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ARTICLE

Fabrication of disposable electrochemical devices using silver ink and office paper

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Disposable electrochemical devices are widely used nowadays for in-field applications. This paper reports a novel and simple approach to fabricate a three-electrode configuration electrochemical cell using an office paper instead of a chromatographic paper. This approach combined established wax printing, office paper, and silver ink to transform the office paper into an electrochemical device, thus decreasing the cost of fabrication by 96.7% compared to that using chromatographic paper. Moreover, we demonstrated that the office-paper electrochemical device is suitable for the quantification of different analytes (e.g., heavy metal ions, halides, and explosives) in aqueous solutions. The manufacturing process shown here is extremely cost efficient and uses off-the-shelf products.

1. Introduction

The applications of paper-based devices in resource-limited settings as well as in emergency cases, military field assignments, and home healthcare are gaining importance globally.¹⁻³ These paper-based devices are mostly common used for colorimetric analysis using an optical-imaging based detection system and they are user-friendly and can be used in remote areas.¹⁻⁵

Chromatography paper has become a promising platform for lab-on-a-chip devices for performing large scale and complex laboratory tests. They are portable, low cost, disposable and facilitate on-site real-time detection in many applications such as in the clinical, food, and environmental sectors where simple and practical analytical devices are required. Moreover, the ability to modify paper with a variety of reagents, thus offering better alternatives over traditional glass- and polymer-based devices^{6, 7}. The increasing demand for point-of-care diagnostics to obtain test results rapidly can potentially be met by paper-based sensors.⁸ In addition, chromatography paper has already been used as a platform for electroanalysis.^{5, 9-12} Electrochemical sensing of glucose, lactate, uric acid, cholesterol, tumour markers, dopamine, and drugs^{10, 13-15} has been reported.

Two main ways to build paper-based devices involve the delimitation of hydrophilic and hydrophobic (waterproof) regions for the reaction using photolithography and screen-printed contacts.^{1, 16}

The photolithographic method is expensive and has complex operation, and the devices are damaged by bending and folding. In screen-printing technology, a design is imposed on a silk screen or other fine mesh with blank areas (where no transfer is intended), coated with an impermeable film, by forcing ink through the mesh onto the printing surface. Screen-printing is used for the fabrication of biosensors and chemical sensors because of miniaturization, versatility, low cost, and the possibility of mass production.¹⁴

Reduced volume consumption, low waste production, and great device portability lead to the fast development of paper-based microfluidic devices. Moreover, the possibility of integration of many analytical procedures in a single device¹ and the ease of fabrication using wax printing technology^{2, 3} resulted in the development of these devices.

Herein, we report a novel electrochemical disposable device (three-electrode configuration) on white office paper by combining the screen-printing (using silver ink) and wax-printing technologies. The use of office paper (HP® Office Paper with 2,500 sheets, 8.5 × 11 inches (593,725 cm², 4.7 × 10⁻⁵ US\$ cm⁻²)) instead of chromatography paper (Whatman® cellulose chromatography paper 1 CHR, US \$28.80 (20,000 cm², 0.00144 US\$ cm⁻²)) reduced the cost of the fabrication method by 96.74%. Moreover, office paper are easily available than chromatography paper.

The applicability of the office-paper electrochemical device was demonstrated for the quantification of different analytes (e.g., heavy-metal ions, halides, and explosives) in aqueous solutions.

2. Experimental

2.1. Material and methods

All the solid reagents were of analytical grade and used as received without further purification. Potassium chloride, sodium acetate, acetic acid, sulphuric acid, and lead nitrate were obtained from Merck (Darmstadt, Germany). The silver conductive ink and picric acid were obtained from Joint Metal Comércio LTDA (São Paulo, Brazil) and Reagen (Rio de Janeiro, Brazil), respectively. The solutions were prepared by dissolving the reagents in deionized water, processed through a water purification system (Direct-Q® 5 Ultrapure Water Systems, Millipore, MA, USA).

