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Detection of the synthetic cathinone 4-methyl-pentedrone using lab-made screen-printed electrodes: A simple and environmentally friendly screening method for forensic applications

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ABSTRACT

4-Methylpentedrone, also known as 4-MPD, is a drug belonging to the class of synthetic cathinones (SCs) widely reported for recreational use due to its potent stimulant effects. The preliminary identification of SCs in seized samples is of great interest in the forensic setting. In this context, we present an attractive screen-printed electrode for SC detection in forensic samples, using 4-MPD as a model analyte. In the quest for sensors with more environmentally friendly characteristics, polyethylene terephthalate (PET) from discarded beverage bottles (recyclable) was repurposed as a substrate for conductive ink deposition featuring glass varnish and carbon nanotubes (CNTs-GV/PET). Characterization studies of CNTs-GV/PET showed that the conductive ink is versatile and robust for application in electroanalysis. The electrochemical detection of 4-MPD was optimized in 0.1 mol/L phosphate buffer at pH 8.0 using the square wave voltammetry (SWV), where this drug exhibited one oxidation and two reduction processes on the CNTs-GV/PET. The proposed method provided a wide linear range for 4-MPD determination (1.0–100.0 μmol/L) with a low limit of detection (0.1 μmol/L). Under optimized conditions, SWV profiles of adulterants and other twelve illicit drugs, commonly found in seizures containing SCs, demonstrated that the proposed method is highly selective for 4-MPD screening in forensic samples. Therefore, the proposed method offers a simple, selective, and environmentally friendly approach for SC detecting with great potential for application in forensic analysis.

1. Introduction

Synthetic cathinones (SCs) are a class of drugs widely illicitly traded worldwide, leading to numerous seizures during law enforcement activities [1]. 4-Methylpentedrone (Fig. S1), also known as 4-MPD, is a dangerous synthetic stimulant belonging to the class of synthetic cathinones (SCs). This illicit drug is the p-methyl analog of pentedrone and a higher homolog of 4-methylmethcathinone (mephedrone). It can be found in various forms such as powder, crystals, rocks, capsules, and tablets. This substance was initially notified to European and French early warning systems in 2014 (Czech Republic) and 2017, respectively [2,3], but it was only in 2020 that a fatal poisoning involving 4-MPD was reported [4]. Consequently, analytical procedures are essential for the preliminary identification of SCs, such as for 4-MPD.

The United Nations Office on Drugs and Crime (UNODC) recommends the use of colorimetric methods based on the Zimmermann reagent [5] as the preliminary test of SCs. While useful, colorimetric methods may yield inaccurate results, as they can be influenced by the presence of other drugs and adulterants, resulting in false positives and false negatives [6,7]. On the other hand, electrochemical methods have proved valuable for detecting SCs and other illicit drugs in forensic applications due to their operational simplicity, low instrumental cost, and high portability [8–26]. However, these methods can be improved for a more realistic application in forensic routine, offering simpler and

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lower cost electrochemical sensors than the previous works. In addition, to the best of our knowledge, there is no reported electrochemical method for the detection and quantification of 4-MPD in forensic samples.

Within the realm of analytical devices, disposable screen-printed electrodes (SPEs) have gained importance in recent years. This trend is particularly evident in the forensic field, where these electrodes find application in the screening of both traditional drugs [27–30] and new synthetic drugs [31–35]. This is due to the need for rapid monitoring of illicit substances right at the seizure site [5]. Thus, SPEs emerge as a promising alternative due to their simplicity, portability, and ability to work with small sample volumes [36–39].

In the development of these new electrochemical devices, proper preparation of conductive ink is crucial, requiring two essential components: a binding agent and a conductive material [40]. Carbon nanotubes (CNTs) exhibit outstanding electrochemical characteristics, such as high sensitivity, selectivity, stability, and reproducibility [41]. Thereby, the combination of CNTs' features with the creation of compact devices provided by screen-printed technology opens new avenues for portable electrochemical detection.

Conductive inks based on CNTs represent a highly promising perspective for the fabrication of electrochemical devices [40]. These inks enable the production of sensors with diverse and adjustable characteristics, serving as an alternative to the limitations of traditional SPEs [42]. The most traditional screen-printed electrochemical sensors are those made with platinum, gold, and silver. These metals offer high conductivity and durability but are relatively expensive compared to carbon-based materials. Additionally, carbon-based conductive materials offer lower manufacturing costs, easier availability, reduced environmental impact after use, and comparable analytical results [43].

Among binding agents, various polymers can be employed in the formulation of conductive inks, with alkyd resin standing out as a commonly used compound in synthetic paints, varnishes, and enamels [44]. In this context, glass varnish (GV), a commercial varnish used for decorative purposes in crafts, contains alkyd resin in its composition, making it a suitable option as a binding agent [42]. GV exhibits characteristics such as flexibility, good solubility, fast drying, great surface adhesion, and relatively low cost [42]. Furthermore, the additional advantage of using GV stems from its widespread commercial availability, providing notable accessibility [42].

In light of the above insights, we present a lab-made disposable electrochemical sensor based on carbon nanotube and glass varnish conductive ink printed on PET (CNTs-GV/PET) for detecting SCs in seized samples using the square wave voltammetry (SWV) technique with 4-MPD as a model analyte. The electrochemical behavior of 4-MPD is investigated on CNTs-GV/PET, revealing a new reduction process for identifying SCs.

The proposed sensor is environmentally friendly, offering a portable, fast, and simple screening method for SCs in forensic applications. The CNTs-GV/PET exhibits an intrinsic peak potential, which can be used as internal standard to enhance the reliability of preliminary identification of 4-MPD and other SCs in seized samples. In addition, CNTs-GV/PET combined with SWV provides adequate sensitivity and selectivity for the detection and quantification of SCs in forensic analysis.

2. Experimental

2.1. Chemicals and samples

The conductive material and binding agent used in this study consisted of multi-walled carbon nanotubes with an outer diameter ranging from 20 to 30 nm and an inner diameter ranging from 5 to 10 nm, sourced from nFinitu Tech LLC® in Florida, United States. The glass varnish (GV) employed is manufactured and distributed by ACRILEX®, São Paulo, Brazil, and comprises a blend of alkyd resin, xylenes, and ethylene glycol monobutyl ether. Polyethylene terephthalate (PET) sheets, obtained from recycled soda bottles, were used as the sensor substrate.

