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Article

Methanol Oxidation Reaction in Alkaline Media using Gold Nanoparticles Recovered from Electronic Waste

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Abstract: Electronic waste (e-waste), characterized by the widespread disposal of electronic devices, shows a growing environmental challenge. In this study, we explore the electrocatalytic potential of gold nanoparticles supported on Vulcan obtained from e-waste for their application as electrocatalysts for the methanol oxidation reaction in alkaline media. Gold nanoparticles are synthesized from gold coatings recovered from Intel Pentium 4 processor pins, highlighting their sustainability as electrocatalyst precursors. Comprehensive characterization, including UV-Vis spectroscopy, transmission electron microscopy (TEM), scanning electron microscopy (SEM) and X-ray diffraction, is performed to assess the electrocatalyst's structural properties. Electrochemical evaluation is carried out in 0.5 M KOH electrolyte employing the rotating disk electrode technique, using cyclic voltammetry for the study of methanol oxidation. Remarkably, the gold nanoparticles show electrocatalytic activity on par with commercially synthesized counterparts, demonstrating the feasibility of this approach for sustainable electrocatalyst production. Thus, this research opens new routes for utilizing e-waste as a source of valuable catalyst materials, addressing environmental and technological challenges in the field of alkaline fuel cells.

Keywords: electronic waste; gold nanoparticles; electrocatalysis; methanol oxidation; alkaline fuel cells

1. Introduction

In today's technology-driven world, electronic waste, often referred to as e-waste, has become a pressing environmental challenge in our technologically driven world. The eminent proliferation of electronic devices and the rapid pace at which they become obsolete result in a continuous accumulation of discarded electronic components and materials. This poses a dual dilemma: the need for sustainable disposal methods to mitigate environmental damage and the potential for resource recovery from this e-waste [1–5].

Among the main reasons for the increase in e-waste generation are factors such as: inadequate infrastructure, less in-house manufacturing, lack of awareness, non-repair/reuse of devices, to obsolete technology and poor reporting system [6]. Thus, recognizing the importance of e-waste recycling is crucial to address these challenges and promote a more sustainable approach to the management of our technological waste.

On the other hand, the chemical composition of e-waste usually varies depending on factors such as the type and model of the electronic device, its manufacturer, the date of manufacture and

the age of the e-waste. For example, e-waste from computer and telecommunications systems tends to contain a higher concentration of precious metals, including base metals such as copper (Cu) and tin (Sn), and precious metals such as silver (Ag), gold (Au) and palladium (Pd) [7]. In this context, the study of e-waste as a source of valuable materials has attracted great interest.

Moreover, as mentioned above, gold is an essential component of many electronic devices, so it is a clear example of this precious resource embedded in e-waste. The recovery of gold from electronic waste has become an area of intense research interest, driven by the need to address both environmental concerns and the potential for resource utilization. Recycling gold from e-waste involves a series of processes that target the extraction and purification of this precious metal. Several methods have been developed, ranging from chemical leaching, hydrometallurgy and processes using food by-products to more sustainable and environmentally friendly approaches [8–11]. These methods aim to isolate and recover gold from electronic components such as printed circuit boards, connectors, and contact points.

Gold nanoparticles (Au NPs) have generated considerable attention in the field of nanotechnology due to their unique properties and versatile applications, as they exhibit remarkable characteristics that make them valuable in a wide range of areas, including but not limited to: catalysis, as a catalyst in a variety of chemical reactions, including oxidation of organic compounds [12,13], these catalytic properties have made them essential in the development of more environmentally friendly and efficient chemical processes; biomedical applications, gold nanoparticles are integral in cancer therapy and photothermal therapy, where they can be heated by light to destroy cancer cells [14,15]; environmental remediation, they have been employed in the removal of contaminants from water and wastewater, such as heavy metals and organic pollutants [16,17], the catalytic activity of Au NPs aids in the degradation of various pollutants, contributing to cleaner and more sustainable environmental practices; electrochemical applications, used as electrocatalysts for fuel cells, electrochemical sensors, and in the development of energy storage devices [18,19], the high surface area and electrocatalytic properties of Au NPs make them valuable in these applications.

