwave pyrolysis. The synthesised graphene oxide achieved 64.53 % carbon content, 345.79 m^2/g surface area, 0.074 cm^3/g micropore volume, large reactive surface area, and ribbon/banded organisation. The performance of carbon-based material is evaluated in the fabrication of a modified screen-printed carbon electrode (SPCE) for selective dopamine (DA) detection in the coexistence of ascorbic acid (AA) and uric acid (UA). The electrostatic interactions between the negatively charged biochar-based graphene oxide (BBGO) on the surface SPCE and opposite-charged dopamine assisted the oxidation potential with interfering species by electrostatic repulsion of AA and UA. This manuscript substantiates the resource recovery from organic materials and sustainable graphene oxide synthesis, underscoring their application in detecting positively charged species.

1. Introduction

Food waste has become a global issue due to environmental, economic, and health effects. The excess food waste generation from the agricultural industry and households is attributed to the increased population and large quantity of non-consumed food [17,37]. It is estimated that in 2025 the food waste will be around 416 million tonnes and 23–65 % of that will correspond to vegetables and fruits [26,40]. Food waste presents two major issues: the loss of large quantities of consumed natural resources and the environmental issues created by the disintegration of food materials. Food waste dumping releases greenhouse gases (GHG), e.g., methane, carbon dioxide, and carbon monoxide during its decomposition, contributing to global warming [26,34].

Recently, the agricultural waste management of crops, fruits, vegetables, etc., involves composting, landfill, incineration, and animal feed [7,33]. Nevertheless, none of these alternatives is practical regarding sustainability, pollution mitigation, scale-up, efficiency, and circular economy. Some agricultural wastes are difficult to process and are considered non-usable materials for certain types of technologies [23]. The selection of biomass treatment is based on waste conversion efficiency, limited resource consumption (energy, chemical, space, etc.), processing time, and target by-products [4].

Microwave-assisted is an efficient conversion method capable of processing biomass in a short treatment time, generation of three by-products (biochar, bio-oil, biogas), uniform heating distribution, and reduced environmental footprint (CO_2 sequestration) [3,40]. The final by-product namely biochar, which has superior properties than other conversion mechanisms (conventional heating), e.g., higher carbon concentration, large reactive surface area, higher microporosity volume, rich carboxylic and hydroxyl surface group, etc. Allende et al., [39,40,6]. Though biochar is a good source of carbon and has already been demonstrated for many applications such as water purification, and carbon sequestration— the properties of the biochar can be improved significantly by activating the biochar for the fabrication of graphene oxide [13,14,16,21,5]. Traditional synthesis methods of graphene oxide, such as Hummers' technique, have several drawbacks. These include the release of toxic gases during the oxidation process involving $NaNO_3$, extended reaction times (over 6 h), complex processing due to the removal of residual Na^+ and NO_3^- ions, and the use of non-sustainable feedstocks [2,49]. In contrast, microwave pyrolysis offers an eco-friendly method for synthesizing graphene oxide, capable of processing large quantities of various wastes in a shorter treatment time [5,

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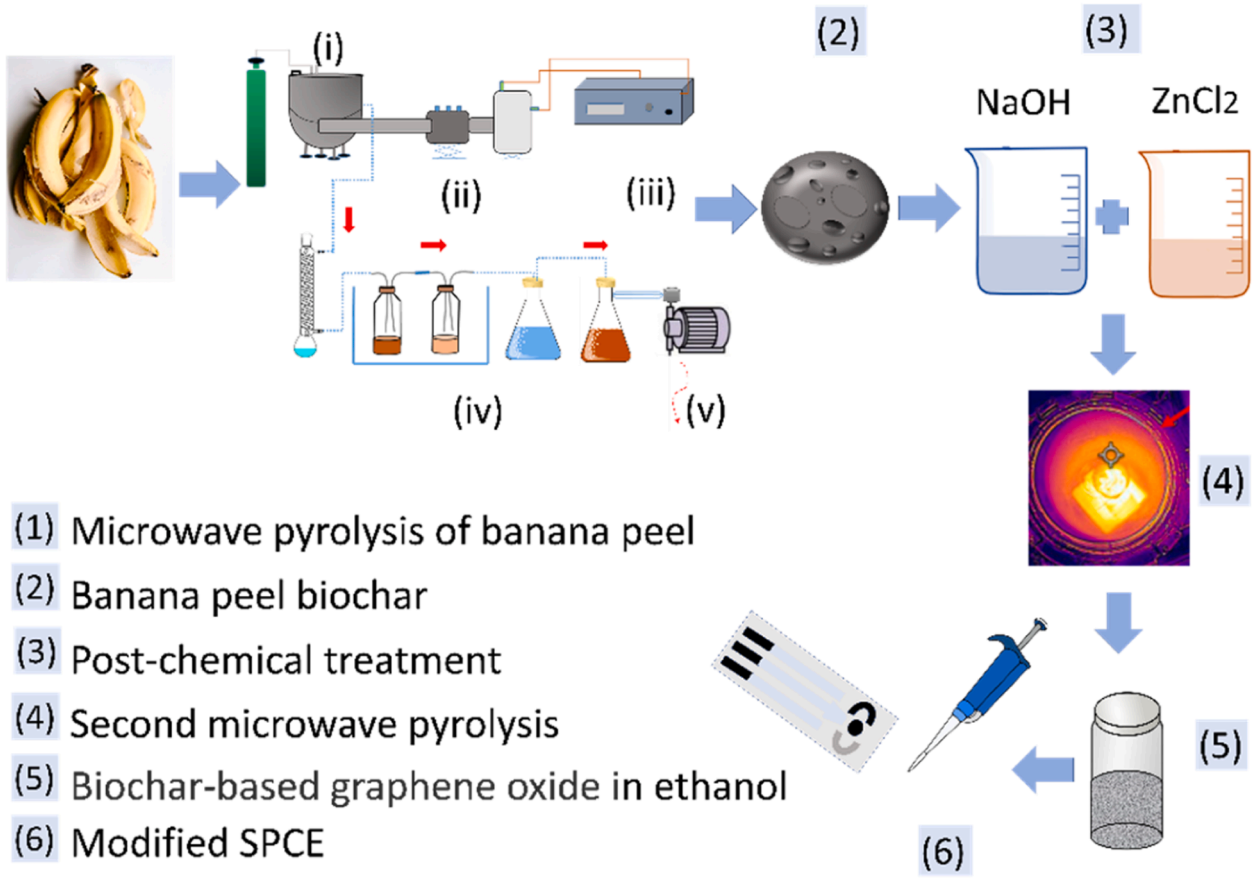

