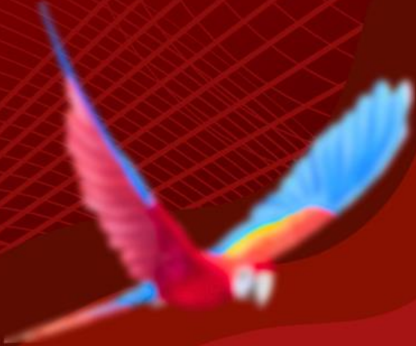
IV RB **SBEE**

IV Reunião Bienal da Sociedade
Brasileira de Eletroquímica e
Eletroanalítica

ARARAS-SP

ANAIS DA IV REUNIÃO BIENAL DA SOCIEDADE BRASILEIRA DE ELETROQUÍMICA E ELETROANALÍTICA

24 E 25 DE OUTUBRO DE 2024



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Sociedade Brasileira de Eletroquímica e Eletroanalítica
SBEE

ANAIS DA IV REUNIÃO BIENAL DA SOCIEDADE BRASILEIRA DE ELETROQUÍMICA E ELETROANALÍTICA

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UFSCar Araras - LSNano - Laboratório de Sensores, Nanomedicina e Materiais Nanoestruturados

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Desde 2015

SUMÁRIO

1.APRESENTAÇÃO

A **IV Reunião Bienal da Sociedade Brasileira de Eletroquímica e Eletroanalítica (IV RB SBEE)** foi realizada presencialmente na Universidade Federal de São Carlos, campus Araras, São Paulo entre os dias 24 e 25 de outubro de 2024. O objetivo do evento foi promover um espaço de discussão para pesquisadores e pesquisadoras que atuam no Brasil e no exterior para debaterem os principais avanços realizados na área de eletroquímica, dentro dos temas: 1) Eletroanalítica (EA) e 2) Eletroquímica (EL).

Durante o evento, os participantes tiveram a oportunidade de acompanhar 6 palestras plenárias ministradas por pesquisadores da área de Eletroquímica/Eletroanalítica, incluindo uma apresentação internacional, que enriqueceram os debates com perspectivas globais e inovadoras. Além disso, foram realizadas duas sessões de pôsteres, compreendendo 91 trabalhos e duas sessões orais, compreendendo 16 trabalhos, nas quais foram apresentadas pesquisas de alta qualidade científica, promovendo discussões intensas e produtivas.

O evento também proporcionou momentos de integração, com uma confraternização por adesão, que permitiu a troca de experiências em um ambiente descontraído, fortalecendo o *networking* entre os participantes.

A IV RB SBEE contou com a participação de 156 inscritos no total. Dentre os participantes, 55,1% (86) se identificam com o gênero masculino e 44,9% (70) com o gênero feminino, de acordo com a Figura 1.

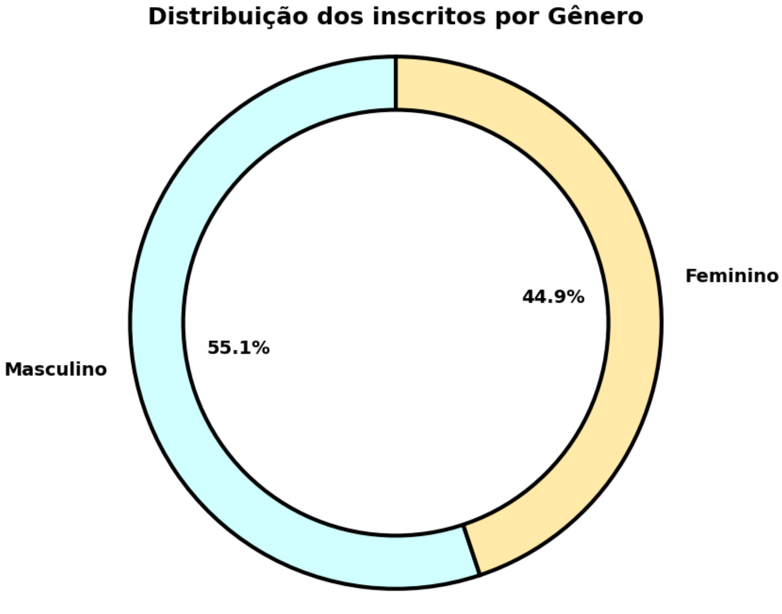

Figura 1. Distribuição dos inscritos por gênero

Além disso, no que tange o grau dos participantes, na Figura 2 é possível observar uma distribuição com predominância na presença de pesquisadores, com 41,7% (65), seguido pelos graduandos, com 31,4% (49) e por fim, os pós-graduandos, com 26.9% (42).

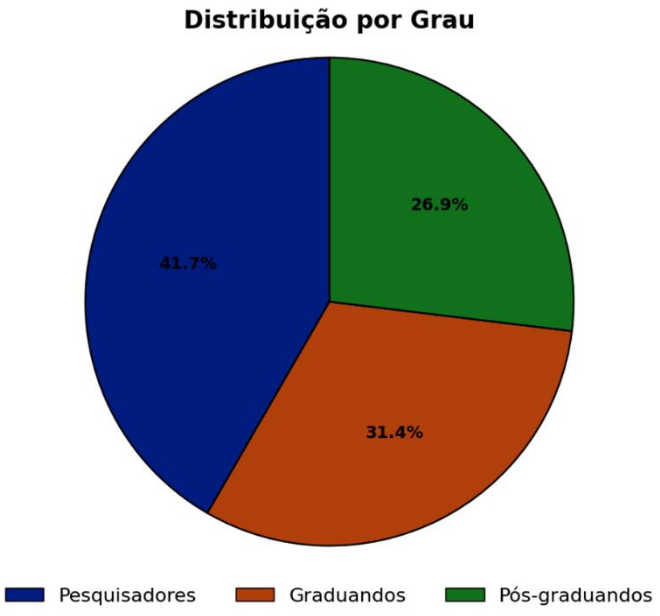

Figura 2. Distribuição dos participantes por grau

Em termos de Brasil, o evento contou com a presença de participantes de 11 estados brasileiros, com a participação majoritária de participantes da região Sudeste (Figura 3A), o que se refletiu no grande número de inscritos provenientes de universidades do estado de São Paulo, como a UFSCar, USP e UNESP (Figura 3B).