On the other hand, the continuous development of energy conversion technologies is essential to address the growing demand for clean and sustainable energy sources. Alkaline fuel cells (AFCs) have emerged as promising devices for efficient energy conversion, using electrochemical oxidation of fuels, such as hydrogen or methanol, to produce electricity with oxygen reduction at the cathode [20]. AFCs offer several advantages, such as the potential use of non-precious metal catalysts, which can significantly reduce costs. However, the performance of AFCs is highly dependent on the efficiency of the electrocatalysts used, particularly at the anode, where the fuel oxidation reaction takes place. The demand for efficient electrocatalysts for alkaline fuel cells (AFC) has grown in parallel with the search for cleaner and more sustainable energy solutions.

In this context, gold nanoparticles (AuNPs) have attracted much attention due to their remarkable electrocatalytic properties [20–24]. To unlock the latent potential of this resource, we delved into the electrocatalytic characteristics of AuNPs recovered from electronic waste, exploring their suitability as electrocatalysts for the methanol oxidation reaction (MOR) in alkaline media. The use of AuNPs as electrocatalysts, in particular those obtained from e-waste, implies a sustainable approach that aligns with the increasing emphasis on recycling and resource efficiency.

Therefore, this study makes an important contribution to this field by demonstrating the feasibility and sustainability of e-waste recycling for the synthesis of electrocatalysts, specifically gold nanoparticles, with promising electrocatalytic properties, in particular for the methanol oxidation reaction in alkaline media. The research shows the potential of using e-waste as a resource for the production of efficient electrocatalysts, thus addressing environmental concerns and offering a sustainable approach for alkaline fuel cell applications.

2. Materials and Methods

2.1. Synthesis of Au/V NPs

The synthesis of gold nanoparticles supported on Vulcan from electronic waste (Au_{e-w}/V NPs) comprised four distinct stages. Initially, gold coatings from Intel Pentium 4 processor pins were recovered using the methodology proposed by Su et al. [16], involving an acid digestion to selectively dissolve accompanying metals (Cu, Fe, Ni) present in the pins. Subsequently, HAuCl₄ was synthesized from the recovered gold coatings. In this step, 2 mg of gold coatings were weighed and reacted with 5 mL of 10% V/V HCl (37%, Meyer, Mexico City, Mexico) and 2 μ L of concentrated HNO₃ (69%, Meyer, Mexico City, Mexico) in a conventional synthesis reactor (Monowave 50, Anton Paar, NSW, Australia), applying for this purpose a heating ramp up to 160 °C for ten minutes, once this temperature was reached it was maintained for two more minutes. After synthesis, HAuCl₄ was recrystallized in a vacuum desiccator. After evaporating the solvent, HAuCl₄ was transferred to a 10 mL volumetric flask and adjusted with deionized water to obtain a 1 mM concentration. The third part involved the synthesis of gold nanoparticles (Au NPs) from HAuCl₄. For this, 5 mL of the prepared 1 mM HAuCl₄ solution was reacted with 0.5 mL of 38.8 mM sodium citrate (C₆H₅Na₃O₇·2H₂O, 100.1%, J.T. Baker, Quebec, Canada) under conditions identical to those used for HAuCl₄ synthesis. The synthesis process was duplicated, yielding a total of 11 mL of gold nanoparticle solution. Finally, the obtained gold nanoparticles were supported on Vulcan® XC-72 (Cabot, Boston, MA, USA) via magnetic stirring for 24 hours (Au/V NPs). The supported Au NPs were vacuum-dried, resulting in a black powder.

On the other hand, in order to compare the structural and electrocatalytic activity of Au_{e-w}/V NPs obtained from e-waste recycling, Au_{com}/V NPs were synthesized using a commercial precursor (HAuCl₄, 99.99 %, Sigma-Aldrich, St. Louis, MO, USA), at the same synthesis conditions.

In this way, this comprehensive synthesis protocol ensured the production of gold nanoparticles supported on Vulcan for subsequent structural and electrochemical characterization.

2.2. Structural Characterization of Au NPs and Au/V NPs

Unsupported gold nanoparticles (Au NPs) were characterized by UV-vis spectroscopy using a Genesys 150 spectrophotometer (ThermoFisher-SCIENTIFIC, Waltham, MA, USA) within the wavelength range of 250 - 1100 nm.