Fig. 1. Synthesis of BP BBGO and modified electrode fabrication.

Table 1
Ultimate analysis of banana peel biochar and BP BBGO.

Analysis	Raw banana peel	Banana peel biochar	BP BBGO
Surface area (m^2/g)			
BET Surface Area		27.65	345.79
t-Plot micropore Area			173.99
t-Plot external surface area		40.83	171.79
Pore volume (cm^3/g)			
t-Plot micropore volume			0.074
Total pore volume calculated <1.0228 nm diameter at p/p°			0.106
Elemental analysis			
N (%)	0.32	4.2	0.7
C (%)	35.88	63.18	64.53
H (%)	1.34	3.13	1.36
O (%)	62.46	29.49	33.41
H/C	0.04	0.05	0.02
O/C	1.74	0.47	0.52

6].

The activation of biochar has gained immense interest in the fabrication of bio-based graphene derived from agricultural waste due to its unique properties, sustainable process methods, and diversity of functions like the development of electrical and optical technologies, which include electrochemistry sensors, supercapacitors, energy storage, biomedicine, electronics, etc. Ahmad Farid, Andou [1,11,48]. This carbon material is highly valued in electrochemical applications, like the detection of numerous molecules by using biochar-modified sensors [19, 38]. Some examples of sensing organic species are AA, DA, and UA. These analytes are electrochemically active biomolecules that coexist in biological fluids [8,25]. AA is a vital vitamin found in different life processes (human metabolism); DA is a neurotransmitter molecule that exists in the central nervous system; and UA is the main final compound of purine catabolism in humans [29,50]. Several studies report the simultaneous detection of AA, UA, and DA [45,50]. However, the selectivity property of bare electrodes is limited for dopamine detection in the presence of other species. The electrochemical oxidation of AA, DA, and UA occurs at similar potential, which means poor selective electrochemical response for the single detection of molecules [18,29]. The electrostatic interactions between the electrode and the analyte have a relevant role in the detection of dopamine, e.g., AA (pKa = 4.10) and UA (pKa = 5.40) are oppositely charged species compared to DA (pKa = 8.87) at physical pH 7.0 [18]. Hence, the fabrication of a modified electrode with electrostatic attraction to positively charged molecules is essential to resolve the overlap oxidation response between the species.

Using banana peel as a feedstock source conversion for the generation of carbon material represents a valuable opportunity to reduce the disposal of organic waste and decrease greenhouse emissions. This research evaluates the synthesis of a negatively charged carbon-based material obtained from the microwave pyrolysis of banana peel (BP) waste into biochar-based graphene oxide (BBGO). The properties of BP BBGO are studied in the fabrication of an environment-friendly modified screen-printed carbon electrode (SPCE) for selective detection of DA in ascorbic acid and uric acid coexistence.

2. Experimental methods

2.1. Synthesis of BBGO

The biomass treatment was developed mainly by a thermochemical conversion process achieved by microwave-assisted pyrolysis. Fig. 1