All solutions were prepared using deionized water with a resistivity of not less than 18.2 MΩ cm (at 25 °C), obtained through the Milli-Q system (Millipore, USA). The analytical standard for 4-Methylpentedrone (4-MPD), supplied by the United Nations Office on Drugs and Crime (UNODC), was dissolved in HPLC-grade methanol. The initial stock solution (1.0×10^{-2} mol/L) of 4-MPD was subsequently diluted in a supporting electrolyte for electrochemical measurements.

The electrochemical behavior of 4-MPD was explored in a Britton–Robinson (BR) buffer solution, formulated from a blend of boric, phosphoric, and acetic acids at varying pH values (ranging from 2.0 to 12.0), all with 10 % methanol (v/v). The BR buffer was chosen for its wide pH range (2–12), allowing the investigation of the analyte's behavior under varying acid-base conditions within a single buffer system while facilitating precise control of ionic strength, which is essential in electrochemical analyses [45]. Sodium hydroxide was employed for pH adjustment.

Additionally, a 0.1 mol/L phosphate buffer solution (PBS) at pH 8.0 with 10 % methanol and a McIlvaine buffer solution (20.55 mL of 0.1 mol/L citric acid + 79.45 mL of 0.2 mol/L Na_2HPO_4) were considered as supporting electrolytes for 4-MPD detection. All reagents were of analytical grade.

Various substances, including caffeine, paracetamol (acetaminophen), ketamine, benzocaine, lidocaine, procaine, cocaine, methylenedioxymethamphetamine (MDMA), methylenedioxyethylamphetamine (MDEA), methamphetamine (MA), amphetamine (A), benzylpiperazine (BZP), meta-chlorophenylpiperazine (mCPP), dibutylone, 3',4'-tetramethylene-α-pyrrolidinovalerophenone (TH-PVP), 3,4-methylenedioxy-α-pyrrolidinohexanophenone (MDPHP), and mephedrone, were assessed as potential interferents in 4-MPD detection using the proposed method.

Real seized samples (tablets) containing 4-MPD, previously confirmed by quadrupole time-of-flight liquid chromatography-mass spectrometry (LC-Q-TOF-MS) by the Civil Police of the Federal District, Brazil, were subjected to investigation using the proposed method for preliminary identification tests. Each sample was macerated and dissolved in 1 mL of methanol, then diluted in the supporting electrolyte for electroanalysis. Additionally, addition–recovery studies of 4-MPD in these seized samples were carried out using the proposed method.

2.2. Instrumental and apparatus

All voltammetric experiments were conducted using a PGSTAT 101 N potentiostat (Metrohm Autolab BV, Utrecht, Netherlands), controlled by NOVA 2.1.7 software. Cyclic voltammetry (CV) and SWV techniques were used for electrochemical behavior studies and the detection of 4-MPD, respectively. SWV parameters were optimized for 4-MPD detection by studying the variation in amplitude (10 – 100 mV), step potential (1 – 10 mV), and frequency (10 – 100 Hz). A system of three electrodes (working, auxiliary, and reference) was used, based on conductive carbon nanotube ink and stained-glass varnish printed on PET sheets obtained from soda bottles (CNTs-GV/PET). For the pH study, an external $Ag/AgCl_{(sat)}$ reference electrode was used.

2.3. Preparation of CNTs-GV/PET sensor

To manufacture the CNTs-GV/PET sensor, we used the method proposed by Carvalho et al. [36]. Fig. 1 schematically illustrates the step-by-step construction of the electrochemical sensor. In Fig. 1A, the cutting of soda bottles to create PET plates is shown. The surface was sanded with conventional sandpaper (220 Grit) and subsequently cleaned with 70 % alcohol to remove residue. To delimit the electrodes that form the sensor's three-electrode system, a Con-Tact® paper adhesive mask was cut with the help of a Silhouette 4 cutting printer, controlled by the Silhouette Studio® software.

In Fig. 1B, the preparation of conductive ink is shown. Initially, GV

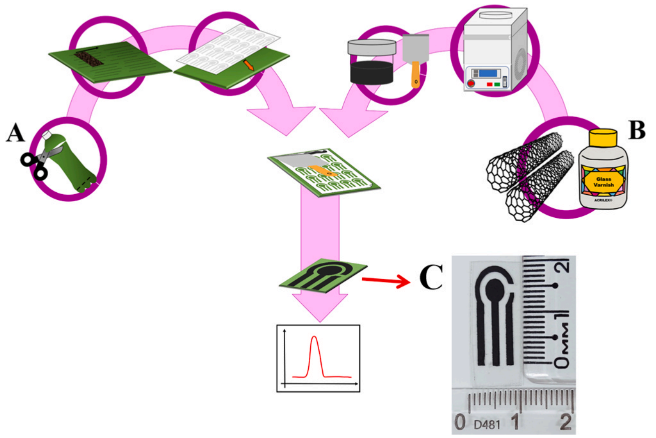

Fig. 1. Schematic illustration of the preparation of CNTs-GV/PET sensors. (**A**) Cutting beverage bottles, sanding, cleaning with 70 % alcohol solution, and applying the adhesive mask to define the three-electrode system; (**B**) Homogenization and shearing of GV and CNT components, followed by spreading the ink over the adhesive mask. The device is then ready for electrochemical measurements. (**C**) Sensor dimensions compared to a ruler.

was mixed with CNTs using a double asymmetric centrifuge (SpeedMixerTM DAC 150.1 FVZ-K FlackTek Inc., South Carolina, United States) to homogenize and shear the particles. Finally, the conductive ink was spread over the adhesive mask with the help of a spatula, and the adhesive mask was subsequently removed, leaving only the three-electrode system on the PET substrate. After 24 h of drying, the electrochemical sensors were ready for use in electrochemical measurements. Fig. 1C shows a photo of the finished sensor compared with a ruler, illustrating its dimensions (1 cm × 2 cm).

For the morphological characterization of the conductive ink, scanning electron microscopy images were obtained using a ThermoFisher Scientific Prisma E scanning electron microscope at an accelerating voltage of 10 kV in low-vacuum mode (50 Pa). X-ray diffraction (XRD) measurements were performed using a Miniflex II X-ray diffractometer (Rigaku) with a CuKa radiation source, ranging from 2° to 90°, with a step of 0.02° and an analysis time of 10° per minute.