On the other hand, the supported gold nanoparticles (Au/V NPs) were subjected to comprehensive characterization. Scanning Electron Microscopy (SEM) was performed using a JSM 7800F microscope (SEMTECH Solutions, North Billerica, MA, USA) at 15 kV. Additionally, Energy Dispersive X-ray Spectroscopy (EDS) was conducted with an OXFORD spectrophotometer coupled to the JSM 7800F microscope. Further structural data were obtained by X-ray diffraction (XRD) using a Bruker D8 Discover diffractometer (Cu-K α 1 radiation, 1.54060 Å, Bruker Mexicana, CDMX, Mexico). Finally, Transmission Electron Microscopy (TEM) was employed, using a JEM2010-FEG microscope (Jeol, Tokyo, Japan) at 200 kV.

2.3. Electrochemical Characterization of Au/V NPs

Electrochemical studies were carried out using a three-electrode electrochemical cell. A saturated Calomel electrode was used as reference electrode, a graphite rod as counter electrode, and as working electrode an Au/V NPs electrocatalytic ink deposited on a glassy carbon disk ($\varnothing$ = 5.0 mm). The measured potential values are referred to the reversible hydrogen electrode (RHE). The electrolyte used was a 0.5 M KOH solution ($\geq$ 98%, Sigma-Aldrich, St. Louis, MO, USA). The electrocatalytic ink preparation process consisted of mixing 2 mg of the respective electrocatalyst (Au/V NPs), 298 μ L of deionized water (18.2 M Ω ·cm), 200 μ L of isopropyl alcohol (99.5%, Meyer, Mexico City, Mexico) and 2 μ L of Nafion (5% in aliphatic alcohols, Sigma-Aldrich, San Louis, MO, USA). The mixture was sonicated using a sonicator (Cole-Palmer, IL, USA) until the ink was homogeneously dispersed. Subsequently, 5 μ L of the resulting ink was deposited on the glassy

carbon disk electrode, previously polished using MicroCloth with 5 and 0.3 μm alumina abrasives. Electrochemical data was acquired using a bipotentiostat/galvanostat (PINE, Wavedriver, AFP2, Durham, NC, USA) and commanded by AfterMath® software (v 1.6.10513, Durham, NC, USA) developed by the same company.

Cyclic voltammetry (CV) studies were performed in the absence of methanol in alkaline media to characterize the electrode surface (Au/V NPs ink). For this purpose, the electrolyte was previously purged with pure N_2 (UHP, Praxair, Mexico City, Mexico) for 30 min. Potential sweeps were performed for 16 cycles, from 0.46 to 1.56 V/RHE at a $20 \text{ mV} \cdot \text{s}^{-1}$ scan rate.

For the electrochemical study of the methanol oxidation reaction, CV studies were performed in the presence of methanol at different concentrations (from 1 to 5 M) (99.8%, Sigma-Aldrich, St. Louis, MO, USA) in alkaline media, using the same conditions as in the absence of methanol.

3. Results and Discussion

3.1. Structural Characterization of Au NPs and Au/V NPs

Figure 1 shows a sequence of processes crucial to our study. First, on the left, are gold coatings meticulously recovered from INTEL® PENTIUM® 4 processors, representing a successful recovery of electronic waste. Moving to the center, we observe the synthesis of HAuCl_4 , which results in a distinctive yellow solution. This synthesis is a fundamental step, involving the transformation of the recovered gold coatings. Finally, on the right, we witness the culmination of the proposed synthesis method: gold nanoparticles (NPs) are successfully synthesized from HAuCl_4 , manifesting as a vibrant red-colored solution. This phenomenon arises from localized surface plasmon resonance (LSP), a distinctive feature of gold nanoparticles, which occurs when the frequency of light coincides with the oscillation frequency of electrons in the nanoparticles [25,26].

This process demonstrates a sustainable approach to harness gold nanoparticles from electronic waste, outlining a new avenue for recycling and electrocatalyst production.