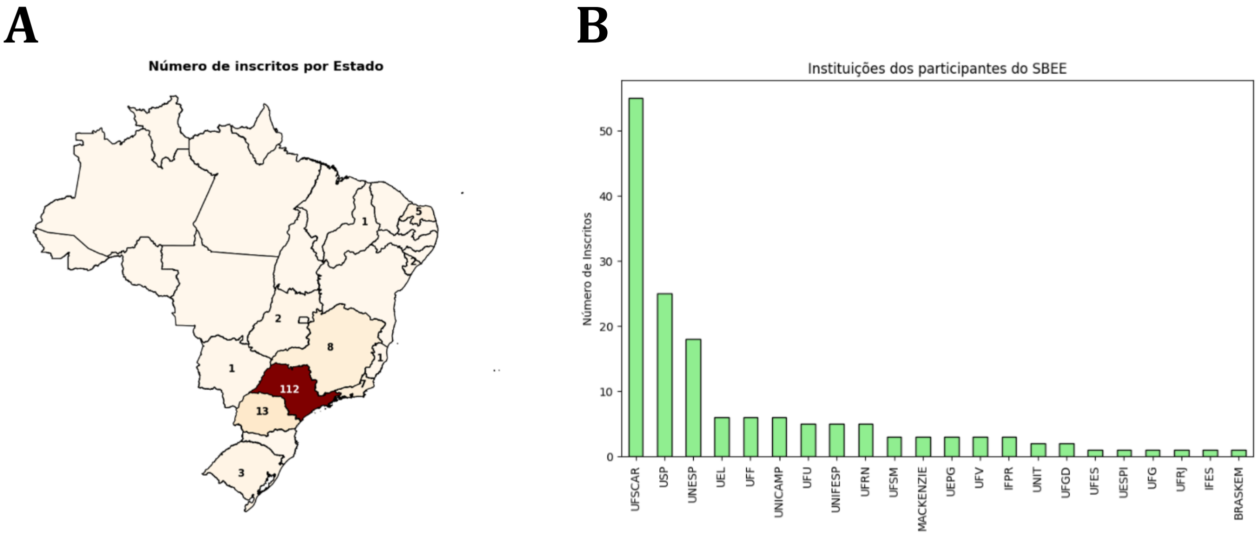

Figura 3. Distribuição dos inscritos **(A)** por Estados do Brasil. **(B)** por instituições

Gostaríamos de estender os nossos agradecimentos aos colegas que avaliaram os trabalhos submetidos, toda a comissão organizadora e científica e aos apoiadores deste evento, sem esse trabalho conjunto não teríamos atingido os objetivos do evento. A IV RB SBEE foi, sem dúvida, um marco para a comunidade de eletroquímica, impulsionando o avanço do conhecimento e o estabelecimento de novas colaborações científicas.

2.PROGRAMAÇÃO

A programação do evento, com todos os detalhes, pode ser observada na Tabela 1 e 2.

Tabela 1. Programação para o primeiro dia (quinta-feira, 24 de outubro de 2024)

Horário	Atividade	Título da atividade
8h – 8h30	Cerimônia de Abertura	Cerimônia de Abertura
8h30 -9h20	Palestra 1 Lúcia H. Mascaro (UFSCar, Brasil)	*Catalysts in Green Hydrogen Generation and the Path to a Sustainable Future*
9h30 – 10h20	Palestra 2 Gustavo A. Rivas (UNC, Argentina)	*Biofunctionalized carbon nanostructures: specialized legos for the construction of electrochemical biosensors?*
10h20 – 11h	Coffee Break	Coffee Break
11h – 11h15	Apresentação Oral 1 Gilson S. Costa (UESPI, Brasil)	*Induction of oxygen vacancies to improve the photocurrent in $CuWO_4$ films used as photoanodes*
11h15 – 11h30	Apresentação Oral 2 Mariane R. Lourenço (UFRRJ, Brasil)	*Development of a carbon paste electrochemical sensor modified with multi-walled carbon nanotubes for the detection of fipronil*
11h30 – 11h45	Apresentação Oral 3 Gustavo A. B. Santana (UFRN, Brasil)	*Electrosynthesis of acetate from ethanol using a BDD electrode*
11h45 – 12h	Apresentação Oral 4 Alencastro G. R. Lopes (UFV, Brasil)	*On the electrochemical behavior of modified electrodes based on deep eutectic solvents-carbon black films*
12h – 14h	Almoço	Almoço
14h – 14h50	Palestra 3 Rodrigo A. A. Muñoz (UFU, Brasil)	*From 3D printing to laser engraving: Affordable strategies for obtaining electrochemical sensors*
15h – 15h15	Apresentação Oral 5 Rafael L. Rosalino (IFES, Brasil)	*Synthesis of a polypyrrole hydrosponge and recycled MnO_2 composite from spent alkaline batteries*
15h15 – 15h30	Apresentação Oral 6 Isabela A. Pereira (UNESP, Brasil)	Metodologia exploratória de sensor eletroquímico para análise e detecção de *Salmonella enterica* em ambientes diversos
15h30 – 15h45	Apresentação Oral 7 Lyniker M. S. Almeida (UFSCar, Brasil)	*Nb_2O_5 electrodes prepared by different methods: photoelectrochemical properties and photocatalytic performance*
15h45 – 16h	Apresentação Oral 8 Willy O. Lima (Mackenzie, Brasil)	*Enhanced glyphosate detection using laser-induced graphene electrodes modified with copper phthalocyanine/gold nanoshells*
16h10 – 17h40	Coffee Break	Coffee Break
16h10 – 17h40	Apresentação de pôsteres	Apresentação de pôsteres
20h – 1h	Confraternização por adesão	Confraternização por adesão