2.2. Fabrication and design of office paper-based electrochemical device

The key factors in the proposed fabrication protocol are the remarkable simplicity of the fabrication method as well as the low

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cost of office paper. Fig. 1 shows the steps involved in the fabrication of the three-electrode sensor on the office paper using a HP® Office White Paper (size: $8.5 \times 11 \text{ in}^2$). First, we printed a specific pattern on the office paper using a Xerox ColorQube 8570 printer from Xerox (Norwalk, Connecticut, USA). The specific pattern consisted of white circles with a diameter of 1.6 cm. The printed sheets were then placed in a thermal press (Hobby Line Metalnox, Santa Catarina, Brazil) for 3 min at $120 \text{ }^\circ\text{C}$. The heating process melted the deposited wax that then penetrated all the layers in the paper, forming a hydrophobic barrier around the hydrophilic spots, Fig. 1, Step 1. In a single sheet of paper, 72 spots were printed, thus affording 72 paper-based electrochemical disposable devices.

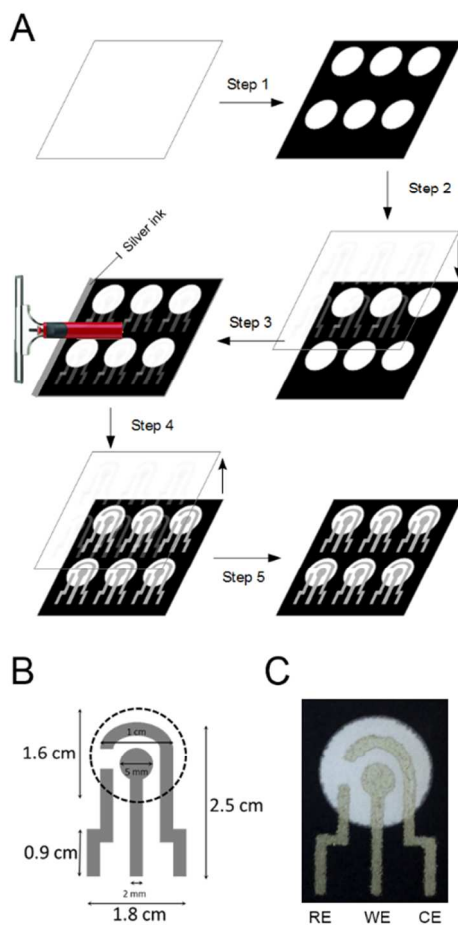


Fig. 1 Steps involved in the fabrication of silver office paper-based electrochemical cell by screen-printed technology (A), a schematic layout of electrodes printed on the office paper (B), and a real picture of the paper-based electrochemical cell, where WE is the working electrode, CE is the counter electrode, and RE is the reference electrode (C).

For the screen-printing, a template mask with the electrodes layout (Fig. 1B) was created using a CorelDraw® software and cut using a gravograph laser cutter machine (Gravograph, La Chapelle-St-Luc, France). The template mask was then fabricated on a transparent polycarbonate sheet. After the fabrication, the template mask was placed over the printed office paper (Fig. 1, Step 2) with the wax, and the silver conductive ink was poured onto the template mask and spread with a squeegee (Fig. 1, Steps 3 and 4). In Step 5, the template mask was removed, and the silver ink was dried at $70 \text{ }^\circ\text{C}$ for 1 h. After the drying, the individual disposable devices were cut

with a scissor. Fig. 1C shows a real picture of the office paper-based electrochemical cell. Before used the office paper-based device, the thickness of the silver ink layer was measure using a Micrometer from Mitutoyo Corporation (Kawasaki, Japan) in order to guarantee the reproducibility of the ink deposition. Only devices with a silver ink layer thickness range of $(14.7 \pm 0.5) \mu\text{m}$ were used. When the fabricated devices were not immediately used after the fabrication process, they were stored in dry and low temperature ($4 \text{ }^\circ\text{C}$) and used up to 48 hours to prevent spoilage by oxide formation. Additionally, all sensors were disposable, i.e., they were used only once for a single experiment.

2.3. Electrochemical Analysis

The paper-based electrochemical cell was connected to the potentiostat using three minialligator clips. All analyses performed in this work were carried out using additions of $10 \mu\text{L}$ stock solution in $200 \mu\text{L}$ of appropriate electrolyte (acetate buffer or acidic solution) in each paper-based electrochemical cell developed. High volumes can be used, but is necessary to increase the diameter of the spots printed, since the drop formed not scattered by office paper due to its low wettability.