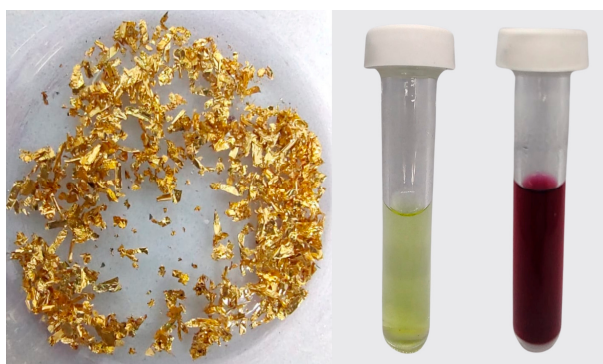


Figure 1. From left to right: Gold coatings, HAuCl_4 (yellowish solution) and unsupported Au NPs (reddish solution) recovered from e-waste.

Figure 2 shows the UV-Vis spectra of gold nanoparticles, with a resonance peak at 526 nm for commercially synthesized gold nanoparticles (Au_{com} NPs) and at 525 nm for those derived from electronic waste ($\text{Au}^{\text{e-w}}$ NPs). This prominent peak is found to be associated with the Localized Surface Plasmon (LSP) resonance, indicative of estimated particle sizes within the range of 15-20 nm [27]. This observation is in agreement with subsequent transmission electron microscopy (TEM) analysis, which confirms the consistency of particle size. Noteworthy is the slightly higher absorbance intensity observed for Au_{com} nanoparticles compared to $\text{Au}^{\text{e-w}}$ nanoparticles, pointing to a potentially high concentration of Au_{com} nanoparticles. This speculation is corroborated by subsequent Energy Dispersive X-ray Spectroscopy (EDS) analysis, which further highlights the differences in hue between the two sources.

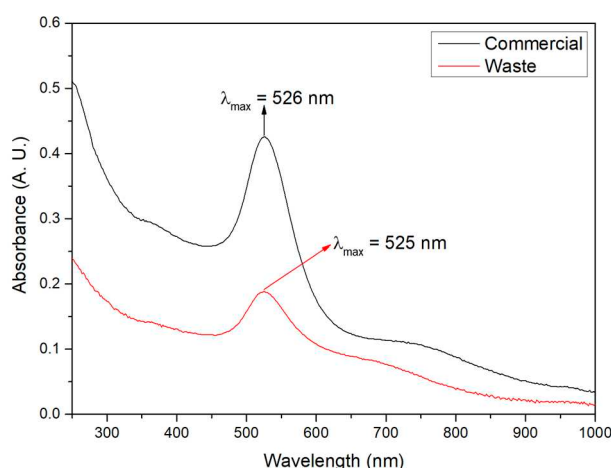


Figure 2. Absorption spectra of the unsupported Au NPs.

Powder XRD patterns (Figure 3) of the supported Au NPs show reflections at 24.97° , 31.85° , 38.41° , 44.54° , 45.60° , 64.78° , 77.78° , 81.90° , and 98.51° for Au_{e-w}/V NPs; and 25.02° , 31.87° , 38.39° , 44.54° , 45.60° , 64.84° , 77.80° , 81.96° , and 98.51° for Au_{com}/V NPs. In both patterns, the reflections situated at $\sim 38.4^\circ$, 44.5° , 64.8° , 77.8° , 81.9° , and 98.5° correspond to the (111), (200), (220), (311), (222), and (400) planes of the Au – FCC structure, according to crystallographic data sheet PDF 01-1172.

The broadened reflection at $\sim 25^\circ$ corresponds to the (002) carbon planes of the Vulcan. In addition, reflections at $\sim 31.9^\circ$ and $\sim 44.5^\circ$ correspond to the (200) and (220) planes, respectively, of sodium chloride, according to ICSD crystallographic record number 96-900-6377. These reflections corroborate the presence of NaCl in the materials, indicating its appearance as a by-product during the synthesis of Au NPs by the method described above. It is also observed that the XRD patterns are slightly more intense for the Au_{com}/V NPs, which could be associated with a higher amount (wt%) of Au in this material, as observed by UV-VIS spectroscopy, this was corroborated by EDS studies.