2.4. Electrochemical measurements

Initially, electrochemical investigations were conducted through CV, employing various scan rates and pH values. Before each measurement, the CNTs-GV/PET sensor was subjected to electrochemical conditioning in a 0.1 M PBS (pH 8.0) using four consecutive anodic sweep scans within the potential window of 0 V to + 1.4 V (*vs.* CNTs/GV). The SWV-generated voltammograms underwent baseline correction through the Residual plot adjustment method, using the Moving Average mode with a window size of 1 in NOVA 2.1.7 software. All investigated solutions underwent a 5-minute treatment with pure nitrogen gas to eliminate oxygen. For each analysis, a volume of 50 μL of standard (or sample) solution was drop-cast onto the proposed sensor. The limits of detection (LOD) and quantification (LOQ) were calculated following IUPAC recommendations [46].

3. Results and discussion

3.1. Morphological characterization of the CNTs-GV/PET sensor

To complement the morphological information on the conductive ink already detailed in the work by Carvalho et al. [36], micrographs of the sensor surface were obtained to observe the behavior of the conductive ink after its application on the PET substrate. Fig. 2 shows scanning electron microscope (SEM) images of the CNTs-GV/PET sensor at different magnifications (31 × to 1000 ×). The images reveal a rough topography with a significant presence of CNT agglomerates distributed throughout the working electrode. The PET substrate shows several grooves due to polishing with conventional sandpaper. At different magnifications, it is possible to note that the conductive ink completely covers the surface of the substrate, indicating no uncovered spaces, which confirms that the spreading was satisfactory. Furthermore, the final appearance of the carbon nanotubes exposed on the surface does not occur in the form of tubes, as their name suggests. This observation highlights the difficulty in dispersing carbon nanotubes in polymeric media and their tendency to agglomerate, forming structures similar to tangled strings or a ball of wool. Thus, it is possible to observe more prominent points throughout the topography of the electrode surface [36]. For reference, according to Carvalho et al. [36], the average thickness of the conductive ink film formed on the substrate, as

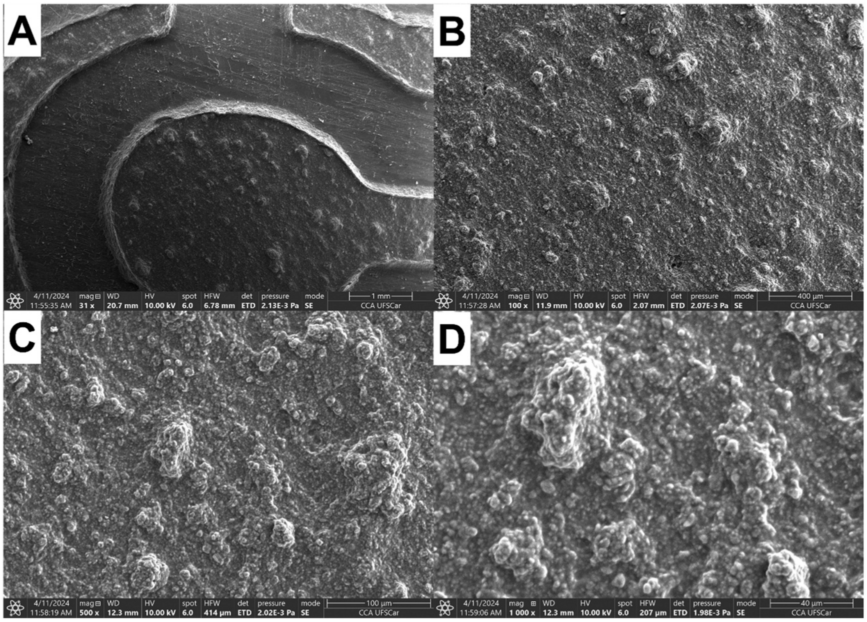

Fig. 2. SEM images of the CNTs-GV/PET sensor at magnifications of 31 ×, 100 ×, 500 ×, and 1000 ×.

determined from SEM images of the working electrode, is 139.4 μm ± 4 μm. However, given the CNT agglomerates at some points, the thickness can reach values close to 190 μm.

In Fig. S2 (Supplementary Information (SI)), X-ray diffraction (XRD) data for the GV (black), the CNTs-GV composite (red), and the CNT powder (blue) are presented. The spectrum obtained for GV (black) demonstrates the expected amorphous behavior for this type of resin. For CNT powder, its spectrum demonstrates characteristic multilayer carbonaceous structures, such as the presence of an intense peak close to 26° corresponding to the (002) plane, similar to graphite, and the presence of peaks closes to 45° and 55°, relative to planes (100) and (004), respectively [47,48]. Additionally, the CNT powder (blue) spectrum features a significant peak at 25.04°, with a crystallite size of 2.6 nm. The spacing between layers obtained by the Bragg's law equation [49] was equal to 0.355 nm, a value very close to the theoretical value of 0.34 nm [50]. For the CNTs-GV composite spectrum (red), we observed the plane of the CNTs shifted slightly to 25.7° without a significant difference in the interlayer spacing.

3.2. Electrochemical behavior of 4-MPD on CNTs-GV/PET

The electrochemical behavior of 4-MPD (100 μmol/L) was investigated by studying the CVs within a pH range of (2.0 − 12.0) using a 0.1 mol/L BR buffer solution (Fig. 3). The influences of pH on peak currents (I_p) and peak potential ($Potential_p$) of the 4-MPD redox processes on CNTs-GV/PET were evaluated and are shown in Fig. S3A and S3B, respectively. Fig. S3A reinforces that pH 8 exhibited the most pronounced peak currents, while Fig. S3B shows that all redox processes are pH-dependent.