2.4. Instrumentation

The electrochemical measurements were carried out using a μ -Autolab III potentiostat (Eco Chemie, The Netherlands) with data acquisition software available from the manufacturer (GPES 4.9.007 version).

3. Results and discussion

3.1. Electrochemical characterization of paper-based electrochemical devices

$[\text{Ru}(\text{NH}_3)_6]\text{Cl}_3$ was used as the model redox-active compound to characterize the electrochemical behaviour of the silver office paper-based electrochemical cell (Fig. 2). The shape of the cyclic voltammetry peak shows a typical reversible (Nernstian) electrochemical reaction, whose rate at the surface of the electrode is governed by the diffusion of the electroactive species to the surface of the planar electrode.¹⁰

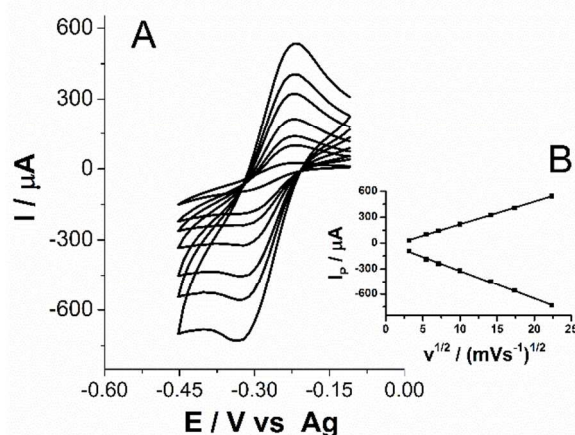


Fig. 2 (A) Cyclic voltammograms obtained using the silver office paper-based device, fabricated as shown in Fig. 1, at different scan rates ($10, 30, 50, 100, 200, 300,$ and 500 mV s^{-1}) in a 0.1 mol L^{-1} acetate buffer ($\text{pH } 4.7$) solution containing 5 mmol L^{-1} ruthenium

hexamine trichloride. The relationship between the anodic and cathodic currents and the square root of the scan rate is shown in the inset (B).

The relationship between the anodic and cathodic currents in Fig. 2B shows that the peak currents for the forward and backward scans are linearly proportional to the square root of the scan rate between 10 and 500 mV s^{-1} . The linearity indicates that the mass transfer in this system is a diffusion-controlled process similar to the behaviour of traditional electrochemical cells, i.e., the device fabricated on the surface of an office paper exhibits the electrochemical behaviour reported by the commercial electrodes or cells. The only noticeable difference when compared with a conventional reference electrode (Ag/AgCl(KCl saturated)) was the shift in the peak potential of 100 mV in the negative potential region, due to the use of a silver pseudo-reference electrode. The active electrochemical area was calculated as 0.63 cm^2 using the slope of the curve, as shown in Fig. 2B, for the forward scan, $D = 9.78 \times 10^{-6} \text{ cm}^2 \text{ s}^{-1}$ for ruthenium hexamine trichloride ($[\text{Ru}(\text{NH}_3)_6]\text{Cl}_3$)¹⁷ and Randles-Sevcik equation.

3.2. Application of the proposed sensor fabricated on office paper

3.2.1. Explosive (picric acid) detection.

The development of methods for the identification and detection of toxic and hazardous compounds such as explosives has advanced significantly because of their enormous applicability in the remediation of explosive manufacturing sites and forensic investigations.^{18, 19}

First, the cyclic voltammogram of picric acid was recorded to evaluate its electrochemical behaviour using the developed sensor. As shown in Fig. 3A, when picric acid was added, a well-defined reduction peak was observed around -0.75 V because of the reduction of the nitro group. In the absence of picric acid, a pair of redox peaks was observed because of the oxidation and reduction of the silver electrodes, and these electrochemical patterns decreased/disappeared by adding picric acid, indicating that picric acid can be adsorbed at the electrode surface and compete with the electrochemical process of the silver dissolution.

