Parabens (PBs) are a group of compounds that comprise a p-hydroxybenzoic acid ester with various alkyl, aryl and benzyl substituents. Because of their broad antibacterial action spectrum, they are often utilized as preservatives in personal care products. PBs were once thought to be the lowest hazardous and safest way to preserve time, particularly in cosmetics and pharmaceuticals.1,2 Current investigations, however, have revealed that PBs are harmful and endocrine-disrupting chemicals, owing to the presence of alkyl chains.3 PBs have been detected in breast cancer cells and marine creatures, indicating that they would be used with caution.4 Several nations have lately prohibited many sunblocks including certain dangerous compounds, containing PBs, to stop coral reef destruction, reaffirming the toxicity of PBs and the importance of their sensitive and precise assessment.5 Especially, Methylparaben (MPB) is linked to the womb and ovarian cancer, endometriosis and other hormonal issues in women.6,7 The bioaccumulation of these chemicals in the body, particularly in the mammary region, has resulted inside consequences.8 Children are also among those in danger. As a result, the determination of MPB would be the major concern in this study.

For determining MPB in a range of matrices, various analyzed approaches have been published, including HPLC,6 GC–MS10 and CE.11 LC–MS.12 Moreover, for the PBs determination, some electroanalytical techniques have been reported.6,13–15 Electrochemical techniques might have certain merits over other analytical techniques such as demanding less sample preparation, consuming a little time, and having a higher limit of detection and linear range.6,13 These techniques have been confirmed to be effective in the development of particularly sensitive and selective procedures for detecting organic compounds in pharmaceutical dosage forms and biological fluids, including medicines and related chemicals.14 From all the above benefits, therefore, the electrochemical technique would be employed to determine MPB within all the experiments.

Modified electrodes, for example, are suitable candidates for constructing electrochemical sensor surfaces for the determination of analytes including phenolic functional groups, which are notoriously associated with electrode fouling on bare electrodes.19 The capacity of modified electrodes to transmit electrons between electrode material and analyte, the electrochemically active surface area, the number of exposed active sites and their stability in reaction fluids are all aspects that influence their results.19–22 Various nanostructures with novel combinations and topologies have been produced, opening new possibilities for nanomaterial-based sensors.23–26 Current results strongly imply that carbon nanofibers,27 and multi-walled carbon nanotubes28 could be used to determine parabens. Nevertheless, depositing these materials onto the detecting electrodes necessitates either several stages process or the usage of expensive noble metals, resulting in materials with perfect anti-fouling qualities. It has led to broad research dedicated to optimizing physicochemical characteristics, morphologies and dimensions of NMTs to cater to the gap between in-lab-technologies and commercial requirements. In addition, in term of market analysis, to detect an analyte at a higher temperature, it is necessary to demand micro-heating assemblages, which increase cost money, energy demand, size and decreases its lifetime because of merging of grain boundaries of nanostructures operated at high temperature.33,34,38–40 Reduced graphene oxide (RGO), on the other hand, is one of the least studied materials for this application such as nanoelectronics, conductive thin films supercapacitors, nanosensors and nanomedicine,41,42 because it possesses extraordinary thermal, mechanical and electrical properties.43 Poly L-lysine (PoL) possesses a high level of biocompatibility, many active amino groups, a flexible structural framework, and good water solubility.44 PoL-RGO (poly L-lysine-modified reduced graphene oxide) is a new composite material with pharmaceutical uses. We employed PoL-RGO material to build a modified electrode for detecting MPB, which is the most extensively employed PBs. Before being used as electrode modification materials for the determination of MPB, PoL-RGO was subjected to comprehensive materials characterization to ensure the formation and be applied to the electrochemical analysis during the work. To the best of our knowledge, a PoL-RGO nanostructured-based sensor for the electrochemical determination of MPB has never been described before.