Tabela 2. Programação para o segundo dia (sexta-feira, 25 de outubro de 2024)

Horário	Atividade	Título da atividade
8h30 -9h20	Palestra 4 Hamilton Varela (USP, Brasil)	A importância da pesquisa básica em eletrocatálise
9h30 – 10h20	Palestra 5 Wendell K. T. Coltro (UFG, Brasil)	*Disposable electrochemical sensors: fabrication and bioanalytical applications*
10h20 – 11h	Coffee Break	Coffee Break
11h – 11h15	Apresentação Oral 9 Gabriel G. Borges (UNESP, Brasil)	*Electrochemical synthesis of nanoporous nickel electrode applied as a cathode for water electrolysis*
11h15 – 11h30	Apresentação Oral 10 Sthéfane V. Almeida (UFSCar, Brasil)	*Imunoensaio eletroquímico para detecção de anticorpos específicos de hanseníase utilizando peptídeo sintético*
11h30 – 11h45	Apresentação Oral 11 Robson S. Souto (USP, Brasil)	*Zeolitic imidazolate framework-Zn structure applied as catalyst of the Printex L6 carbon for H_2O_2 electrogeneration by using a Gas Diffusion Electrode*
11h45 – 12h	Apresentação Oral 12 Luiz F. G. Luz (UFU, Brasil)	*Electrochemical genosensor for rapid and simplified detection of zika virus DNA using safranin as DNA genomic intercalator*
12h – 14h	Almoço	Almoço
14h – 14h50	Palestra 6 Elisama V. Santos (UFRN, Brasil)	*Integrated-hybrid approach for biomass electroreforming and carboxylic acids recovery*
15h – 15h15	Apresentação Oral 13 Kauan L. Gomes (UNICAMP, Brasil)	*Electroreduction of nitrate to ammonia through Co_3O_4 nanowires supported on TiO_2*
15h15 – 15h30	Apresentação Oral 14 Arielly Cardoso (UEL, Brasil)	*Amperometric method for determination of venlafaxine using a BIA system and BDDE*
15h30 – 15h45	Apresentação Oral 15 Rayane F. Viveiros (USP, Brasil)	*Xylene biodegradation and simultaneous bioelectricity production in a microbial fuel cell using mixed culture present in mangrove sediments*
15h45 – 16h	Apresentação Oral 16 Amanda S. M. Freitas (UFSCar, Brasil)	*Simultaneous detection of paraquat and carbofuran through electrochemical technique using a modified polymeric electrode*
16h10 – 17h40	Coffee Break	Coffee Break
16h10 – 17h40	Apresentação de pôsteres	Apresentação de pôsteres
17h40 – 18h30	Palestra de encerramento Murilo Santhiago (LNNano, Brasil)	*Tuning the surface chemistry and defects of layered materials on electrochemical interfaces*
	Homenagem ao Prof. Lauro Tatsuo Kubota	*Homenagem ao Prof. Lauro Tatsuo Kubota*

3.PALESTRANTES

Lucia Helena Mascaro

Pesquisadora e professora titular do Departamento de Química da Universidade Federal de São Carlos. Tem experiência na área de Química, com ênfase em Físico-Química, atuando principalmente nos seguintes temas: Eletroquímica, Eletrodeposição de metais ligas e semicondutores, corrosão, sensores eletroquímicos e baterias.

Gustavo Adolfo Rivas

Professor do Departamento de Físico-Química da Universidade Nacional de Córdoba (UNC) e investigador superior do Consejo Nacional de Investigaciones Cientificas y Tecnicas (CONICET). Tem experiência em Química Analítica, com ênfase em sensores e biossensores eletroquímicos, nanomateriais e técnicas eletroanalíticas.

Rodrigo Alejandro Abarza Muñoz

Pesquisador e professor associado da Universidade Federal de Uberlândia e membro afiliado da Academia Brasileira de Ciências. Tem experiência na área de Química, com ênfase em Química Analítica, atuando principalmente nos seguintes temas: eletroanalítica, sensores eletroquímicos, química forense, nanomateriais, grafeno e novos materiais para impressão 3D.

Wendell Karlos Tomazelli Coltro

Pesquisador e professor Associado do Instituto de Química da Universidade Federal de Goiás Tem experiência na área de Química Analítica com ênfase em Instrumentação Analítica, Microfabricação, Microfluídica, Eletroforese, Detecção Eletroquímica e Sensores Químicos.

Hamilton Brandão Varela de Albuquerque

Pesquisador e professor titular do Instituto de Química de São Carlos (IQSC) da Universidade de São Paulo. Tem experiência na área de Físico-química, com ênfase em Sistemas Complexos, Dinâmica Não-Linear e Eletroquímica.

Elisama Vieira dos Santos

Pesquisadora e professora associada da Universidade Federal do Rio Grande do Norte. Têm experiência em eletroquímica, com ênfase no desenvolvimento de processos eletroquímicos para um meio ambiente mais limpo, remoção de poluentes persistentes em água e solo; eletrocatálise e fotoeletrocatálise; acoplagem de energias verdes para processos de remedição eletroquímica; eletrossíntese de oxidantes; hidrogênio verde como um vetor energético para processos eletroquímicos; construção de sensores eletroquímicos; e disseminação científica e ações para igualdade de gênero.

Murilo Santhiago

Pesquisador no Laboratório Nacional de Nanotecnologia (LNNano) onde trabalha principalmente com dispositivos eletroquímicos. Tem experiência em estudo de defeitos químicos no plano basal de materiais bidimensionais; dispositivos eletroquímicos flexíveis miniaturizados; eletrocatalisadores suportados em matrizes flexíveis.