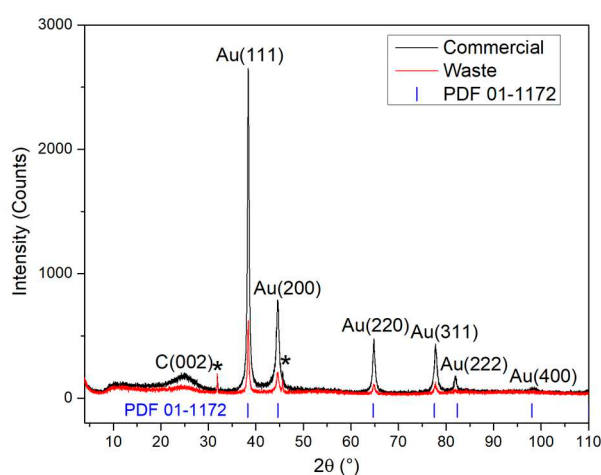


Figure 3. X-ray diffraction patterns of the supported Au NPs.

Figure 4 shows the SEM micrographs of the supported Au NPs, where a uniform distribution of gold nanoparticles on Vulcan is observed for both Au_{e-w}/V NPs (Figure 4a) and Au_{com}/V NPs (Figure 4b). EDS analysis of Au/V NPs, as summarized in Table 1, verifies the presence of key elements such as Au, Na, Cl and O in both samples. Surprisingly, the weight percentage of Au is approximately 1.7 times lower in Au_{e-w}/V NPs compared to Au_{com}/V NPs. This observation agrees with the results of X-ray diffraction and UV-Vis spectroscopy analyses.

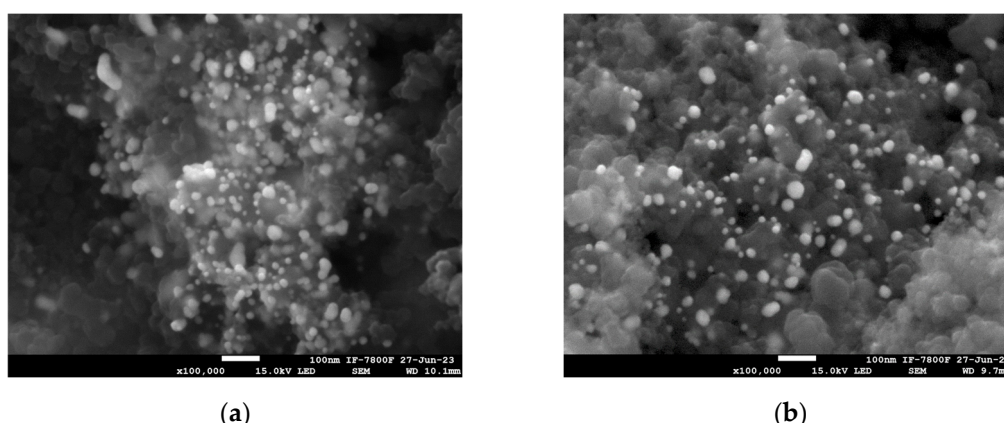


Figure 4. Scanning electron micrographs of the supported Au NPs. (a) Au_{e-w}/V NPs, (b) Au_{com}/V NPs.

Table 1. Chemical composition by EDS analysis of the supported Au NPs.

Element	wt% (Au _{e-w} NPs)	wt% (Au _{com} NPs)
Au	16.40	27.29
Na	31.84	19.95
Cl	43.55	18.12
O	8.21	34.64

Finally, TEM micrographs confirm that Au_{e-w} (Figure 5a) and Au_{com} NPs (Figure 5b) are uniformly dispersed on Vulcan and do not form agglomerates. The Au_{e-w}/V NPs exhibit a particle size of 14.1 ± 3.7 nm, while the Au_{com}/V NPs have a particle size of 18.5 ± 2.7 nm. Notably, the supported e-waste Au NPs (Figure 5a) shows a more crystalline phase on the Vulcan surface. These crystalline areas are likely associated with NaCl (a by-product of the synthesis process).