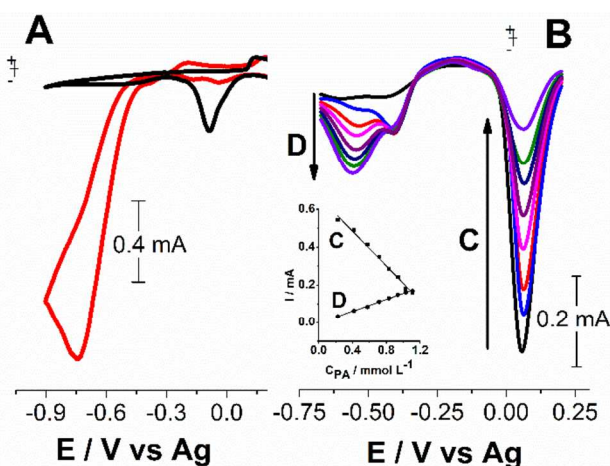


Fig. 3 (A) Cyclic voltammograms recorded in a 0.1 mol L^{-1} acetate buffer solution ($\text{pH} = 4.7$) in the absence (black line) and presence (red line) of 5 mmol L^{-1} picric acid. Scan rate: 100 mV s^{-1} , (B) Differential pulse voltammograms obtained with silver office paper-

based electrochemical cell in a 0.1 mol L^{-1} acetate buffer solution ($\text{pH} = 4.7$) with successive additions of a 10 μL of picric acid solution resulting in a final concentration of 0.2–1.1 mmol L^{-1} . Parameters: step, 0.03 V and amplitude, 0.05 V. Inset: Analytical curve obtained from the differential pulse voltammograms. Regression linear equation: $I_C \text{ (mA)} = -2.15 \times 10^{-6} + 0.158 (C_{\text{PA}}/\text{mmol L}^{-1}, R^2 = 0.999$ and $I_D \text{ (mA)} = 6.68 \times 10^{-4} + -0.458, C_{\text{PA}}/\text{mmol L}^{-1}, R^2 = 0.996$.

Studies^{19, 20} have shown that at moderately acidic conditions, nitroaromatic compounds are reduced to hydroxylamines in two steps involving four electrons (Eqs. 1 and 2).²⁰ These two processes refer to the peaks observed around -0.15 V (Eq. 1) and -0.75 V (Eq. 2) in the cyclic voltammograms as shown in Fig. 3 (red line).



To improve the sensitivity of the method, differential pulse voltammetry was used, as shown in Fig. 3B, by performing the scan from 0.2 to 0.7 V. The reduction peak of the silver at 0.10 V decreased linearly with increasing concentration of picric acid. Further, a signal for the reduction of the nitro group appeared at -0.6 V that responded linearly to the analyte amount, indicating that these two signals can be used for the quantification of picric acid with a detection limit of less than 30 $\mu\text{mol L}^{-1}$ (Curve D, Fig. 3B).

3.2.2. Environmental (chloride) analysis.

The determination of halide ions is crucial in clinical diagnosis, environmental monitoring, and industrial applications.^{21, 22} Therefore, the potential of silver office paper-based electrochemical cell for the analysis of chloride ions was evaluated, as shown in Fig. 4.

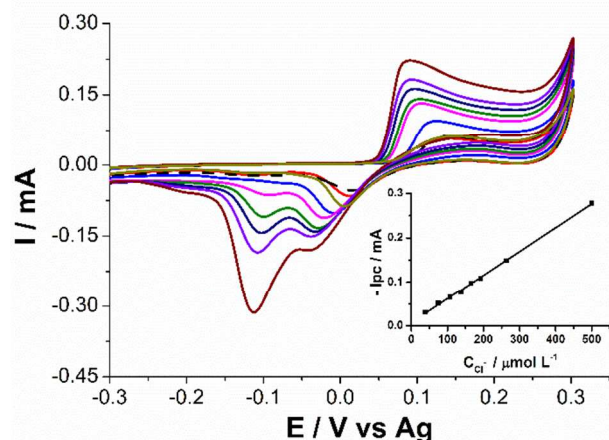


Fig. 4 Cyclic voltammograms recorded in a 0.5 mol L^{-1} sulphuric acid solution using a silver office paper-based electrochemical cell in the absence (dotted line) and presence (solid line) of successive additions of 10 μL of a chloride solution, with a final concentration of 38 to 500 $\mu\text{mol L}^{-1}$ chloride. Scan rate: 50 mV s^{-1} . Inset: analytical curve obtained from the peak current of the reduction of the silver chloride. Regression linear equation: $I \text{ (mA)} = 0.008 + 537 (C_{\text{Cl}^-}/\mu\text{mol L}^{-1}, R^2 = 0.999$.