**Experimental**

**Material.**—Methylparaben (C₈H₈O₃, 99%), L-lysine, reduced graphene oxide (RGO), disodium hydrogen phosphate (Na₂HPO₄, ≥ 99%), potassium dihydrogen phosphate (KH₂PO₄, ≥ 99%), diocetyl sulfosuccinate sodium salt (AOT, ≥ 97%), gasoline (98 octanes, un-leadred), acetone (C₂H₅OH), ethanol (EtOH, C₂H₅OH), were received from Sigma-Aldrich Co. Ltd. and employed as a reagent without further purification. Before beginning each set of tests, stock solutions for MPB were produced in PBS (0.1 M) pH 7. Deionized (DI) water was also utilized in this whole work.
The procedure of microemulsion polymerization was utilized to make poly L-Lysine (PoL): 4.5 g l-Lysine dissolved in 2.5 ml water with 0.5 g NaOH. The next step is to acquire a homogeneous solution, 0.25 ml of the prepared L-Lysine solution was added to thirty ml of 0.1 M AOT/gasoline solution and agitated for a half at 1000 rpm. Then, forty μl of THPC was poured as a crosslinker to the solution and poly L-Lysine was precipitated in excess acetone after 5 min. To eliminate unreacted and surfactant compounds, the produced poly L-Lysine was washed orderly by acetone (C₃H₆O), EtOH (C₂H₅OH) and DI water not less than three times each.

Preparation of PoL-RGO and PoL-RGO/GCE.—RGO (1.5 mg) were poured into a PoL (1.5 mM) solution in a 30 ml aqueous solution. The PoL solution was ultrasonicated for one hour. The supernatants were obtained by centrifuging the solution at 3500 rpm for 10 min, then being treated at 80 °C for 24 h. The functionalized PoL was the resultant precipitate. The RGO functionalized with PoL was denoted as PoL-RGO. To make a colloidal PoL-RGO solution, 1.5 mg PoL-RGO was put in a 30 ml aqueous solution. This mixture was sonicated for ten mins. To compare dispersion, the PoL (1 mg) was also sonicated within ten mins in 20 ml of distilled water. The PoL-RGO powder was disseminated in one solution within the concentration value was 1.5 mg ml⁻¹, then ultrasonicated for 1.5 h. To make the PoL-RGO/GCE electrode, the suspension solution was put within the GCE surface and gradually dried. The PoL-RGO/GCE has then been employed for MPB determination investigations as well as other research.

Instrumentation and preparation of real sample.—The UV–vis spectrums were recorded on the DR6000 spectrograph machine.

Synthesis of PoL.—The procedure of microemulsion polymerization was utilized to make poly L-Lysine (PoL). 1.25 g l-Lysine dissolved in 2.5 ml water with 0.5 g NaOH. The next step is to acquire a homogeneous solution, 0.25 ml of the prepared L-Lysine solution was added to thirty ml of 0.1 M AOT/gasoline solution and agitated for a half at 1000 rpm. Then, forty μl of THPC was poured as a crosslinker to the solution and poly L-Lysine was precipitated in excess acetone after 5 min. To eliminate unreacted and surfactant compounds, the produced poly L-Lysine was washed orderly by acetone (C₃H₆O), EtOH (C₂H₅OH) and DI water not less than three times each.

Preparation of PoL-RGO and PoL-RGO/GCE.—RGO (1.5 mg) were poured into a PoL (1.5 mM) solution in a 30 ml aqueous solution. The PoL solution was ultrasonicated for one hour. The supernatants were obtained by centrifuging the solution at 3500 rpm for 10 min, then being treated at 80 °C for 24 h. The functionalized PoL was the resultant precipitate. The RGO functionalized with PoL was denoted as PoL-RGO. To make a colloidal PoL-RGO solution, 1.5 mg PoL-RGO was put in a 30 ml aqueous solution. This mixture was sonicated for ten mins. To compare dispersion, the PoL (1 mg) was also sonicated within ten mins in 20 ml of distilled water. The PoL-RGO powder was disseminated in one solution within the concentration value was 1.5 mg ml⁻¹, then ultrasonicated for 1.5 h. To make the PoL-RGO/GCE electrode, the suspension solution was put within the GCE surface and gradually dried. The PoL-RGO/GCE has then been employed for MPB determination investigations as well as other research.