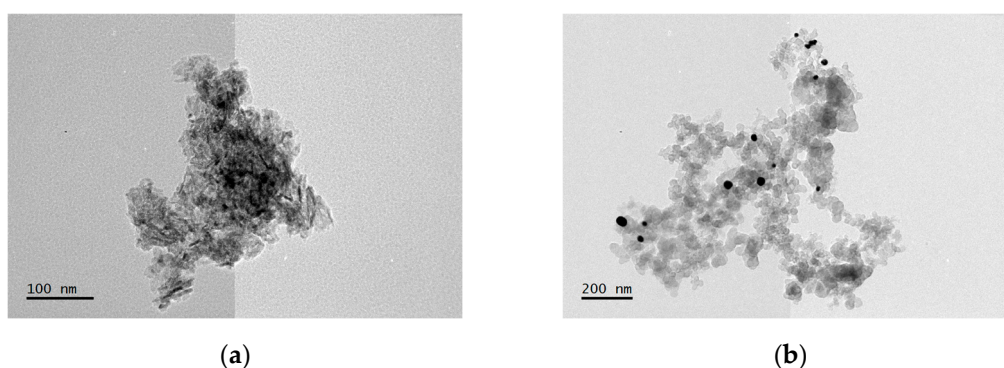


Figure 5. Transmission electron micrographs of the supported Au NPs. (a) Au_{e-w}/V, (b) Au_{com}/V.

These results support the crystalline quality of the gold nanoparticles, as well as providing additional information on the presence of NaCl as a by-product, as well as a higher gold content (wt%) for the Au_{e-w} NPs.

3.1. Electrochemical Characterization of Au/V NPs

Figure 6 shows the cyclic voltammograms in the absence of methanol for Au_{e-w}/V and Au_{com}/V NPs. The cyclic voltammograms are normalized with respect to mass current density ($\text{mA} \cdot \text{mg}^{-1}_{\text{Au}}$). For both materials, distinct regions characteristic of Au NPs in alkaline media can be identified [22,28]: from 0.98 to 1.06 V/RHE, the anodic region of OH⁻ adsorption and pre-oxidation species formation on the Au NPs surface are observed; from 1.06 to 1.21 V/RHE, processes of superficial oxidation of Au NPs occur; at ~ 1.09 V/RHE, a cathodic peak associated with the reduction of surface oxides of Au NPs is observed; and at 0.86 V/RHE, a cathodic peak associated with OH⁻ desorption is

also observed. The only difference between the two materials is that $\text{Au}_{\text{e-w}}/\text{V}$ NPs show a slightly higher current intensity than $\text{Au}_{\text{com}}/\text{V}$ NPs, mainly in the oxygen evolution zone ($> 1.5 \text{ V/RHE}$).

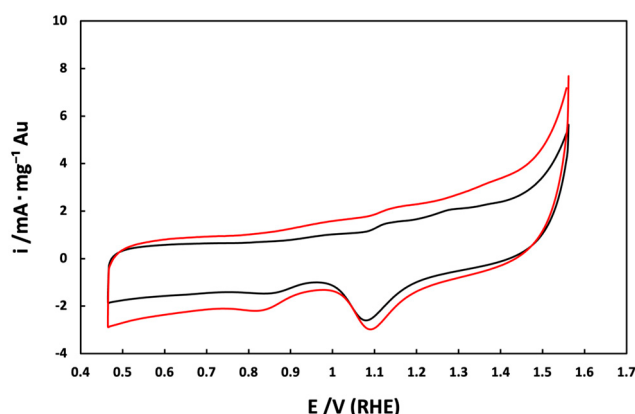


Figure 6. Cyclic voltammograms of the supported Au NPs: (—) $\text{Au}_{\text{e-w}}/\text{V}$ and (—) $\text{Au}_{\text{com}}/\text{V}$ in 0.5 mol L^{-1} KOH as electrolyte and at a sweep rate of $20 \text{ mV} \cdot \text{s}^{-1}$.

On the other hand, Figure 7 shows the cyclic voltammograms recorded in the presence of methanol (from 1 to 5 M) for both materials. Noteworthy is the anodic peak observed at 1.22 V/RHE , which is attributed to the methanol oxidation reaction. As the methanol concentration increases, there is a proportional increase in the mass current density.