4. LISTA DE TRABALHOS

4.1. APRESENTAÇÃO DE PÔSTERES

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Área	ID	Título do resumo	Dia	Página
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Área	ID	Título do resumo	Dia	Página
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Área	ID	Título do resumo	Dia	Página
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6.

RESUMOS

Estudo da Influência Heterojunção Metal-Suporte na Eletroquímica Intensificada por Plasmônica

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A sociedade moderna enfrenta diversas dificuldades devido ao uso abusivo de combustíveis fósseis na produção de energia. Por isso, problemas como o esgotamento desses e a degradação ambiental gerada pelo seu uso, que agrava situações climáticas extremas vistas nos últimos anos. Com isso, vê-se a necessidade de conseguir novas fontes de energia duradoura e limpa, como a solar e a eólica. Essas fontes possibilitam uma produção de energia maior que o uso imediato, porém, devido a sua sazonalidade também necessitam de estratégias de armazenamento. Uma estratégia para isso é a geração de gás hidrogênio, um combustível de combustão livre de compostos carbonados e de alto poder energético. Esse gás pode ser obtido pela eletrólise da água, onde no ânodo há a formação de O2 e no cátodo, H2. O processo anódico possui uma forte limitação cinética e uma estratégia para diminuir essa limitação é a eletroquímica intensificada por plasmônica. Nesse processo, os elétrons presentes em nanopartículas metálicas (Au, Ag ou Cu) de tamanho coerente com a luz incidente entram em ressonância com o campo magnético (plásmons), excitando os elétrons energeticamente, gerando os portadores de cargas "quentes". Nesse processo, os elétrons vão para níveis de energia mais altos e, consequentemente, também há a geração de buracos de baixa energia. Esses portadores de carga "quentes", por serem mais energéticos, auxiliam na catálise redox, aumentando a atividade eletroquímica e/ou diminuindo a sobretensão. No geral, esses portadores podem se recombinar e gerar um efeito fototérmico convertendo a energia luminosa em calor e impedindo seu uso em catálise. Para otimizar a aplicação desses portadores em catálise, é proposto o uso de suporte em materiais semi-condutores, o que gera uma barreira Schotkky na região interface metal-suporte. Essa heterojunção normalmente é composta de defeitos estruturais, como vacâncias de oxigênio, e aumentam o tempo de meia-vida dos portadores “quentes”, intensificando sua aplicação na eletrocatálise. Por isso, este trabalho propõe-se a analisar a influências dos defeitos de heterojunção na interface metal-suporte Au/óxidos de tungstênio. O material foi obtido a partir do processo mecanoquímico, controlando o aumento da população desses defeitos, em fase sólida, descartando o uso de solventes e outros compostos que pudessem bloquear algum sítio ativo dos materiais. Na análise, a eletrólise da água foi considerada como um todo, os processos de redução e oxidação, no qual foi avaliada a importância dessa população de defeitos antes e depois dos processos no claro e no escuro.

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Electrochemical detection of gallic acid in complex matrices samples employing a completely 3D printed device

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Gallic Acid (GA) is a phenolic compound that exhibits various beneficial properties for humans, such as antioxidant, anticancer, and anti-inflammatory activities [1]. This compound can be found in different types of products and foods. However, the excessive consumption of products with high concentrations of GA can cause various health problems, particularly affecting the liver and kidneys [2]. Therefore, the development of new analytical devices for monitoring and quantifying GA in different matrices, especially complex matrices such as wine and tea, is of paramount importance to ensure product quality and consumer safety. In this regard, the present study addresses the development of a portable electrochemical platform through additive manufacturing technology using non-conductive and conductive materials, such as polylactic acid and carbon black, for the determination of GA in beverages and water samples. The GA detection was performed using differential pulse voltammetric technique after optimization of operational parameters through multivariate experiments. An analytical curve ranging from 20.0 to 1000.0 μmol L^{-1} was obtained, with limits of detection and quantification of 0.17 and 0.56 μmol L^{-1}, respectively. Furthermore, the analysis of GA in wine, tea, tereré, and water samples yielded satisfactory results, with recovery values ranging from 96 to 104%. Thus, the proposed platform shows great potential to be applied in the detection of GA in complex matrices, with a low cost, appropriate detectability, and allowing on-site and fast application.

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Characterization of Nb_2O_5 anodic oxides obtained by Plasma Electrolytic Oxidation (PEO)

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The method of plasma electrolytic oxidation (PEO) is derived from the standard anodization process, in which inside an electrochemical cell with the metal substrate as an anode and platinum as the cathode. [1]. In the PEO process, the voltages applied are above the dielectric breakdown voltage of the electrode, resulting in the appearance of plasma discharges on the surface of the electrode being anodized, which often results in more crystalline anodic oxides [2]. In this study, the Nb substrate utilized was polished and rinsed before anodization. The PEO method was performed with current density of 20 mA/cm^2 for 70 minutes, under temperatures of 2 or 15 °C, in varying electrolyte solutions of 0,1 mol/L of KOH, $C_2H_2O_4$ and H_3PO_4. The samples obtained were characterized by MEV, XDR and photocurrent experiments, demonstrating varying morphology, as well as varying crystalline structures, which could be attributed to the different electrolyte solution compositions. Photoelectrochemistry experiments are conducted to study the electronic structure of the oxide films obtained.

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Development of a sustainable electrochemical sensor for agricultural monitoring.