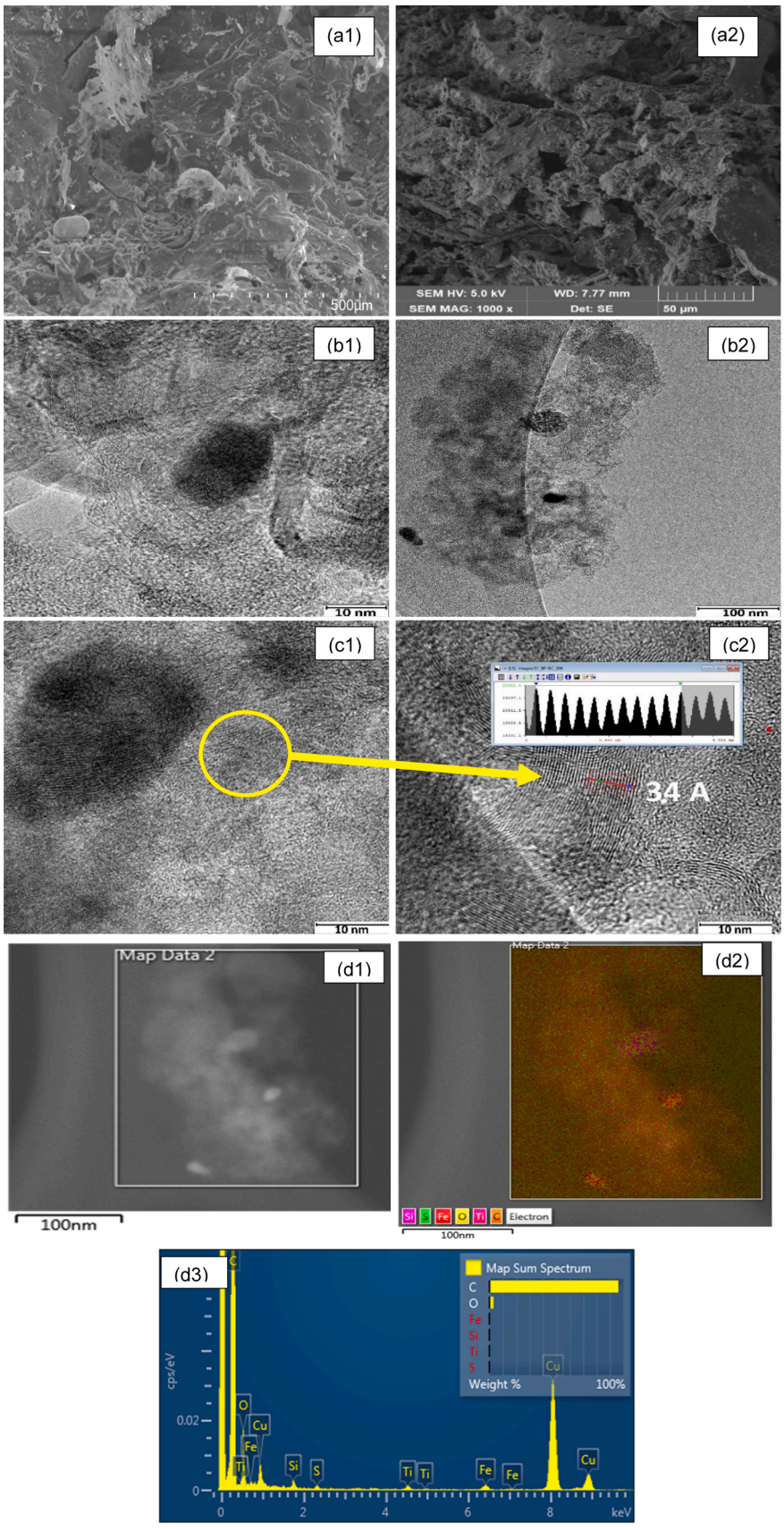

Fig. 2. SEM images of BP biochar (a1) and BP BBGO (a2) and, (b1) (b2) TEM, (c1) (c2) lattice spacing, and (d1) (d2) (d3) TEM-EDS images of BP BBGO.