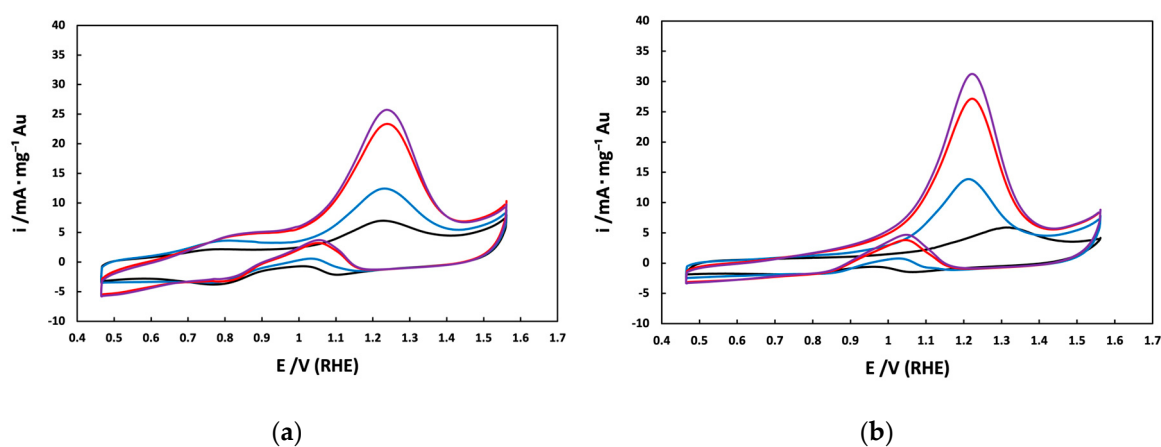


Figure 7. Cyclic voltammograms of the supported Au NPs in the presence of methanol: (—) 1 M, (—) 2 M, (—) 4 M and (—) 5 M in 0.5 mol L^{-1} KOH as electrolyte and at a sweep rate of $20 \text{ mV} \cdot \text{s}^{-1}$. (a) $\text{Au}_{\text{e-w}}/\text{V}$; (b) $\text{Au}_{\text{com}}/\text{V}$.

It is also worth noting that the mass current density for Au NPs derived from e-waste is just slightly lower than that obtained from the commercial reagent. For example, at a methanol concentration of 5 M, they exhibit values of 25.5 and $30.1 \text{ mA} \cdot \text{mg}^{-1} \text{Au}$, respectively. Nevertheless, the observed activity of Au nanoparticles in the methanol oxidation reaction remains comparable between the two sources. It is also important to note that these values are in agreement with those documented in the existing literature [22,28].

This suggests that Au NPs obtained from electronic waste show a potential application as an anode in alkaline fuel cells, i.e., for the methanol oxidation reaction.

4. Conclusions

This study explores into the promising field of sustainable electrocatalyst production from electronic waste (e-waste) as a resource. The research focuses on the electrocatalytic potential of gold nanoparticles supported on Vulcan, derived from e-waste, specifically Intel Pentium 4 processor pins. The synthesis of gold nanoparticles from recovered gold coatings highlights the sustainability of e-waste as electrocatalyst precursors.

Notably, the electrocatalytic activity exhibited by the gold nanoparticles is comparable to commercially synthesized counterparts, affirming the viability of this innovative approach for sustainable electrocatalyst production. This research not only contributes to the field of electrochemistry but also pioneers the utilization of e-waste as a valuable source for electrocatalytic materials.

The study encourages the exploration of new frontiers, advocating for the transformation of e-waste into a reservoir of electrocatalytic materials that not only addresses the environmental concerns associated with e-waste, but also presents a viable solution to the technological challenges in the field of alkaline fuel cells.

Author Contributions: Conceptualization, M.B.-S., J.O.-G. and E.B.-A.; methodology, M.B.-S., J.O.-G. and E.B.-A.; software, J.O.-G.; validation, M.B.-S. and L.M.-C.; formal analysis, M.B.-S.; investigation, E.B.-A., L.M.-C. and J.R.-A.; resources, E.B.-A.; data curation, L.M.-C. and J.R.-A.; writing—original draft preparation, E.B.-A.; writing—review and editing, E.B.-A., L.M.-C. and J.R.-A.; visualization, M.B.-S. and J.O.-G.; supervision, E.B.-A.; project administration, E.B.-A.; funding acquisition, E.B.-A. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflict of interest.

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