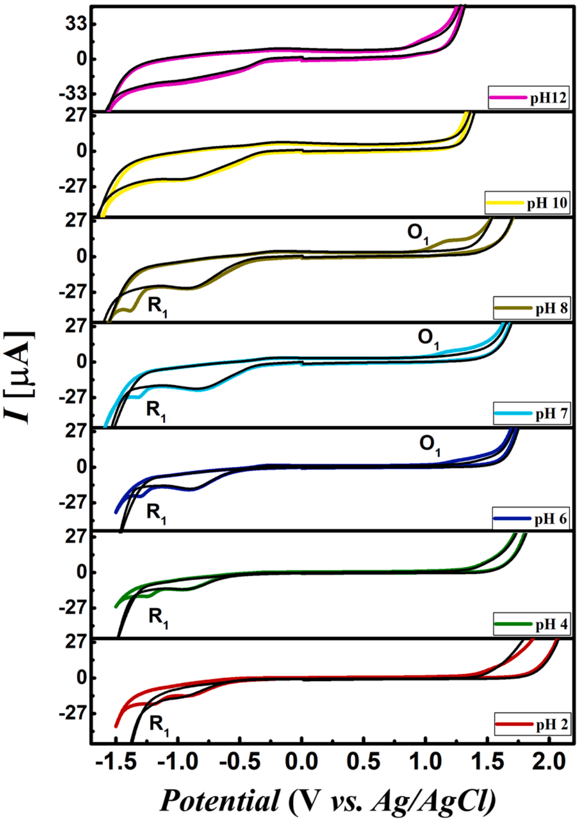

Fig. 3. 2D plot of CVs recorded on CNTs-GV/PET for 100 μmol/L 4-MPD in 0.1 mol/L BR buffer solution at different pH values (2.0, 4.0, 6.0, 7.0, 8.0, 10.0, and 12.0). Black lines show the blank voltammograms. All potential scans started at 0.0 V in the negative-going direction with a scan rate of 50 mV/s. Two redox processes obtained for 4-MPD are labeled as R_1 e O_1, which are only highlighted (inset) at the pH values where they were clearly exhibited on CNTs-GV/PET.

First, it is noteworthy that the CNTs-GV/PET exhibits a characteristic reduction peak between −0.50 and −1.00 V (*vs.* Ag/AgCl), which is also observed in the blank measurements in Fig. 3. Since all solutions were rigorously deaerated, this process is likely associated with functional groups on working electrode surface. Furthermore, Fig. 3 shows that 4-MPD presents two distinct electrochemical processes on CNTs-GV/PET, with reduction (R_1) and oxidation (O_1) peak currents occurring at around −1.3 V and + 1.2 V (*vs.* Ag/AgCl(sat)), respectively. As observed in Figure S4, the I_{R1} *vs.* $v^{1/2}$ plot (Fig. S4C) and log I_{R1} *vs.* log v plot (Fig. S4D) exhibited linear regressions ($R^2 > 0.99$). Based on these investigations, the electrochemical process of 4-MPD is diffusion-controlled on the CNTs-GV/PET surface, as the peak currents were more linearly proportional to the square root of the scan rate (Figure S4C) than to the direct scan rate (Figure S4B), which did not exhibit linear behavior. Furthermore, the log I_{R1} *vs.* log v plot (Figure S4D) showed a linear regression with a slope close to 0.5, again indicating that the reduction process of 4-MPD on the CNTs-GV/PET surface is indeed predominantly diffusion-controlled.

According to its pH distribution and the obtained studies by CV (Fig. 3), when 4-MPD is fully deprotonated, its electrochemical processes cannot be favored on CNTs-GV/PET, explaining why they were not observed beyond pH 10.0 (Fig. 3). On the other hand, when 4-MPD is still protonated in a basic medium, redox processes on CNTs-GV/PET are favored, exhibiting lower peak potentials and higher peak currents at pH 8.0. Thereby, pH 8.0 was chosen for 4-MPD detection using the proposed method.

Subsequently, phosphate, BR, and McIlvaine buffer solutions were assessed as supporting electrolytes for the detection of 4-MPD at pH 8.0. The phosphate buffer solution (PBS) was selected for further analyses, since this supporting electrolyte is easier to prepare and demonstrated a slightly higher peak currents for 4-MPD at CNTs-GV/PET than the other buffer solutions. Under these conditions, a CV of 500 μmol/L 4-MPD is shown in Fig. 4, with a proposed mechanism for all electrochemical processes of this SC on CNTs-GV/PET.

As can be seen in Fig. 4A, 4-MPD exhibited a second reduction process (R_2) at −1.74 V (vs. CNTs-GV), which was not previously observed in BR solution. Fig. 4B shows a proposed mechanism for the electrochemical reactions of 4-MPD on CNTs-GV/PET, including oxidation (O_1) and reduction processes (R_1 and R_2). The oxidation (O_1) and reduction (R_1) mechanisms were based on previously reported reaction pathways for other SCs, such as ethylone, mephedrone, and N-ethylpentylone [32,51,52]. In these works, both O_1 and R_1 electrochemical processes presented independent and irreversible electrochemical behavior using different working electrodes, as also observed on CNTs-GV/PET. As shown in Fig. 4B, the oxidation process (O_1) generates a demethylated analog, which is formed by the oxidation of the secondary amine and subsequent hydrolysis of the intermediate imine. On the other hand, the R_1 process involves reduction of the ketone to a secondary alcohol derivative. The R2 mechanism for synthetic cathinones has not been previously proposed. We suggest that it may involve alcohol dehydration to form an alkene [52,53], followed by subsequent reduction to an alkane [54].

3.3. Detection and quantification of 4-MPD by SWV

The instrumental parameters for 4-MPD detection by SWV were studied concerning the sensitivity and stability of the electrochemical response for the oxidation of this drug on the CNTs-GV/PET surface. The SWV parameters were selected based on the optimal response, determined by the ratio of peak current to peak width at half-height. An amplitude of 90 mV, a step potential of 8 mV, and a frequency of 30 Hz were chosen accordingly (Fig S5). Under these optimal conditions, sensitivity was considerably improved with well-defined peaks observed for all electrochemical processes of 4-MPD (50 μmol/L) on CNTs-GV/PET, as shown in Fig. 5B.