Instrumentation and preparation of real sample.—The UV–vis spectrums were recorded on the DR6000 spectrograph machine.
HACH, German) with the examination of PoL-RGO solution which was formed by diluting with deionized water. FT-IR analyses were reported on a Shimadzu IRPrestige-21 (Japan) was utilized to study the functionalized groups of the chemicals in the sample. The analytes were analyzed and compared utilizing Thermo Ultimate 3000 UHPLC equipment. Electrochemical experiments were done by applying a CPA-HH5 Computerized Polarography Analyzer (Vietnam). The working electrode was GCE having a 2.8 mm

**Figure 3.** (a) CV curves of MPB on PoL-RGO/GCE at different pH: 4, 5, 6, 7, 8, 9, 10; (b) The linear plot between $I_p$ and pH. Condition: The range of potential scans from 0.2 and 1.8 V, scan rate 0.2 V s$^{-1}$, $C_{MPB} = 50$ $\mu$M in 0.1 M PBS.

**Figure 4.** The DPVs of MPB at separate concentrations: (a) 5, (b) 50 and (c) 100 $\mu$M. Conditions: the potential scans from 0.2 to 1.6 V; scan rate 0.2 V s$^{-1}$. 

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RGO and PoL-RGO. As can be seen in Fig. 1a, the spectrum of RGO revealed the existence of $O$ peak was observed at 1371 cm$^{-1}$ of PoL and PoL-RGO. Furthermore, a characteristic C$\equiv$O($v_{C=O} = 1654$ cm$^{-1}$) and C$=$O($v_{C=O} = 1654$ cm$^{-1}$) were obviously seen in the situations of PoL and PoL-RGO. Furthermore, a characteristic C$\equiv$N absorption peak was observed at 1371 cm$^{-1}$ (binding with the aromatic ring), indicating that RGO was successfully functionalized with PoL (described in Scheme 1).

Figure 5. DPVs of 50 $\mu$M MPB with various modified PoL-RGO/GCE in 0.1 M PBS buffer solution (pH 7). The conditions in this experiment are the potential scans from 0.2 to 1.6 V; scan rate 0.2 V s$^{-1}$.

Table I. Effect of several interferents on the peak current of 50 $\mu$M MPB in 0.1 M PRS buffer solution (pH 7).

| Interferent          | Interferent: MPB (M/M) | RE (%) |
|----------------------|------------------------|--------|
| CaCl$_2$             | 120                    | -4.0   |
| Fe(NO$_3$)$_3$       | 80                     | 2.5    |
| Na$_2$SO$_4$         | 150                    | 2.3    |
| KHC0$_3$             | 160                    | -3.7   |
| Al(NO$_3$)$_3$       | 110                    | -4.1   |
| ZnCl$_2$             | 100                    | -4.7   |
| Sodium benzene sulfonate| 80                   | -3.1   |
| Methyl salicylate    | 70                     | 4.5    |
| $m$-trihydroxybenzene| 130                    | 4.1    |
| Cinnamaldehyde       | 90                     | 4.3    |
| p-nitro phenol       | 90                     | -4.6   |

Cyclic voltammetry.—Employing the cyclic voltammetry (CV) experiments at physiological pH of 7, the electrochemical behavior of the four above-mentioned electrodes for detecting MPB was examined (Fig. 2). For the unmodified GCE electrode in the addition of 50 $\mu$M MPB, as seen in this figure, no anodic peak could be detected during the experiments. For the modified electrode, the difference in the MPB’s anodic peak could be observed as follow: the defined peaks at 0.86 V and 0.8 V would be determined with the use of RGO/GCE and PoL-RGO/GCE (adding to 50 $\mu$M MPB), respectively, while this peak is not found with the support of PoL-RGO/GCE (without the MPB existence). In addition, the MPB peak current scanned by PoL-RGO/GCE is sharply greater than, around 3.1 times, compared to the RGO/GCE. These performances suggest that the modified PoL-RGO/GCE greatly improved to transfer of electrons, implying that the catalytic oxidation of MPB at this electrode was more effective than the others.