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The use of pesticides can cause contamination of soil, water, air and food, which makes it necessary to develop simple and portable detection methods for monitoring environmental pollution caused by agricultural producers. In this context, a composite of polylactic acid (PLA) and graphite (g) of different composition ratios is being studied for an electrochemical sensor. PLA is melted at the temperature range of 180 to 200 °C and graphite is incorporated into it. This process eliminates the use of organic solvents, making it more environmentally friendly. Samples of PLA/graphite composites were studied in the following proportions: 50:50, 40:60 and 30:70 (m/m). The samples were tested in an electrochemical cell and the current density was measured using a potentiostat. The samples were tested in a potassium ferrocyanide redox probe solution and the data obtained by cyclic voltammetry indicate that the 30:70 PLA/graphite ratio results in the highest current density for both oxidation (0.44 mA.cm2) and reduction (-0.37 mA.cm2), suggesting greater electrochemical efficiency. The electrode was then functionalized to achieve an enhanced electrochemical response. To this end, nanostructured films were coated using the Layer by Layer (LbL) technique, using poly(allylamine hydrochloride) - PAH - as the positive polyelectrolyte and copper phthalocyanines (CuTsPc) as the negative polyelectrolyte. Electrochemical characterization was carried out using cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS). The materials were further analyzed using Fourier Transform Infrared Spectroscopy (FTIR) and contact angle measurements. Future research will focus on utilizing the substrate modified by LbL for the electrochemical detection of pesticides.

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Electrodeposition study of nickel nanostructures using deep eutectic solvents as electrolytic medium

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Among the techniques of chemical deposition of a metal on a substrate, electrodeposition has some advantages, such as: accuracy, reliability, simplicity, low cost, and versatility [1,2]. In this context, the present work aimed to study some parameters of nickel (Ni) electrodeposition on an alternative substrate based on silicon and copper (Cu/Si) and using deep eutectic solvents (DES) as electrolytic medium. The prepared DESs (Reline, Etaline and Glyceline) were characterized by FT-IR, NMR-^{1}H and NMR-^{13}C. Different electrodeposition parameters were studied, including type of DES, applied potential (E_{app}), deposition time (t) and temperature (T). The results obtained by cyclic voltammetry showed cathodic and anodic peaks that refer to the redox processes of nickel (Ni$^{0/2+}$) both using the glassy carbon electrode (GCE) and the Cu/Si substrate as working electrodes. In addition, Ni films with different morphologies were obtained using the different studied DES, as demonstrated from SEM analysis. By using as main criteria the obtaining of well-adhered and uniform Ni films on the proposed Cu/Si substrate and SEM-EDS and XRD characterization data, the most suitable condition for Ni electrodeposition was reached: DES-Reline, $E_{\text{app}} = -1.1$ V, $t = 2$ h and $T \cong 25$ °C. All the reported results prove the efficiency of using a more sustainable and green solvent towards nickel electrodeposition and can also be exploited for the electrodeposition of other metals.

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Desenvolvimento de magneto-imunoensaio utilizando nanopartículas de prata voltado para o diagnóstico sorológico de hanseníase

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A hanseníase é uma doença tropical negligenciada com alto percentual de subnotificação. Ela é causada pela *Mycobacterium leprae* e sua transmissão ocorre através das vias respiratórias, para isso sendo necessário o contato prolongado [1]. Nesse trabalho foi desenvolvido um magneto-imunoensaio para detecção de anticorpos anti-*M. leprae* em amostra de soro, utilizando partículas magnéticas modificadas com peptídeos sintéticos. Também foram utilizadas nanopartículas de prata (AgNPs) marcadas com anticorpo anti-IgG humano tendo a função de marcador eletroquímico. As medidas eletroquímicas foram realizadas utilizando eletrodos impressos (SPEs) através da técnica de Voltametria de Pulso Diferencial (DPV). As AgNPs foram sintetizadas seguindo a metodologia de Hao et. al [2] e caracterizadas por espectroscopia de UV visível, sendo o conjugado entre AgNP e anti-IgG humano também caracterizado da mesma forma. Otimizações foram realizadas com o objetivo de alcançar maiores valores de corrente de pico, sendo essas realizadas em uma amostra originada de um paciente diagnosticado com hanseníase multibacilar (MB). Ao se encontrar as melhores condições avaliadas foi construída uma curva ROC e um gráfico de pontos para se verificar, através de amostras MB e saudáveis, qual era a porcentagem de sensibilidade e especificidade do método, sendo eles respectivamente 91,7 e 75,0%. O magneto-imunoensaio foi considerado adequado em diferenciar amostras de indivíduos saudáveis de doentes com hanseníase MB.

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Determination of paracetamol using an electrochemical sensor with graphite paste electrode modified with cork

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On the raw cork (RAC) and regranulated cork (RGC) modified graphite (Gr) paste electrode, paracetamol can be determined in the presence of sulfuric acid without interference. The results clearly showed that the cork-graphite sensors, GrRAC and GrRGC, exhibited a linear response over a wide range of paracetamol concentration (25–1000 µM), with R2 of 0.99 and 0.98, respectively. The limits of detection (LOD), estimated at 2.4 and 8.03 µM for GrRAC and GrRGC, suggest greater sensitivity and reproducibility than the unmodified conventional graphite sensor (Fig. 1a,b). The low-cost cork-graphite sensors were successfully applied in the determination of caffeine in soft drinks and pharmaceutical formulations, presenting well-defined current signals when analyzing real samples. When comparing electrochemical determinations and high-performance liquid chromatography measurements, no significant differences were observed (mean accuracy 3.0%), highlighting the potential use of these sensors to determine paracetamol in different samples.