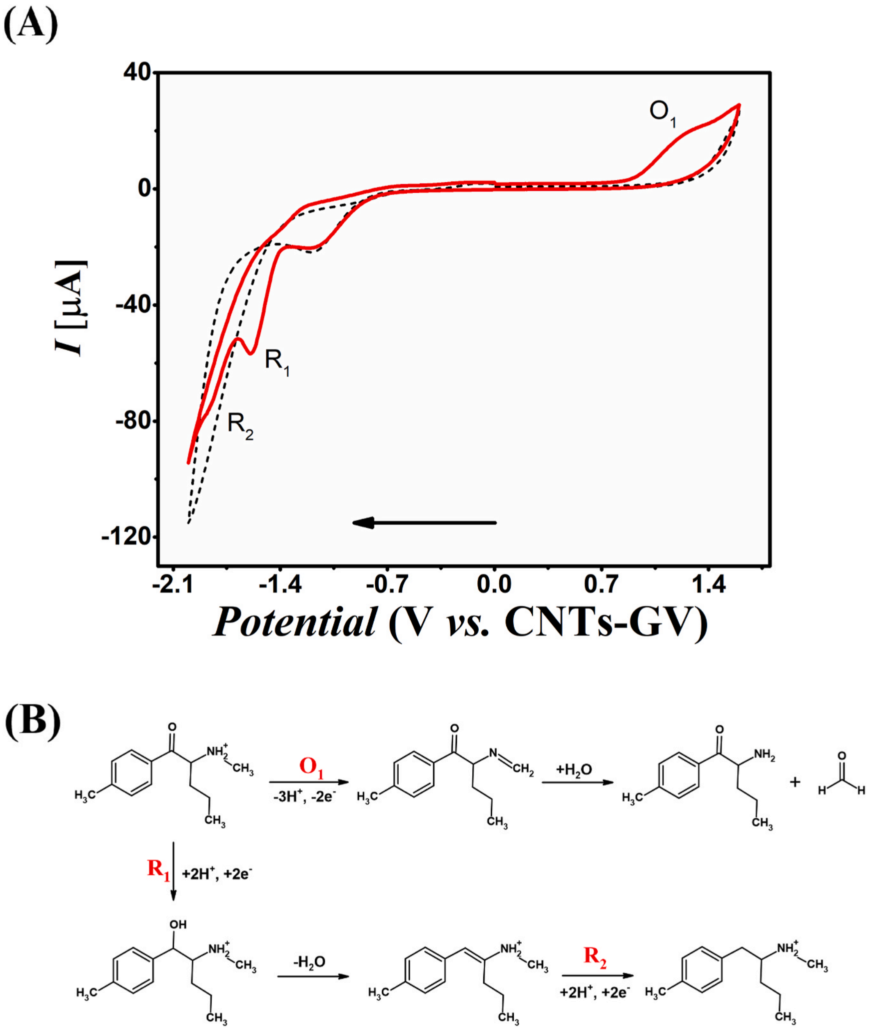

Fig. 4. (**A**) CV recorded on CNTs-GV/PET in 0.1 mol/L PBS at pH 8.0 before (black-dashed-line) and after the addition of 500 μmol/L 4-MPD (red-line). All potential scans started at 0.0 V in the negative-going direction with a scan rate of 50 mV/s. The three redox processes obtained for 4-MPD are labeled as O_1, R_1 and R_2. (**B**) Proposed reaction mechanism for 4-MPD oxidation (O_1) and reduction processes (R_1 and R_2) at CNTs-GV/PET.

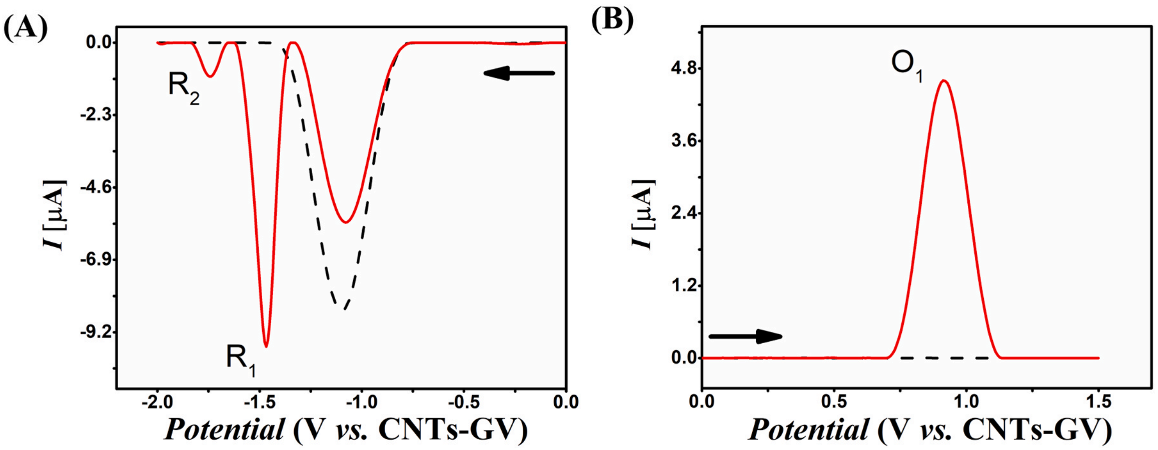

Fig. 5. SWV in 0.1 mol/L PBS at pH 8.0 on CNTs-GV/PET before (black-dashed-lines) and after addition of 50 μmol/L 4-MPD (red-lines), starting at potential 0.0 V in the cathodic direction (**A**) and in the anodic direction (**B**). Experimental conditions: amplitude of 90 mV, step potential of 8 mV, and frequency of 30 Hz.

The CNTs-GV/PET sensor exhibits good potential stability and intra- and inter-sensor reproducibility, similar to other electrochemical devices with carbon-based pseudo-references [42,53–56]. A common issue with all electrochemical devices that use pseudo-reference, including CNTs-GV/PET, is the lack of thermodynamic equilibrium. Therefore, different CNTs-GV/PET sensors can cause a displacement of peak potentials, which may lead to misinterpretation in forensic samples analyses, as peak potential values are crucial for the preliminary identification of drugs such as 4-MPD and other SCs. In this sense, as the CNTs-GV/PET sensor presents an intrinsic reduction process at −1.10 V (Figs. 3 and 5), this electrochemical response can be used as an internal standard to correct the shift of peak potentials of 4-MPD and other drugs. This strategy significantly reduces peak potential variations when using different CNTs-GV/PET sensors, as well as, variability in electrochemical responses obtained from the same device. In order to confirm this hypothesis, a test was performed with a redox probe, 1.0 mmol/L [Fe(CN)6] $^{3-/4-}$, using three different CNTs-GV/PET sensors (Fig. S6). As can be seen in Fig. S6, the potential variation (ΔE) between the intrinsic reduction process on CNTs-GV/PET and the peak potentials (oxidation and reduction) from the redox probe remained practically constant for both CV and SWV techniques. Thereby, indeed, the intrinsic peak potential on CNTs-GV/PET surface can be used as an internal standard, enhancing reliability in the preliminary identification of 4-MPD and other SCs in seized samples. In addition, this strategy could be extended to various applications using different electrochemical sensors.

Under optimized conditions, the working linear range for 4-MPD detection was investigated in triplicate at concentrations ranging from 1.0–100.0 µmol/L. Fig. 6 shows the obtained SWVs and the corresponding working linear ranges for the electrochemical processes of 4-MPD on CNTs-GV/PET.