Figure 3a illustrates the study of pH dependence on electrochemical responses when applying the CV technique. The electrochemical signals are almost completely influenced by the pH buffer solution, indicating that the electro-oxidation procedure is largely dependent on proton concentration. As could be seen in the figure, when increasing the pH from 4 to 7, there is a significant growth in the oxidation peak current and the peak value current magnitude achieves the highest value at pH of 7 (Fig. 3b). As a result, the pH value of 7 was chosen for the whole work. Furthermore, when the pH is increased from 7 to 10, the peak potential drops gradually from 0.8 V to 0.6 V, demonstrating that protons are engaged in MPB oxidation at the modified electrode.

The repeatability, and reproducibility studies.—The repeatability of the PoL-RGO/GCE for the DPV technique was analyzed 10 times with the three MPB concentrations: 5, 50 and 100 $\mu$M in 0.1 M of the PBS buffer solution and the value of pH is 7 (described in Fig. 4). The Relative Standard Deviations (denoted as RSD) calculated as 2.2, 1.6, 1.4 corresponding to 5, 50 and 100 $\mu$M MPB solutions are all smaller than ½RSD$\text{Horwitz}$ (2.9, 2.1, 1.5 for 5, 50 and 100 $\mu$M MPB, respectively). The value of RSD$\text{Horwitz}$ was calculated by the formula as follows: 46 $\text{RSD}_{\text{Horwitz}} = 2.210^{-0.5logC}$, where C is the fraction concentration of the analysis compound. As a result, it could be concluded from the above results that the PoL-RGO/GCE possesses good repeatability and would be suitable to determine MPB at various concentrations.

The reproducibility of the PoL-RGO/GCE through the DPV technique was tested by evaluating 50 $\mu$M of the MPB solution in a 0.1 M PBS buffer solution with a pH value is 7. The experiment was executed by recording the DPV curves of MPB employing 05 PoL-RGO/GCE (one particular bare GCE was modified by PoL-RGO material with 05 times). The results, as shown in Fig. 5, indicated that the modified electrode by PoL-RGO material has great reproducibility since the value of RSD for $I_p$,MPB signals applying 05 separated PoL-RGO/GCE were 4.7%.

UV–vis spectroscopy was used to confirm the formation of PoL and RGO. The UV–vis spectrum of PoL only displayed a strong absorption peak at 212 nm, as illustrated in Fig. 1b. At 266 nm, a high absorption was reported in RGO. These 2 absorption peaks showed a modest change after PoL was applied to modify the RGO surface. The UV–vis peak absorption corresponding to RGO decreases from 266 to 254 nm, while the peak related to RGO was discovered to move from 212 to 214 nm. All the above performances described the interactions between PoL and RGO to generate a novel composite, PoL-RGO.

Characterization.—Figure 1a shows the FTIR spectrum of PoL, RGO and PoL-RGO. As can be seen in Fig. 1a, the spectrum of RGO revealed the existence of O–H ($v_{O-H} = 3439$ cm$^{-1}$), C–C ($v_{C-C} = 1638$ cm$^{-1}$) and the oxygen functions adsorption bands vanished, like that of pure graphite. Some distinctive absorption peaks of PoL functional groups including N–H ($v_{N-H} = 3420$ cm$^{-1}$) and C=O ($v_{C=O} = 1654$ cm$^{-1}$) were obviously seen in the situations of PoL and PoL-RGO. Furthermore, a characteristic C–N absorption peak was observed at 1371 cm$^{-1}$ (binding with the aromatic ring), indicating that RGO was successfully functionalized with PoL (described in Scheme 1).