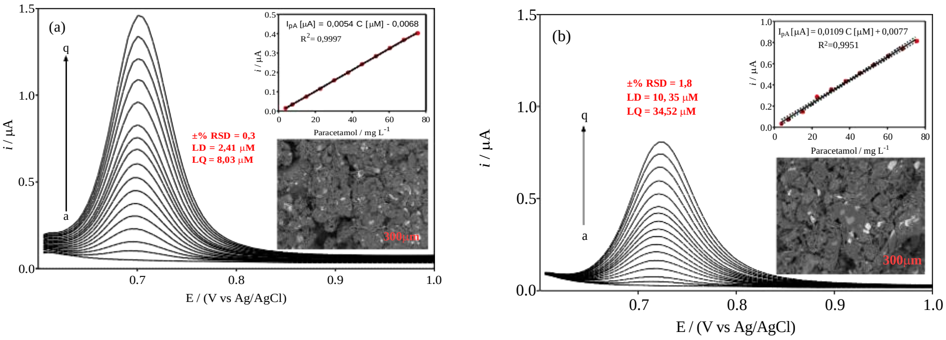

Figura 1 – Differential pulse voltammograms of paracetamol in 0.5M H_2SO_4. Standard additions of paracetamol solution (0.01 M): (a) supporting electrolyte, (b) 25, (c) 50, (d) 100, (e) 150, (f) 200, (g) 250, (h) 300, (i) 350, (j) 400, (k) 450, (l) 500, (m) 600, (n) 700, (o) 800, (p) 900 and (q) 1000 µM. Inset: linear calibration plots of paracetamol concentration in solution versus current peak. Insert: Micrograph of modified and unmodified graphite paste.

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Glycerol electro-oxidation over palladium based catalysts

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Biofuels, such as biodiesel, are renewable energy sources with several advantages, including biodegradability and low toxicity. However, their production generates significant amounts of glycerol as byproduct.[1] Electro-oxidation emerges as an alternative to add value to the excess glycerol, converting it into higher-value sustainable chemicals. The efficiency and selectivity of the glycerol electro-oxidation reaction depend on various factors, including the choice of catalyst, its crystalline structure, and composition.[2] Different catalysts have been extensively researched to enhance the electro-oxidation of glycerol and to aim for product selectivity. Among all the catalysts, noble metals such as platinum, gold, and palladium are the most researched due to its stability and activity.[3] In this work, different palladium-based catalysts are used to study glycerol electro-oxidation in alkaline media. They were analyzed by its stability and activity through cyclic voltammetry experiments and by Tafel plots analysis. Among the metallic catalysts tested, which include Palladium, Palladium-Iron, Palladium-Cooper and Palladium-Iron-Copper, Palladium-Iron showed the best performance, with high current density of 1.59 $mAcm^{-2}$, one of the lowest onset potentials at 0.65 V, and lower Tafel Slope of 62 mV/dec.

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Operating parameters optimization of a semi-pilot electrolyzer for H_2 production from wastewater

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Current hydrogen production relies on fossil fuel utilization such as natural gas in the steam methane reforming process, leading to substantial carbon dioxide emissions. Consequently, efforts have been directed towards developing sustainable pathways to mitigate CO_2 emission during hydrogen generation. Especially, electrolysis-based water splitting has emerged as a promising method to produce high-purity hydrogen albeit its high cost production.[1] To enhance the economic viability of hydrogen, we propose the modification of a conventional electrolyzer by replacing the anodic oxygen evolution reaction with the electrooxidation of organic pollutants from wastewater. This modification not only facilitates hydrogen generation but also tackles environmental concerns linked with decentralized disposal of organic pollutants.[2] Herein, it will be shown the optimization of the operating parameters of the 24 cm^2 anode/cathode semi-pilot electrolyzer cell by investigating variables such as operating temperature, electrolyte composition, operating current density, membrane properties, and associated costs. Combining statistics, such as Box-Behnken design, TOC (total organic carbon) and COD (chemical oxygen demand) analyses, we found the optimized electrolyzer operation at mild current density using nickel/boron doped diamond (cathode/anode), anion exchange membrane in concentrated KOH environment towards 60 °C.

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Characterization of modified electrodes for OER through electrochemical and AFM-IR techniques

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Oxygen evolution reaction (OER) or water oxidation reaction is one of the most relevant challenges from the chemical point of view in the water splitting process. Reducing the costs of green hydrogen is essential for creating a cleaner energy matrix. In this sense, the search for new and Earth-abundant catalysts continues to involve great efforts of the scientific community. Given this scenario, catalysts such as Co-Prussian blue analogues ($Co_3[Fe(CN)_6]_2$)can be highlighted due to the performance of OER under mild conditions and the ability to be used as the precursor for oxide production[1]. However, heat treatment of $Co_3[Fe(CN)_6]_2$ (up to 500 °C) leads to the formation of a mixture of oxides and potassium carbonate. The catalyst was deposited on the FTO electrode by electrodeposition, drop casting, spray coating and spin coating and subsequently heat treated at 500 °C. The values under mild conditions (0.5 M KNO_3 solution) of η@10mA/cm^2 (mV) / R_{ct} (Ω) were respectively: electrodeposition (865/253), drop casting (987/388), spray coating (969/258) and spin coating (1035/252). The produced oxides were characterized using the Bruker - NanoIR2-s (AFM coupled with IR spectroscopy) located at LNNano and we could observe the presence of $CoFe_2O_4$ and K_2CO_3 on the surface of the electrodes prepared by different methods.

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Pt e Pt-Sn suportados em Nióbio-Carbono Vulcan para reação oxidação de etanol em solução ácida

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Células a combustível são dispositivos capazes de converter energia química em energia elétrica com alta eficiência. A célula a combustível de membrana trocadora de próton (PEMFC) operam em baixas temperaturas (60-100C°) e convertem energia com uma alta eficiência. As PEMFC podem ser alimentadas com etanol direto (DEFC) no ânodo, e neste caso o eletrocatalisador de Pt-Sn tem apresentado um melhor desempenho eletroquímico para oxidação do etanol [1]. Entretanto ainda há desafios a vencer como a quebra da ligação C-C para a completa oxidação de etanol à CO_2 e H_2O. Assim, este trabalho mostra um estudo da influência do nióbio no suporte do catalisador Pt-Sn. Os catalisadores foram sintetizados utilizando $NaBH_4$ como agente redutor e suportados em Nióbio e Carbono Vulcan (50% m/m). Foram realizados experimentos de voltametria cíclica e cronoamperometria em H_2SO_4 0,5 mol L^{-1} e com etanol 1,0 mol L^{-1} no eletrólito. Os catalisadores Pt/C, Pt/C(50%)NbO_5(50%), Pt/C(50%)Nb_2O_2(50%), PtSn/C, Pt-Sn/C(50%)NbO_2(50%) e PtSn/C(50%)Nb_2O_5(50%), apresentaram atividade catalítica; o PtSn/C(50%)Nb_2O_5(50%) apresentou um menor potencial de início de oxidação de etanol do que Pt/C(50%)NbO_5(50%). Por outro lado, comparando os materiais de Pt/C e Pt/C(50%)Nb_2O_5(50%) o material sem o oxido de Nióbio apresentou um menor potencial de início de oxidação.