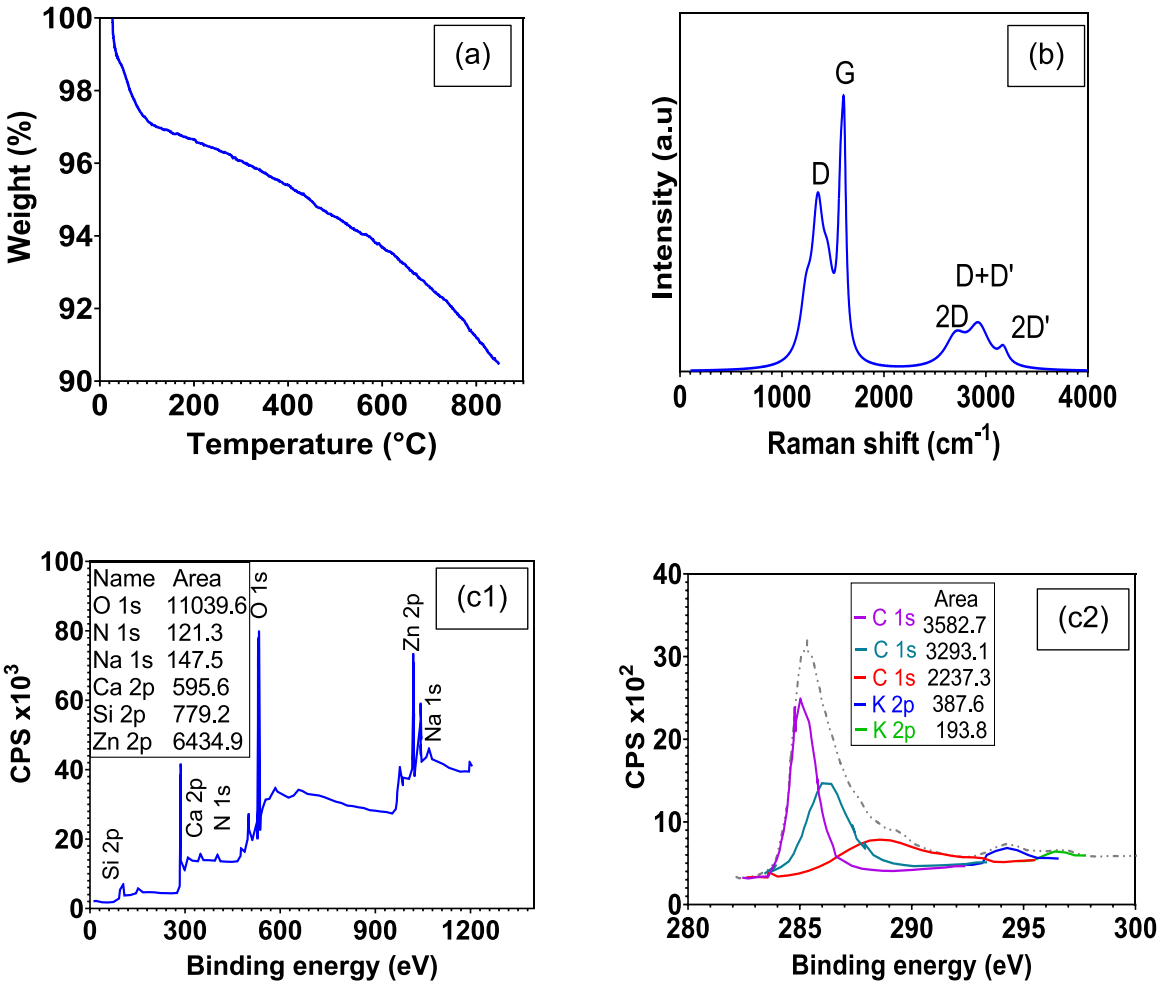

Fig. 3. (a) TGA, (b) Raman spectra, and (c1) (c2) XPS curves of BP BBGO.

Table 2
Zeta potential data of BP BBGO.

	Value
Zeta Potential (mV)	−9.34
Conductivity (mS/cm)	0.0134
Measured Current (mA)	0.00358
Measured Voltage (V)	149

describes the synthesis stages used for the synthesis of graphene oxide; the microwave pyrolysis system is comprised of the following units: (i) a custom-made chamber where pyrolysis occurs under an oxygen-free environment applying 5 L/min nitrogen as a carrier gas, (ii) a manual tuner allows controlling reflected power; (iii) a 3 kW microwave generator; (iv) flask condensers system (liquid collection), and (vi) a vacuum pump (-50 kPa). The synthesis of BBGO involves four stages. First, 200 g of selected biomass was pyrolyzed at 1.5 kW for 1.5 h, considering the 10 % microwave susceptor of biomass weight. Banana peel (BP) was used as feedstock. The resulting banana biochar was immersed in NaOH with distilled water in a 1:1 ratio for 2 days at room temperature. After this period the treated biochar was washed successive times with distilled water and then cured with $ZnCl_2$ in a 1:1 ratio for three days. The next activation procedure of BP biochar requires successive rinsing with distilled water and drying at 80 °C for five hours. Then the treated biochar was subjected to a second pyrolysis at 1 kW for 40 min, resulting in BBGO from the banana peel.

2.2. Preparation of modified SPCE

The biochar obtained from the chemical and thermochemical treatment (microwave heating) was ground in a mortar until it reached a uniform particle size. The BP BBGO was dispersed in ethanol and then exposed to ultrasonication (~30 min) until reached fine biochar particles. Then 1 μL of 1 mg mL^{-1} dispersed material was used as a coating on the surface of the SPCE. The modified SPCE was dried at room temperature.

2.3. Reagents and apparatus

The chemical treatment of biochar was developed using sodium hydroxide pellets from Univar and Zinc Chloride Solution (1 M) purchased from Sigma. The reagents used in the electrochemical analysis (AA, DA, and UA) also were obtained from Sigma. The buffer solution (PBS, pH 7.0) was produced using dibasic-Na_2HPO_4 and monobasic-NaH_2PO_4. ItalSens SPCE was used in the electrochemical study purchased from PalmSens. The SPCE characteristics are global dimensions of 0.8 ×4.5 cm, working electrode dimensions of 7.06 mm^2, substrate of polyester, 200 μm thickness, 2.54 mm contact pad pitch, and 2 % coefficient of variation. The electrochemical measurements were succeeded using PalmSens4 potentiostat (PalmSens, Netherlands). The surface area of BP BBGO was measured using a micromeritics 3-flex surface and porosity analyser; FlashSMART used for CHNSO elemental analysis, scanning electron microscopy (JEOL 7001 F SEM); transmission electron microscopy (JEOL 2100 TEM), thermogravimetric analysis (TGA) using Netzsch STA 449F3 Jupiter Simultaneous Thermal