Results and Discussion

Table I. Effect of several interferents on the peak current of 50 $\mu$M MPB in 0.1 M PRS buffer solution (pH 7).

| Interferent          | Interferent: MPB (M/M) | RE (%) |
|----------------------|------------------------|--------|
| CaCl$_2$             | 120                    | -4.0   |
| Fe(NO$_3$)$_3$       | 80                     | 2.5    |
| Na$_2$SO$_4$         | 150                    | 2.3    |
| KHC0$_3$             | 160                    | -3.7   |
| Al(NO$_3$)$_3$       | 110                    | -4.1   |
| ZnCl$_2$             | 100                    | -4.7   |
| Sodium benzene sulfonate| 80                   | -3.1   |
| Methyl salicylate    | 70                     | 4.5    |
| $m$-trihydroxybenzene| 130                    | 4.1    |
| Cinnamaldehyde       | 90                     | 4.3    |
| p-nitro phenol       | 90                     | -4.6   |

Five cosmetic samples (05), were collected and treated in the following: the samples were dissolved by the mixture MeOH/H$_2$O with a ratio of 80/20 (v/v), then the obtained product was sonicated at 40 $^\circ$C for 30 min. In the next stage the solution was mixed with MeOH/H$_2$O (80/20, v/v) to achieve a 50 ml volume. The resulted solution was centrifuged at 12000 g and the obtained product was sonicated at 50 $^\circ$C for 30 min.

The UV–vis absorbance peak at 212 nm, as illustrated in Fig. 1b. At 266 nm, a high absorption was reported in RGO. These 2 absorption peaks showed a modest change after PoL was applied to modify the RGO surface. The UV–vis peak absorption corresponding to RGO decreases from 266 to 254 nm, while the peak related to RGO was discovered to move from 212 to 214 nm. All the above performances described the interactions between PoL and RGO to generate a novel composite, PoL-RGO.
MPB, with 70–90-fold concentration of methyl salicylate, Fe(NO₃)₃, Sodium benzene sulfonate, cinnamaldehyde, p-nitro phenol or with 100–160-fold concentration of ZnCl₂, Al(NO₃)₃, CaCl₂, m-trihydroxybenzene, Na₂SO₄ and KHCO₃. The RE% for substrates were less than 5%, illustrating that no interference was caused to the peak current of MPB (Table I). This leads to describe the great selectivity of the modified sensor—PoL-RGO/GCE.

Limit of detection, Limit of quantification.—The MPB was determined at varying concentrations from 1 to 200 μM employing the differential pulse anodic stripping voltammetry (DP-ASVs) technique, which is much more sensitive than the CV technique. The operation conditions were tuned in the following: the potential scans from 0.2 to 1.6 V; scan rate 0.2 V s⁻¹.

Figure 6a describes the DPV curves corresponding to the increasing MPB concentrations in 0.1 M PBS pH 7 at the proposed modified electrode. At 0.83 V, a well-defined DP-ASV peak is observed. Figure 6b shows the corresponding calibration graph. The regression equation between the MPB concentration and the peak current is illustrated in the following:

\[ I_p = (0.0287 \pm 0.0007)C + (0.23 \pm 0.06) \]

\[ R^2 = 0.996 \]

The obtained LOD and LOQ for MPB are 0.20 μM, and 0.7 μM, respectively (employing 3σ/s, in which S is the sensitive performance and σ is the SD (standard deviation) performance of the black signal). Table II compares the determination performance of MPB at the given modified electrode with the other works. It is worth noting that MPB’s linear range and LOD at the PoL-RGO/GCE are lower or comparable to those previous works. Therefore, the modified PoL-RGO material is a useful electrode modification for detecting MPB in water solutions.