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Studying the use of a catalyst based on Co, Ni, Fe, and P to overall water electrolysis

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The number of extreme weather events has been increasing in the last years as a consequence of the greenhouse effect, which increase the pressure to replace the fossil fuels by renewables one [1]. One option is the production of green hydrogen by water electrolysis, which indeed has been highlighted and received investment from the governments and efforts from research community. Thus, in this work, it is investigated the use of CoNiFeP alloys electrodeposited on 304 stainless steel for overall alkaline water electrolysis. The electrodeposition was carried out in a galvanostatic regime and the overpotential to reach |10| mA cm^{-2} (η_{10}) for the hydrogen evolution reaction (HER) and for the oxygen evolution reaction (OER) in KOH 1.0 mol L^{-1} was used as a response to the optimization of alloy electrodeposition. The best parameters were reached by applying –0.2 A cm$^{-2}_{geo}$ for 500 s in electrolyte containing 0.5 mol L^{-1} H_3PO_4, 1.0 mol L^{-1} $NiSO_4$, 0.5 mol L^{-1} $CoSO_4$, 0.01 mol L^{-1} $FeSO_4$, and 1.0 mol L^{-1} NaH_2PO_2 (dissolved in that order). Under these conditions, a catalyst load of 4.11 ±0.44 mg cm$^{-2}_{geo}$ is obtained and a η_{10} of 111 ±6 mV for HER and 292 ±13 mV for OER are obtained. According to energy-dispersive X-ray spectroscopy (EDS) analysis, the alloy has a mass composition of 58.7 ±1.9% Co, 27.6 ±1.2% Ni, 2.8 ±0.2 % Fe, 6.1 ±0.3% P and 3.9 ±0.9% O. Even after catalyst stability testing for 24 hours at η_{10} followed by 12 hours at |100| mA cm$^{-2}_{geo}$ (η_{100}) no significant variations were observed by EDS. The reversibility of the system, that is, the use of the same electrode containing the electrodeposited catalyst (i) as cathode, (ii) as anode, and (iii) again as cathode was investigated too. The activity behavior showed that after 6 hours (each step) of OER at 10 and 100 mA cm$^{-2}_{geo}$ the catalyst exhibits almost the same values to η_{10} and η_{100} under HER compared to the first analysis using it as cathode. Although, after OER, the catalyst increases its activity while the HER takes place, which means that the active sites for HER can be regenerated. However, it is necessary to understand what is the effect of system reversal on catalyst stability over long-term experiments.

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Electrochemical determination of citalopram by batch injection analysis and multiple pulse amperometry using boron-doped diamond electrode

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Citalopram (CT) is an antidepressant that acts as selective serotonin reuptake inhibitor (SSRI), and it has been classified as a contaminant of emerging concern [1]. In the literature many works were found, in which electroanalytical methods were developed to detect and quantify different sample types. The boron doped diamond electrode (BDDE) has advantages such as robustness and wide range of electrochemical potential [2]. The batch injection analysis system (BIA) offers advantages such as high analytical frequency and sensitivity and simple instrumentation [3]. Thus, the objective of this work was to develop an electroanalytical method to the detection and quantification of CT, using the cyclic voltammetry (CV) and multiple pulse amperometry (MPA) techniques coupled to a BIA system, and a BDDE. The cyclic voltammograms indicated an irreversible oxidation process at approximately 1.4 V. However, an adsorption process of the product generated in the CT oxidation was observed on the BDDE surface and, to minimize it, it was necessary to carry out an electrochemical pre-treatment (-1.5 V for 30 s) on its surface. For the detection, MPA was used in which two potential pulses were applied: 1.6 V for 0.15 s, for the detection, and -1.0 V for 0.1 s, to clean the electrode surface and minimize its passivation. Then it was evaluated that a better response was obtained using a cathodic pretreatment and a speed of 123.2 μL s^{-1} and volume of 200 μL in the BIA system parameters. Based on that, the analytical curve was made in the range of 5.0 x 10^{-7} to 1.0 x 10^{-5} mol L^{-1}, presenting a linear equation of I(μA) = (3.5 ± 0,6) x 10^{5} [CT] + (2.6766 ± 0,1) x 10^{-1} R^2= 0.998. As next steps, the possible interferers of the analyte will be studied. However, the BDDE coupled to the BIA system and the MPA technique showed great potential for the development of a simple and fast method for the detection and quantification of CT in different pharmaceutical, biological and environmental samples.

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Comparative study of 3D-printed graphene/PLA and carbon black/PLA electrodes under different conditions of electrochemical pretreatment