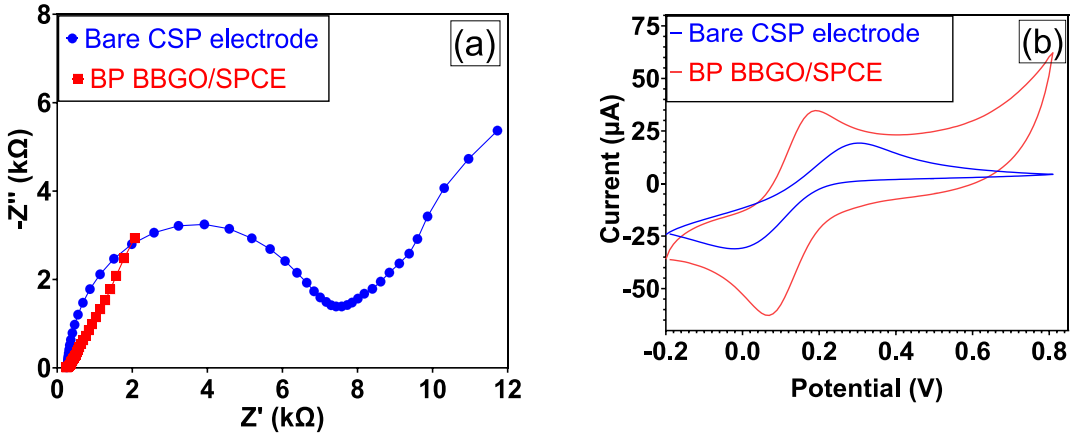

Fig. 4. (a) EIS and (b) CV curves of bare SPCE and BP BBGO/SPCE in 0.1 M KCl containing 5 mM K_3 [(Fe $(CN)_6$)] at 5 mV potential amplitude.

Analyser; X-ray photoelectron spectroscopy was performed using a Kratos Axis Supra, Raman spectroscopy completed by Renishaw In-Via Micro-Raman spectrometer. The electrostatic behaviour of BBGO was studied using zeta potential - Malvern ZetaSizer for suspended particles <1000 nm, considering an experimental condition pH of 7.0.

3. Result and discussion

3.1. Characterisation of BP BBGO material

BP BBGO's quality and electrochemical analytical performance mainly depend on its CHNSO elemental analysis and high specific surface area. The pre/post-pyrolysis and chemical treatment favoured the BET surface area and micropore volume formation [51]. After the impregnation with NaOH, the carbon structure of biochar changed (Na_2CO_3 breakdown) by the microwave heating method, releasing CO, CO_2 and H_2 ($2Na + 2Na_2CO_3 + 3H_2$) and causing higher porous generation [22]. Adding $ZnCl_2$ contributes to the physically enhanced porosity and surface functionalisation [22,47].

Table 1 shows the surface area and elemental analysis of banana peel biochar and BP BBGO. A significant increase in surface area was observed after the thermochemical treatment of BP biochar, achieving 345.79 m^2/g BET surface area, 173.99 m^2/g micropore area and 0.074 cm^3/g micropore volume. A high surface area is crucial for increasing the electrochemically active surface area between the carbon material and electroactive molecules [18,29]. The thermochemical activation process enables the formation of a microporous structure in BP BBGO, enhancing its adsorption performance and facilitating mass and electron transport. This process also improves the presence of surface-active functional groups [20,43,52]. After the microwave pyrolysis process and chemical activation, the feedstock suffered an increased carbon content of 80 % and oxygen reduction of 53 %. The carbon content of BP BBGO is 2 % higher than raw biochar. After activation, the H/C and O/C ratios were reduced by 60 % and 10 %. Reduced H/C value indicates high biochar carbonisation, large aromaticity degree, and high aromatic structure [24]. A low O/C ratio of activated biochar denotes a reduction of hydrophilic and polarity properties [6,24].

Fig. 2 (a1) and (a2) shows the SEM images of raw banana peel biochar and BP BBGO, respectively. These figures reveal a notable difference in the biochar structure. BP biochar has a solid and uniform surface, but after the chemical and thermochemical process biochar experienced the breakdown of fibre compounds, forming pores and some carbon layers. Fig. 2 (b1) and (b2) correspond to the TEM images of BP BBGO. The particles observed are more clusters of mixed material than layered, with areas of ribbon/banded organisation, where the lattice spacing is ~ 3.4 A—shown in Fig. 2 (c1) and (c2). The TEM-EDS data confirms that carbon and oxygen are primary elements in the base matrix with a minor presence of heavier metal and organic-based particles (Ca, Fe, P, Si and Ti). Some carbon areas exhibit semi-organisation in crumpled packed particles, not layers.

The TGA curve of BP BBGO is shown in Fig. 3(a). The thermal decomposition exposes an initial weight loss between 100 °C and 200 °C. This mass weight reduction is associated with moisture content. A constant mass loss was observed between 200 °C and 800 °C, decreasing by 9.55 %. This result means that at this temperature range, the BBGO reached its thermal stability and its fraction of volatile components. The residual mass detected was around 90.45 % at 849.8 °C. The Raman spectroscopy curve indicates a high G peak at 1600 cm^{-1}, which denotes a high graphitization grade [6]. Lower intensity is detected in the D band, which involves the existing defects in the structure [28]. The Raman curve of BP BBGO was comparable to the GO material reported by [27,46]. Fig. 3 (c1) and (c2) of XPS results denote an atomic concentration of 59.5 % carbon and 30.7 % oxygen. A minor presence of calcium, potassium, nitrogen, sodium, silicon, and zinc was detected. Nitrogen and other heteroatoms are favourable for electron transfer and ion insertion [12,38]. On the other hand, Table 2 shows the zeta potential of BP BBGO, which is a negative charge (-9.34 mV). This value indicates the electrostatic behaviour on the carbon material surface and gives an idea about the electrostatic interaction with different charge species.