As can be seen in Fig. 6A, for the R_1 of 4-MPD, a linear range was obtained between 2.5 and 100 µmol/L ($R^2 = 0.997$, I_{pc1} (µA) = -4.8 × 10^{-7} (± 0.2 × 10^{-7}) - 0.26 (± 0.01) (µA/µmol/L) [4-MPD]). As shown in Fig. 6B, two distinct linear ranges were obtained for the O_1 of 4-MPD: (I) between 1.0 and 17.5 µmol/L ($R^2 = 0.992$, I_{pa} (µA) = -1.86 × 10^{-7} (± 0.1 × 10^{-7}) + 0.28 (± 0.01) (µA/µmol/L) [4-MPD]) and (II) between 25.0 and 100.0 mol/L ($R^2 = 0.965$, I_{pa} (µA) = 3.36 × 10^{-6} (± 0.6 × 10^{-6}) + 0.08 (± 0.01) (µA/µmol/L) [4-MPD]).

The theoretical limits of detection (LOD) for 4-MPD determination were calculated to be 0.25 µmol/L and 0.12 µmol/L, for the R_1 and O_1 processes, respectively. The theoretical limits of quantification (LOQ) were established at 0.83 µmol/L and 0.40 µmol/L, for the R_1 and O_1, respectively. It is worth highlighting that the lowest mensurable signals, detected at 2.5 µmol/L for R_1 and 1.0 µmol/L for O_1 (zoom-in of Fig. 6), are sufficiently low to access 4-MPD and other SCs in seized samples.

The first reduction process (R_1) of 4-MPD was used to investigate the repeatability and reproducibility of the proposed method. Three concentration levels within the working linear range for 4-MPD determination were evaluated (Fig. 7). The experiments involved three analyses for each selected concentration, conducted not only using the same sensor but also with three different CNTs-GV/PET sensors. The tested concentration levels were 15 µmol/L, 60 µmol/L, and 90 µmol/ L. The peak potential obtained for each concentration are shown in Fig. 7A, 7B, and 7C, respectively. The peak currents corresponding to these analyses are presented in Figures S7A, S7B, and S7C, respectively.

Fig. S7 shows that the peak currents, I_p, for the R_1 of 4-MPD presented reasonable stability, with a relative standard deviation (RSD) < 5.9 % when using the same CNTs-GV/PET sensor and RSD < 4.2 % when using different CNTs-GV/PET sensors. Additionally, Fig. 7 shows that the peak potential for R_1 on CNTs-GV/PET remained consistent across all measurements (RSD ≤ 2.1 %), indicating that this variation is sufficiently low to enable a reliable preliminary identification of 4-MPD in forensic samples based on peak potential. Furthermore, these RSD values are even lower when considering the Δ peak potential – the difference between the intrinsic reduction peak potential on CNTs-GV/PET and the R_1 peak potential of 4-MPD. Thereby, SWV with CNTs-GV/PET can be effectively utilized as a screening method for the identification of 4-MPD and other SCs in seized samples.

3.4. *Interference studies*

The apprehended samples containing drugs of abuse, such as SCs, have been reported with the presence of different adulterants and/or other illicit substances [57]. These mixtures of substances can be related to many factors, such as enhancing psychotropic effects; masking adverse effects; or reducing production costs. In this context, an interference study was conducted using the proposed method to detect common adulterants, including caffeine and paracetamol, as well as anesthetics including ketamine and lidocaine, and other illicit drugs usually found in samples containing 4-MPD. These interfering substances were selected according to reports from forensic laboratories and drugsdata.org, an independent laboratory drug analysis program of the Erowid Center [57]. In addition, the electrochemical profiles of other structurally similar SCs, such as mephedrone and dibutylone, were examined in order to compare their potential as interfering substances in the proposed method. The electrochemical reponses for 4-MPD detection by SWV, obtained from anodic and cathodic scans on CNTs-GV/PET, were evaluated separately (Fig. 8).

In Fig. 8, the dashed lines represent the average peak potential for each electrochemical process of 4-MPD on CNTs-GV/PET (R_1 at −1.47 V, R_2 at −1.74 V, and O_1 at +0.92 V (all *vs.* CNTs-GV)). Based on these values, Fig. 8 shows symbols ✔ and ✖, indicating the absence or

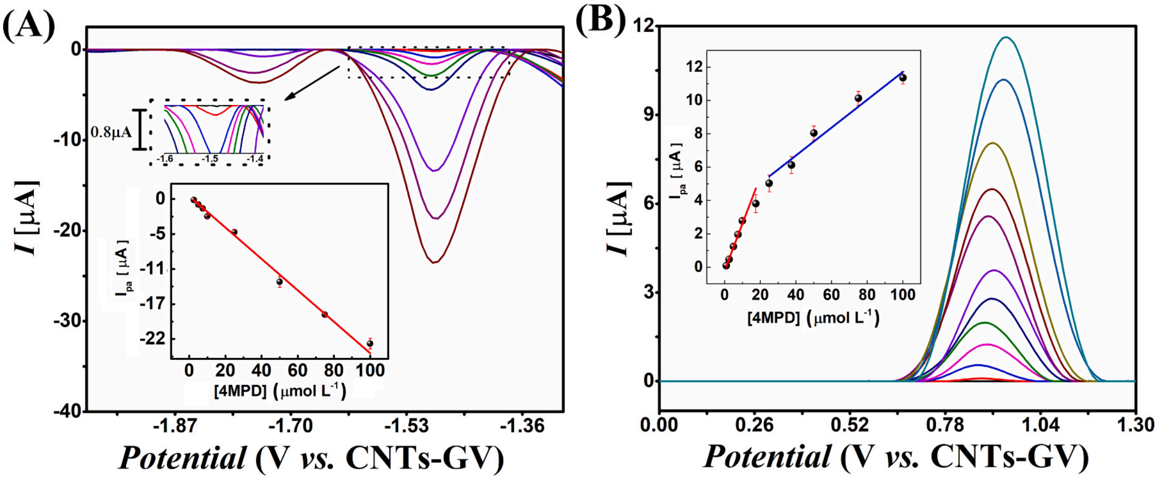

Fig. 6. SWVs in 0.1 mol/L PBS at pH 8.0 on CNTs-GV/PET before and after the addition of 2.5–100 µmol/L 4-MPD (N = 3) for the cathodic scan (**A**) and 1.0–100 µmol/L 4-MPD for the anodic scan (**B**). Insets show the corresponding linear regressions and zoom-in of the lowest measurable signals. Experimental conditions: amplitude of 90 mV, step potential of 8 mV, and frequency of 30 Hz.