Figure 4 shows the oxidation peak near 0.1 V corresponding to the formation of a silver chloride precipitate at the electrode surface. In

the reverse scan, a potential was observed for the reduction peak of the silver chloride. Both the peaks can be used for the quantification of chloride ions in aqueous solutions, and a detection limit of $7 \mu\text{mol L}^{-1}$ can be obtained (the slope of the I_{pc} calibration curve was used).

Chiu et al.²¹ reported a voltammetric determination method with well separated redox peaks for different silver halides using a silver substrate as the sensing agent. The well separated peak potentials were found to correlate with the solubility product (K_{sp}), which determines the interaction between the surface silver atoms and the halide anions thermodynamically.²¹

We tested the possibility of using the silver office paper-based electrochemical device for the simultaneous detection of chloride, bromide, and iodide ions (not shown), and the results were found to be in agreement with the literature,^{21, 22} offering a possibility for halide sensing.

3.3.2. Heavy metals quantification.

We also evaluated the potential application of the silver paper-based electrochemical device for the analysis of heavy metals.

Figure 5 shows the potential of the silver office paper-based electrochemical device for the application in the stripping analysis of Pb^{2+} ions. A series of stripping responses for the addition of Pb^{2+} ions obtained over the ranges $9\text{--}48 \mu\text{mol L}^{-1}$ are shown. Each experiment was performed after a collection time of 20 s at -1.0 V deposition potential in a 0.1 mol L^{-1} acetate buffer solution ($\text{pH} = 4.7$), and the electrode was replaced after each run. The proposed method achieved a detection limit of $1.7 \mu\text{mol L}^{-1}$.

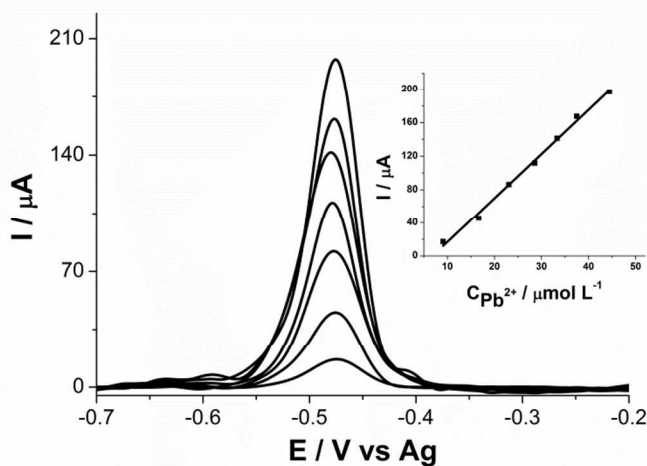


Fig. 5 Square-wave anodic stripping voltammograms (SWASV) obtained with the silver office paper-based electrochemical cell in a 0.1 mol L^{-1} acetate buffer solution ($\text{pH} = 4.7$) with successive additions of $10 \mu\text{L}$ of a picric acid solution with a final concentration of $9\text{--}48 \mu\text{mol L}^{-1}$. SWASV was performed in the potential range of -0.7 to -0.2 V under the following optimized conditions: frequency, 20 Hz ; amplitude, 10 mV ; and step potential, 10 mV . The deposition was performed at -1.0 V for 20 s . Inset: analytical curve obtained from the SWASV. Regression linear equation: $I (\mu\text{A}) = -15.88 + 5.284 (C_{\text{Pb}^{2+}} / \mu\text{mol L}^{-1})$, $R^2 = 0.995$.

3.4 Analytical parameters of the silver office paper-based electrochemical cell

The stability and repeatability tests of the silver office paper-based electrochemical cell were conducted in a 0.1 mol L^{-1} acetate buffer at $\text{pH} 4.7$ by measuring the peak current of the cyclic voltammetry in a 1.0 mmol L^{-1} chloride solution. The paper-based electrode showed a good repeatability for chloride determination. The relative standard deviation (RSD) for 15 determinations of 0.5 mmol L^{-1} was 0.9% . In the stripping analysis, up to four measurements can be carried out on the same device without significant changes in the signal (less than 3%).