**Table II. The report of LOD and linear range of the previously published works with several techniques.**

| No. | Electrode | Technique | LOD (μM) | Linear range (μM) | References |
|-----|-----------|-----------|----------|-------------------|------------|
| 1   | GCE/(MWCNTs-LB) | LSV       | 0.4      | 1.0–80            | 2          |
| 2   | RGO/RuNPs/GCE    | DPV       | 0.24     | 0.5–3             | 47         |
| 3   | ZnO/GCE         | SWV       | 7.25     | 20–120            | 48         |
| 4   | Au/(MNP/Ppy)₃   | DPV       | 0.0095   | 0.0–131.40        | 49         |
| 5   | CPE/MWCNTs/Hb   | DPV       | 0.025    | 0.1–13            | 50         |
| 6   | GCE/(AuNP-RGO-CS) | SWV       | 0.0138   | 0.03–1.3          | 3          |
| 7   | PoL-RGO/GCE     | DPV       | 0.20     | 1–200             | This work |

Note: LB: Langmuir-Blodgett film; RGO/RuNPs: reduced graphene oxide—ruthenium nanoparticles; Hb: hemoglobin; AuNP: gold nanoparticles; GCE: glassy carbon electrode; MWCNTs: multi-walled carbon nanotubes; CPE: carbon paste electrode; Ppy: polypyrrole; MNP: magnetite nanoparticles; CS: chitosan; Au: gold; PoL: poly L-lysine.

**Table III. The determination performance of MPB concentrations (μM) in 05 biological fluids samples through the two methods of DPV and HPLC.**

| Samples | DPV method | HPLC method |
|---------|------------|-------------|
| Found (μM) | Re (%) | Found (μM) |
| S1 | 12.8 | 102.8 | 11.5 |
| S2 | 60.9 | 98.1 | 62.0 |
| S3 | 10.5 | 97.0 | 9.5 |
| S4 | 44.9 | 104.3 | 45.3 |
| S5 | 28.1 | 98.5 | 29.3 |

Real samples.—To evaluate the applied ability to examine MPB of the method, the suitable experiment conditions have been enforced profitably to determine MPB in various cosmetics samples obtained from five different volunteers. These cosmetics samples were purified by using a 0.45 μm pore glass fiber filter paper and heating at 4 °C. The cleaned samples, then, were analyzed by the proposed method and HPLC method for comparison (Table III). The recovery (denoted as Re (%) shown in Table III with the value ranging between 97% and 104.3%, which was within the suitable scope, indicates that the recovery method is acceptable. The statistical analysis illustrated that both DPV and HPLC analyses have no difference when investigating MPB in biological fluids.
samples (Paired-Samples t-test, $\alpha = 0.05$; $t(4) = 0.153$; $P_{\text{t}-\text{ailed}} = 0.886 > 0.05$). According to the obtained results, the modified PoL-RGO/GCE would be employed to precisely determine the MPB concentrations in human biological fluids.

**Conclusions**

In this work, the PoL-RGO material was synthesized, and its structure was characterized by UV–vis and FT-IR spectrum to confirm the successful synthesis process. Moreover, this composite was employed to modify bare GCE with the target of determining MPB using voltammetric techniques, including cyclic voltammetry and differential pulse voltammetry. The PoL-RGO/GCE presents a sharp enhancement of the current response and great electrocatalytic activity towards MPB oxidation. The proposed technique expresses a good linear relation between MPB concentration and the current response in the concentration ranging from 1 to 200 $\mu$M with a limit of detection of 0.2 $\mu$M. In addition, by employing the differential pulse voltammetry technique, the PoL-RGO/GCE-modified GCE indicates that this electrode possesses good repeatability (the value of RSD is 2.2%, 1.6%, 1.4%) and reproducibility (RSD of 4.7%). Thenobilia recovery rates were greatly satisfactory and suggest the great electrochemical results of PoL-RGO/GCE for detecting MPB exists in cosmetic samples ($n = 5$).

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