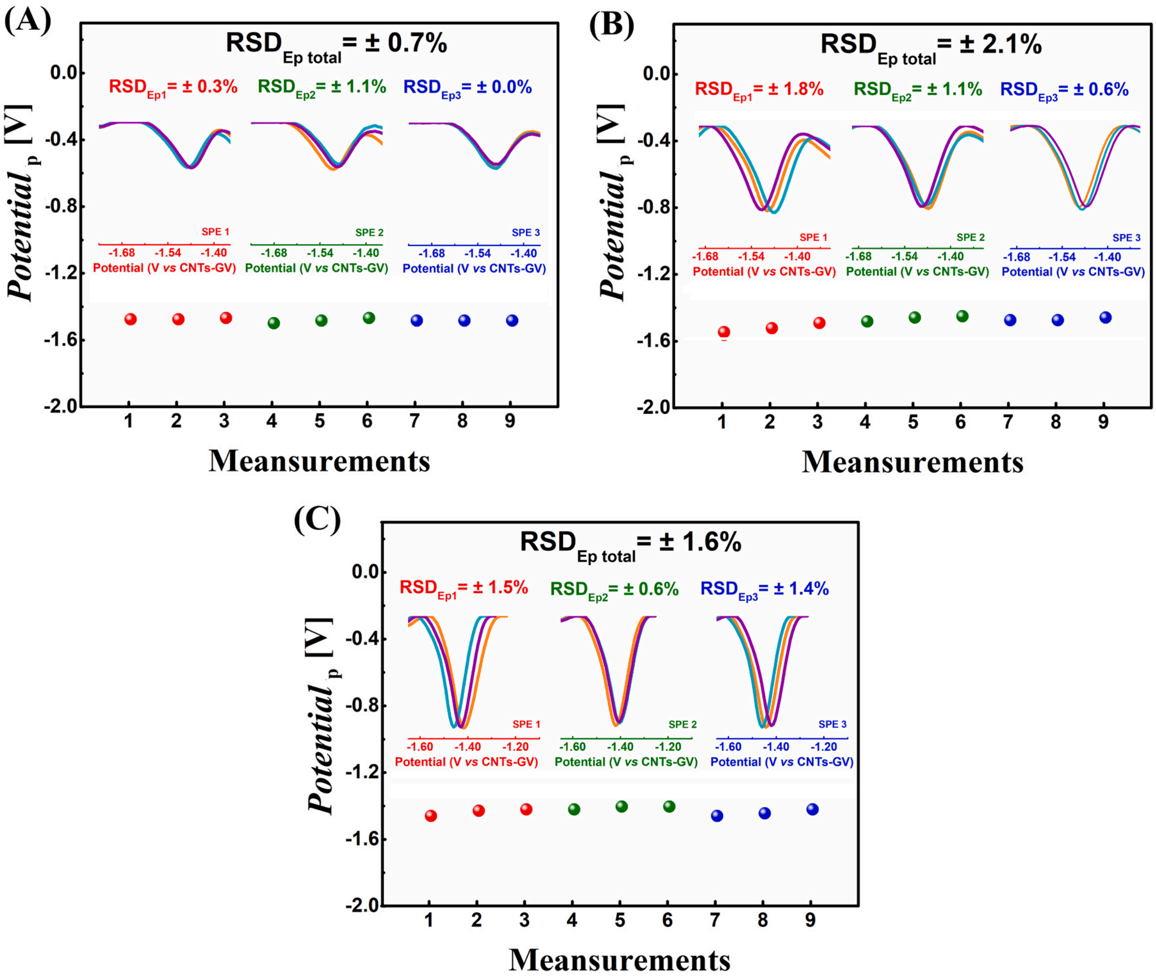

Fig. 7. Plots of peak potential (Potential$_p$) *vs* the number of measurements, conducted in triplicate on three different CNTs-GV/PET sensors at three concentration levels of 4-MPD: 15 µmol/L (**A**), 60 µmol/L (**B**), and 90 µmol/L (**C**) in 0.1 mol/L PBS at pH 8.0. The insets depict respective SWVs and RSDs from Potential$_p$ obtained for the R$_1$ of 4-MPD. Experimental conditions: amplitude of 90 mV, step potential of 8 mV, and frequency of 30 Hz.

presence of a potential interference, respectively, in 4-MPD identification by the proposed method. In this sense, as shown in Fig. 8, the investigated adulterants, caffeine and paracetamol, do not interfere with 4-MPD detection by SWV, regardless of whether the anodic (Fig. 8A) or cathodic (Fig. 8B) scans are used. However, other common illicit drugs, such as BZP, mCPP, and cocaine, could yield false-positive results for the identification of 4-MPD using O$_1$ (Fig. 8A). Nevertheless, it is still possible to selectively identify 4-MPD based on its reduction processes (R$_1$ and R$_2$) on CNTs-GV/PET, even in the presence of these substances.

Amphetamine-like drugs and anesthetics exhibited oxidation processes on CNTs-GV/PET, as shown in Fig. S8A and Fig. S9A, respectively. In this sense, 4-MPD identification using O$_1$ on CNTs-GV/PET is only possible when these drugs do not present overlapping electrochemical processes with this SC. This is the case for A and MA (Fig. S8A), and lidocaine (Fig. S9A). Conversely, the presence of other substances such as MDMA, MDEA, procaine, benzocaine, and ketamine could produce false-positive results when identifying 4-MPD in seized samples using O$_1$.

On the other hand, except for ketamine, all the studied amphetamines and anesthetics did not present reduction processes on CNTs-GV/PET (Fig. S8B and Fig. S9B), enabling a reliable preliminary identification of 4-MPD in the presence of these drugs. In addition, ketamine does not interfere with 4-MPD identification, since their reduction processes do not overlap on CNTs-GV/PET (Fig. S9B).

As expected, the other SCs also exhibited oxidation (Fig. S10A) and reduction (Fig. S10B) processes on CNTs-GV/PET, reflecting their structural similarities. Despite this, SCs can exhibit electrochemical processes at different peak potentials on CNTs-GV/PET, making it possible to differentiate 4-MPD from some SCs, mainly using its R$_2$ peak potential (Fig. S10B). With the exception of dibutylone, none of the investigated SCs posed significant interference. It is worth mentioning that the presence of 4-MPD in seized samples with any other SC has not been reported, and mixtures of SCs are rarely found in forensic samples [57]. In summary, the interference studies demonstrate that the proposed method provides a selective detection of 4-MPD, even in the presence of multiple adulterants and illicit substances.