For the fabrication process of the devices, a good reproducibility was obtained with a standard deviation of 8% between the constructed electrodes ($n = 25$). This result shows that the variation in the electrode area because of the simplicity of the proposed fabrication. However, to achieve higher precision (below 8%), either the electrode area could be calibrated before the detection using a model redox-active compound or an electroactive internal standard could be used to circumvent the fluctuations in the electrode area.

Some advantages of the proposed device are low cost of the office paper as substrate, as mentioned previously, no necessity of a insulation process with a polymer on the back face of the device to avoid leakage during the measurements^{23, 24} and no deformation of the sensor substrate when wetted by liquid like the devices made with chromatography paper. Table 1 shows a comparison between substrates, electrodes, materials and instrumentation required for fabrication of some paper-based devices reported in the literature.

As reported in Table 1, the proposed method required a less sophisticated procedure compared with other articles reported in the literature. Notwithstanding, cost of the silver ink are higher than graphite pencil as electrode material, but our process of fabrication can also support the graphite pencil approach to fabricate the electrodes. Hence, the choice of the electrode material can be made by the user depending of the analytical problem that will be solved, for example, silver ink for chloride quantification or graphite pencil as amperometric detector for ascorbic acid²⁴.

Regarding the fabrication time, the steps to print a specific pattern and wax procedures are fast. Hence, the limiting step of the fabrication process is the drying of the silver ink. Thus, it is possible to estimate an average time of $60\text{--}65$ minutes to building 72 paper-based electrochemical devices.

In conclusion, the use of white office paper combining screen-printing (using silver ink) and wax-printing technologies provides a low cost procedure associated with a fast fabrication method that makes the proposed method an attractive option for the development of disposable devices for field analysis.

Conclusions

The paper-based electrochemical device developed in this study was successfully used for the analysis of toxic compounds and pollutants in the environment; it has great potential for application in routine analysis in locations with poor laboratory infrastructure, thus offering a lab-on-paper technique. The device is inexpensive compared to the chromatography paper devices, portable, easily fabricated and manipulated, disposable, and it generates low amount of waste. The device can be manufactured in large scale using off-the-shelf products.

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Table 1 - Comparison between substrate, electrodes, material and instrumentation required for fabrication of paper-based electrochemical system reported in the literature.

References	Substrate	Electrodes	Materials and instrumentation required
Dossi et al. ²⁴	Filter paper with polyethylene layer	Pencil-drawn	Wax printing, hot plate, thermal insulation process
Santhiago et al. ²⁵	Chromatographic paper	Graphite pencil electrode (WE) and silver ink	Wax printing, hot plate, transparency sheet
Dungchai et al. ¹³	Filter paper and photoresist	Carbon ink (WE and CE) and silver chloride ink (RE)	Laser printer, Photolithography, photoresist spin-coater, transparency, UV lamp, plasma cleaner
Nie et al. ¹⁰	Chromatography paper and photoresist or polyester-cellulose blend paper	Carbon ink (WE and CE) and silver chloride ink (RE)	Photolithography or wax printing, UV lamp, hot plate
Dossi et al. ²³	Filter paper with polyethylene layer	Dual Pencil-drawn	wax printing, hot plate, thermal insulation process
Present work	Office paper	Silver ink	wax printing, heat press, transparency sheet

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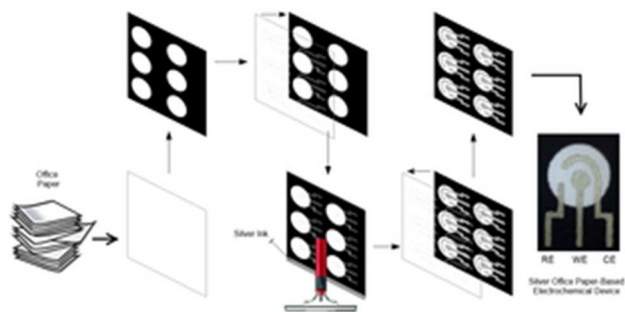
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