3.2. Electrochemical characterisation

The electrochemical properties of BP BBGO/SPCE are determined by the material sensor properties on the electrode surface, evaluating its active surface area and the electron transfer behaviour of the carbon material [15]. The electron transfer resistance of modified SPCE was studied from electrochemical impedance spectra (EIS) measurement. The EIS spectra are based on two main responses: (i) a semicircle that reveals electron transfer resistance (R_{ct}) and (ii) a linear section that represents solution diffusion resistance [6]. Fig. 4(a) shows the Nyquist plot of bare and modified electrodes. The surface property of BP BBGO/SPCE from EIS denotes a smaller semicircle diameter (0.3 kΩ) than the bare electrode (7.5 kΩ). This result implies that the modified electrode has lower charge-transfer resistance than bare SPCE. The cyclic voltammetry (CV) response of modified SPCE at 5 mV and 5 mM potassium ferricyanide was also analysed in Fig. 4(b). The current density of BP BBGO/SPCE was significantly higher than that of the bare SPCE, confirming the superior electrochemical active surface area of the modified SPCE. The potential peak-to-peak separation of bare SPCE and the modified electrode was 0.2 V and 0.1 V, respectively. This small

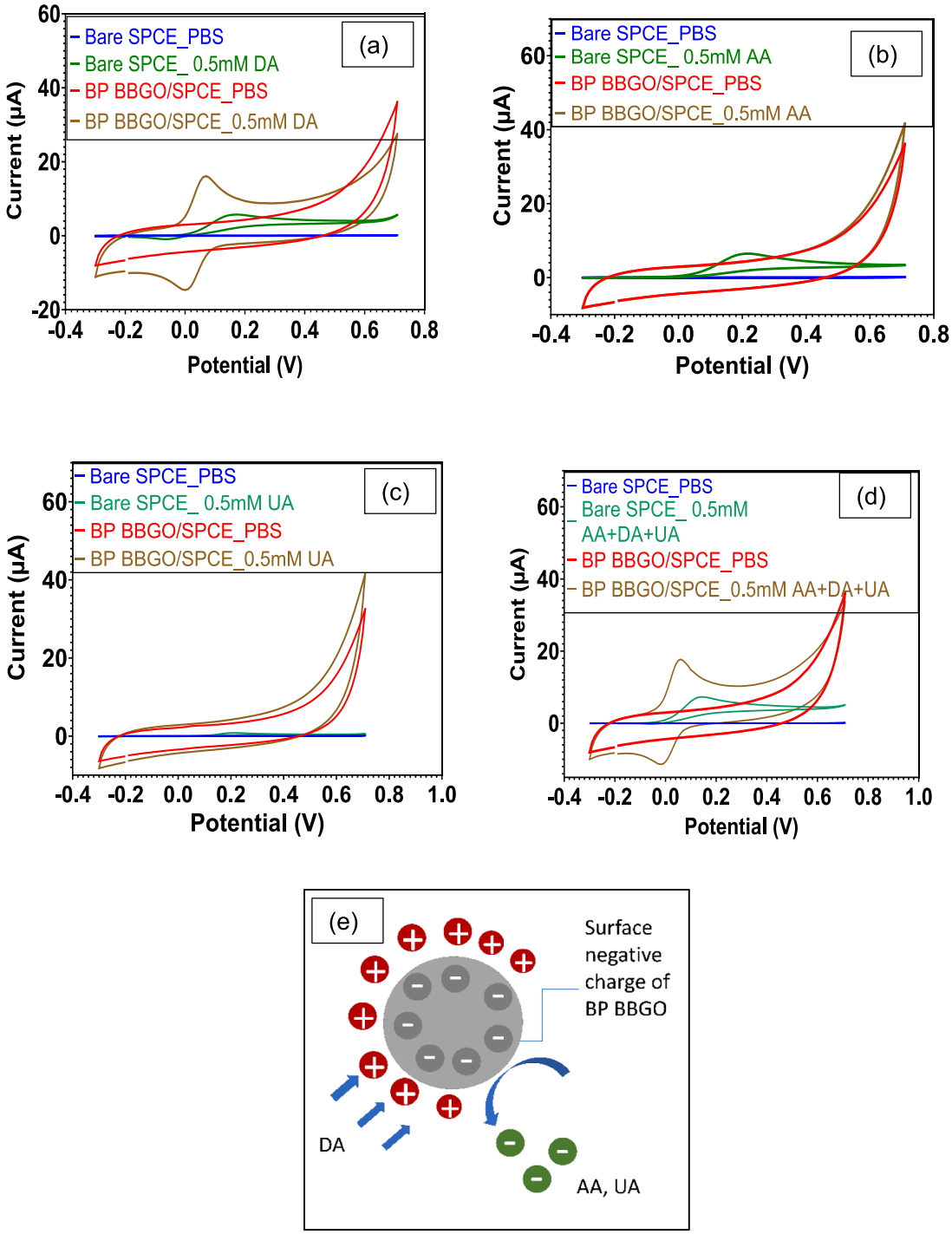

Fig. 5. CV curves of bare SPCE and BP BBGO/SPCE in 0.5 mM AA (a), 0.5 mM DA (b), 0.5 mM UA (c), and a combination of 0.5 mM AA+ 0.5 mM DA+ 0.5 mM UA (d) in 0.1 M phosphate buffer solution (pH 7.0) at 0.05 V/s scan rate, (e) surface potential reaction between the surface negative charge material and the charged molecules.