3.5. Application in forensic samples

The applicability of the proposed method was examined using an authentic seized sample containing 4-MPD, previously confirmed by the Civil Police of the Federal District, Brazil. Fig. S11 illustrates that the investigated sample displayed an electrochemical behavior resembling that of 4-MPD on CNTs-GV/PET, with its peak potentials aligned at R$_1$ and R$_2$ (Fig. 5A), as well as O$_1$ (Fig. 5B). Consequently, this similar electrochemical profile indicates the presence of 4-MPD in the seized sample, as previously confirmed by LC-Q-TOF-MS. Additionally, an addition-recovery study was conducted on the seized sample (Fig. S11), yielding recovery values between 109 % and 118 %, suggesting the presence of matrix effects when quantifying 4-MPD using an external calibration curve. Thereby, a standard addition calibration curve was used in the proposed method for quantifying 4-MPD in the seized

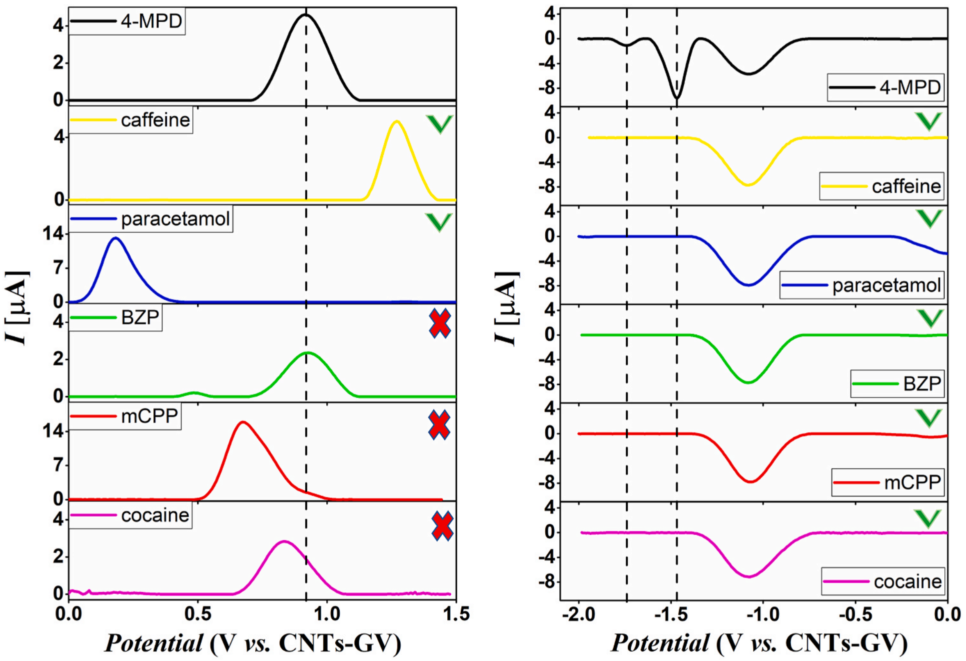

Fig. 8. SWVs on CNTs-GV/PET recorded separately by anodic (**A**) and cathodic (**B**) scans in 0.1 mol/L PBS at pH 8.0 with 50 μmol/L 4-MPD, caffeine, paracetamol, BZP, mCPP, and cocaine. Experimental conditions: amplitude of 90 mV, step potential of 8 mV, and frequency of 30 Hz.

sample, resulting in a measured concentration of 17.1 ± 0.4 mg per tablet, which is in agreement with the reported values for other SCs in similar samples [57,58]. In addition, regarding the detection range, the real sample required a 15,000-fold dilution to bring the response current within the linear range. This implies that 4-MPD could still be detected even in a tablet containing 1500 times less of the substance if only a 10-fold dilution was performed in the supporting electrolyte.

It is worth highlighting that this study presents, for the first time, the electrochemical behavior of 4-MPD and its determination in forensic samples using an analytical method. Due to the novelty of this investigation, a comparison with previous electroanalytical methods was not possible. On the other hand, the proposed method offers two important advantages compared to other reported electrochemical sensors for SCs detection: the use of a low-cost and environmentally friendly sensor, and the potential for routine forensic analysis.

4. Conclusions

The electrochemical behavior and detection of 4-MPD are presented for the first time using a simple and sustainable screen-printed electrode. The proposed lab-made sensor, fabricated from recycled soda bottles, utilizes conductive carbon nanotube ink and stained-glass varnish (CNTs-GV/PET), demonstrating an environmentally friendly approach to forensic electroanalysis. The electrochemical profile of 4-MPD exhibited two reduction processes and one oxidation process on CNTs-GV/PET, enabling a selective preliminary identification of this drug in seized samples, even in the presence of several substances or structurally similar SCs. In addition, the intrinsic electrochemical process of the CNTs-GV/PET sensor serves as an internal standard, correcting peak potential shifts of 4-MPD, thereby providing a highly reliable presumptive test. The CNTs-GV/PET sensor with SWV demonstrated high sensitivity, with theoretical LODs of 0.25 μmol/L (R_1) and 0.12 μmol/L (O_1), values that are sufficiently low for 4-MPD identification in seized samples. Therefore, the generic approach presented in this article can be successfully applied for the preliminary identification of 4-MPD and other SCs in seized samples. Furthermore, the proposed method offers a simple, fast, and cost-effective screening test for application in forensic analysis.

CRediT authorship contribution statement

Lomba Kaliston A.: Writing – original draft, Methodology, Investigation, Conceptualization. **Carvalho Jefferson H.S:** Writing – original draft, Methodology, Investigation. **Pimentel Dilton M.:** Supervision, Methodology, Investigation. **Wallans T.P.dos Santos:** Writing – review & editing, Visualization, Supervision, Resources, Project administration, Conceptualization. **Lívia M. F.Costa:** Methodology, Investigation. **Arantes Luciano C.:** Writing – review & editing, Methodology, Investigation, Conceptualization. **Janegitz Bruno C.:** Writing – review & editing, Visualization, Supervision, Resources, Project administration, 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.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.snb.2025.137513.

Data availability

Data will be made available on request.

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