peak-to-peak difference and potential shift (~ 1 V) indicate a greater electron transfer reaction [44].

3.3. Dopamine detection in the presence of interfering species

AA, DA, and UA are usually found simultaneously in biological human fluids. However, it was reported that these species have similar redox potentials [29,42], which makes it challenging to detect DA. Herein, the selectivity of BP BBGO/SPCE towards determining DA in the coexistence with these interferents was tested by voltammetry. Fig. 5(b) and (c) show the oxidation current response of non-modified and modified SPCE in the presence of individual AA and UA. The electrochemical results of modified SPCE do not exhibit a current response to AA and UA. These CV curves were analogous to the modified electrode in the blank solution. Nonetheless, a distinctive oxidation peak was observed in the presence of 0.5 mM DA (12.12 μA peak), whose current intensity was around three times higher than the bare electrode, shown in Fig. 5(a). A potential shift (reduction) of 0.09 V was also noticed, the oxidation peak of bare SPCE was achieved at 0.15 V, while the BP BBGO/SPC electrode was at 0.06 V.

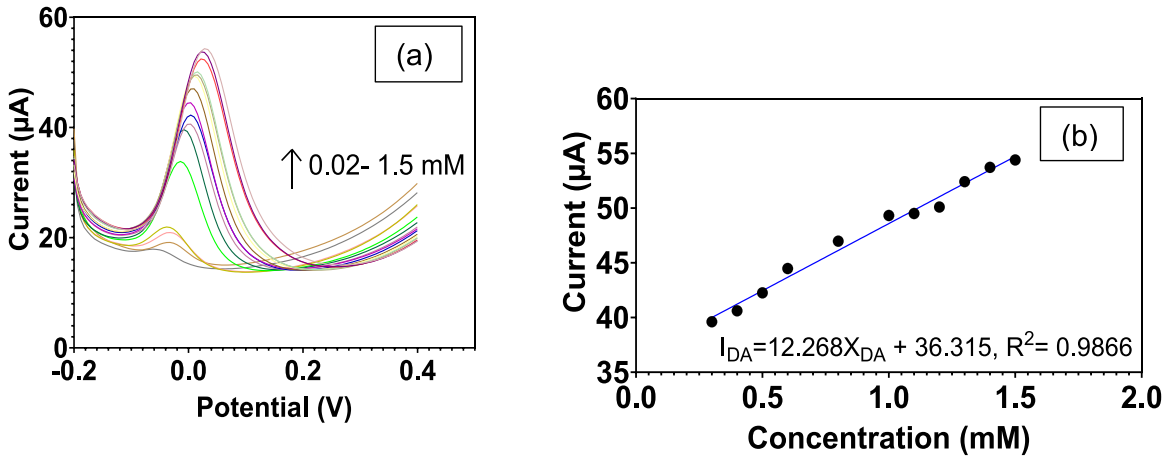

Fig. 6. DPV curve of DA (0.02–1.5 mM) at the (a) BP BBGO/SPCE in the co-existence of 0.5 mM AA and 0.5 mM UA (b) and the corresponding calibration curve.

Table 3
Electrochemical sensing performance of various modified electrodes for DA detection.

Electrode	Linear range (μM)	LOD (μM)	Reference
Sulfonated graphene	0.2–20	0.02	[18]
PtNPs-rGO-ITO	0.1–20	0.075	[29]
PtAu/GCE	24–384	24	[41]
S-MC−1/GCE	0.01–5	3	[10]
Au/Gr-Au	10–100	30	[31]
PG/GCE	5–710	2	[32]
BP BBGO/SPCE	20–1500	20	This work

Table 4
Estimation cost of graphene oxide produced from the microwave pyrolysis process.

Item	Biochar (g)	BBGO (g)	Energy (kWh)	(c/kWh)	(AUD/g)	(AUD)
Feedstock			-	-		-
MW susceptor			-	-		-
Pyrolysis 1.5 kW/ 1.5 hr	20	12	2.25	20.19		0.45
Carbonization 1 kW/ 40 min			1.005			0.20
NaOH activation					0.16	3.17
$ZnCl_2$ activation					0.17	3.12
Pyrolysis maintenance						0.08
Total operating cost						**7.03**
Item						
Commercial GO	(g)	(AUD/g)	(AUD)			
	12	0.6	7.20			
Total gained			**0.17**			

In terms of selectivity, the bare electrode shows a similar oxidation potential for single detection of the species, e.g., the current response of ascorbic acid was 6.47 μA at 0.21 V, DA was 5.72 μA at 0.18 V, uric acid was 0.8 μA at 0.2 V, AA+UA+DA was 7.29 μA at 0.135 V. The non-modified electrode evidences a notable limitation for dopamine detection due to the bare SPCE showing only one oxidation peak in the co-existence of molecules, and it was not able to sense the analytes separately in the presence of 0.5 mM AA+UA+DA. This indicates that the oxidation peaks for all the species overlapped. In contrast, as shown in Fig. 5(a) and (d) BP BBGO/SPCE only shows an oxidation peak in the presence of DA, with no electro-activity for interfering molecules. This finding reveals that the carbon material on the electrode surface has improved the electrochemically active area for dopamine oxidation.

The electrostatic interactions between the carbon material on the surface of the SPCE and the interfering species were analysed to resolve the oxidation peaks of DA. This phenomenon can be explained by the intrinsic properties of BP BBGO, e.g., the carbon material is negatively charged (shown in zeta potential data), the same as ascorbic acid (pKa = 4.10) and uric acid (pKa = 5.40), but DA is oppositely charged (pKa = 8.87) at physical pH 7.0 [18]. Therefore, the electrostatic interactions on the surface of the modified electrode repel the negatively charged species of AA and UA, however, high attraction to positive change DA molecules. These properties are the main principles for achieving selective DA detection and resolving the oxidation response in co-existence with interference species. The electrostatic reaction between the carbon material and molecules is illustrated in Fig. 5(e).

Differential pulse voltammetry (DPV) is an electrochemical technique commonly used with a highly sensitive and low detection limit [30]. This technique was applied to compare the properties of modified CSPEs. The sensitivity of the BP BBGO/SPCE was studied in Fig. 6(a) and (b). The DPV curves show that the oxidation current response of DA was not affected by the presence of AA and UA. The linear range region of the modified SPCE was between 0.02 mM and 1.5 mM DA. The linear regression equation is represented by I_{DA}= 12.268X_{DA}+ 36.315 with a correlation coefficient of 0.9866 and a limit of detection (LOD) of 0.02 mM. Table 3 reports the sensing characteristics of different DA sensors. Considering that some of the modified electrodes reported in the literature are fabricated using complex and non-sustainable techniques, such as the traditional Hummer's method [31], the BP BBGO/SPCE electrode demonstrated higher electrochemical performance for DA detection.

3.4. Economic analysis

The economic analysis of the synthesis of BBGO using microwave pyrolysis is shown in Table 4. The estimated cost related to the microwave pyrolysis process was calculated based on previous work reported by Allende et al. [3]. For this calculation, two pyrolysis conditions were considered: raw biomass processing (1.5 kW for 1.5 hr) and the calcination process of activated biochar (1 kW for 40 min). An electricity cost of 20.19c/kWh was used [3]. From 200 g of feedstock, 20 g of biochar was obtained, which was then calcined to produce 12 g of BBGO. Furthermore, the expenses for the chemical activation were estimated using a market price of 0.16 AUD/g for NaOH [35] and 0.17 AUD/g for $ZnCl_2$ [36]. Additionally, the commercial value of graphene oxide was

estimated at 0.6 AUD/g, as reported by [9].

The economic analysis demonstrated that microwave pyrolysis is $14 cheaper than the market price for producing 1 kg of graphene oxide. The low manufacturing cost of BBGO allows for the fabrication of a sustainable electrochemical sensor and ensures higher production efficiency compared to other forms of graphene.

4. Conclusion

This study reports the use of food waste as feedstock for the synthesis of graphene oxide. The BBGO material has carbon and oxygen as primary elements in the base matrix with a minor presence of heavier metal and organic-based particles, ~ 3.4 A lattice spacing, 345.79 m^2/g BET surface area, 173.99 m^2/g micropore area, and 0.074 cm^3/g micropore volume. The graphene oxide synthesised using microwave-assisted pyrolysis shows excellent opportunities for bulk production of GO in addition to the overall cost-effectiveness and environmental approaches. Though GO could be used in several applications, it is demonstrated for developing an electrochemical sensor for dopamine detection in this work. The fabrication of the modified SPCE using BP BBGO showed higher selectivity and sensitivity of DA detection compared to the bare electrode. The improvements are related to the intrinsic properties of the modified electrode, such as high electrical conductivity, lower charge-transfer resistance, a higher electroactive area that increases the charge propagation of analytes and surface negative charge that attracts positively charged DA and repelling the negatively charged species like AA and UA. The electrochemical detection of DA in the modified electrode demonstrated better selectivity in co-existence with interference molecules than the bare SPCE. This green carbon material has the potential to be applied in the sensing of other positively charged molecules. Overall, the developed technique is a highly efficient method for adding value to agricultural waste and converting the biochar into much more valuable graphene oxide, thereby developing various sensors.

CRediT authorship contribution statement

Mohan V. Jacob: Writing – review & editing, Validation, Supervision, Data curation. **Muhammad Adeel Zafar:** Writing – review & editing, Data curation. **Yang Liu:** Writing – review & editing, Validation, Supervision, Data curation. **Scarlett Allende:** Writing – original draft, Methodology, Investigation, Formal analysis, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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