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3D-printed electrochemical sensors have been highlighting by enabling the creation of a wide variety of designs. These electrodes are printed using a filament based on a combination of a polymer matrix, such as polylactic acid (PLA) with a conductive material, including carbon black (CB) and graphene (GR). To improve their electrochemical response, chemical and electrochemical pretreatments (PTs) have been studied [1]. In this work, the electrochemical performance of 3D-printed electrode based on carbon black (3D-CBPLA) or graphene (3D-GR-PLA) and polylactic acid was compared, and the effect of different electrochemical PTs was investigated using dopamine (DOP) as an electrochemical probe. CB-PLA and 3D-GR-PLA electrodes (30.1x11.1x1.8mm) were printed using an Ender-3, Creality FDM 3D. Two electrochemical PTs were evaluated: PTE-VC and PTC-Pot. In the PTE-VC, the 3D-CB-PLA and 3D-GR-PLA electrodes were pretreated firstly using chronoamperometry technique, applying a potential of 1.8 V for 900 s, following of cyclic voltammetry technique under a potential range of 0.0 to -1.8 V. In the PTE-Pot, the 3D-GR-PLA was subjected to treatment using chronopotentiometry (-100 mA/cm^{-2} for 200 s). The electrochemical studies for comparison of CB-PLA and GR-PLA were performed using cyclic voltammetry with 2.0 mmol L^{-1} DOP in KCl solution 0.1 mol L^{-1}. The current intensity of the anodic and cathodic peaks obtained with GR-PLA was 6 times higher than that obtained with CB-PLA, indicating that a 3D-GR-PLA electrode presents a higher electroactive area. After that, the effects of PTE-VC and PTE-Pot on the electrochemical response for DA using the 3D-GR-PLA electrode were evaluated. It was observed that after the PTE-Pot, GR-PLA exhibited a higher peak current and a lower ΔEp. The advantage of this pretreatment is mainly related to the reduction of time; while PTE-VC takes 40 minutes, PTE-Pot only takes 4 minutes. Therefore, the comparative study of 3D CB-PLA and GR-PLA, being obtained a better electrochemical response with electrode based on graphene, the evaluation of electrochemical PT was performed for GR-PLA and the application of -100 mA cm^{-1} for 200 s provided a better electrochemical behavior for DOP.

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Evaluation of PtRu/C as an electrocatalyst for the ammonia oxidation reaction

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In the quest for a transition to renewable and carbon-free energy sources, ammonia is emerging as a promising candidate, with applications in the safe storage and transportation of hydrogen [1]. Furthermore, the ammonia oxidation reaction (AOR) could be utilized for the removal of ammonia from wastewater and in the anode of a direct ammonia fuel cell [2]. To enhance the viability of these possibilities, further studies are required to investigate the AOR. In the present work, AOR was studied in alkaline medium with commercial PtRu as catalyst supported on carbon. In order to understand the effects of the alloy, we conducted the same experimental procedure with Pt and Ru supported on carbon. The approach of online electrochemical mass spectrometry (OLEMS) was used to identify the products of the oxidation during the sweep of potential. The PtRu catalyst exhibited a reduction of 100 mV at the onset of AOR and demonstrated mitigation in the poisoning effect when compared to Pt and Ru. The gaseous products detected during the experiments were N_2, N_2H_4, NH_2OH, HN_3, NO and N_2O. Nitrogen was identified as the major product of the AOR, a finding consistent with recent studies [3].

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Estudo da Influência das Funcionalidades Oxigenadas de Materiais Grafíticos na Reação de Oxidação de SO_2

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Os motores a combustão são um dos responsáveis pela produção e liberação de gases tóxicos e agravamento das mudanças climáticas. Assim, o uso de energia a partir de fontes renováveis, menos poluente, como o H_2 gerado pela reação de eletrólise da água, usando energia solar; é uma alternativa. Nesse trabalho, propõe-se estudar a reação de oxidação de SO_2 ($ROSO_2$), um gás tóxico emitido pela combustão de combustíveis fósseis que muitas vezes contém compostos sulfurados, utilizando eletrodos de materiais grafíticos e carbonáceos, os quais podem substituir os eletrodos feitos de materiais nobres. A $ROSO_2$ possui menores E^0 e limitações cinéticas que a reação de desprendimento de O_2, permitindo maior produção de H_2. Utilizou-se uma célula eletroquímica padrão de três eletrodos, eletrólito foi H_2SO_4 0,5 mol L^{-1} contendo Na_2SO_3 1 mol L^{-1} e perturbações triangulares de potencial. Para as medições, foi utilizado um eletrodo de trabalho composto por carbono vítreo, em primeiro momento, e modificado por suspensões de óxidos de grafeno reduzidos com diferentes processos de redução, consequentemente, diferentes funcionalidades oxigenadas superficiais. Foi observado que a maior atividade obtida é utilizando apenas o carbono vítreo, ocorrendo uma piora ao utilizar os RGOs. Isso é inesperado, segundo a literatura, pois, indica que com a existência de grupos funcionais no material, a atividade é reduzida, podendo sugerir que a adsorção do SO_2 ocorra sobre sítios de C livres. Sobre os eletrodos de carbono vítreo, observou-se dois picos de oxidação nas varreduras no sentido positivo, atribuídos, incialmente, a dois processos de oxidação do SO_2. Porém, ao aumentar a velocidade de varredura, o pico localizado a potenciais mais positivos diminui sua intensidade, chegando a não ser observado a velocidades superiores a 100 mV s^{-1}. Simultaneamente, um pico de redução foi observado. em detrimento, sugerindo um mecanismo eletroquímico-químico-eletroquímico para a $ROSO_2$.

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Energy-Saving Electrochemical Hydrogen Production Coupled with Landfill Leachate treatment

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Leachate is effluent rich in nitrogenous compounds and inorganic salts, originating from solid waste in landfills, requiring treatment to reduce its pollutants. Leachate treatment by electrochemical oxidation (EO) is a promising approach to deal with these chemical pollutants in nature. In this work, EO of this effluent was carried out using Boron-Doped Diamond (BDD) and Platinum (Pt) anodes, with an area of 20.8 cm^2, and a Ni/Fe mesh as cathode, with the aim of obtaining value-added products, including carboxylic acids and green hydrogen. For this, the experiments were carried out for 10 hours, under a current of 3A, without adding supporting electrolyte. With these conditions, a Chemical Oxygen Demand (COD) decay of 30% was observed using the Pt anode and 80% using the BDD anode, for the same conditions. The production of 72 and 92mg L^{-1} of formic acid, 57 and 232 mg L^{-1} of acetic acid was achieved, using Pt and BDD anodes, respectively. According to the energy-saving estimations, a production of 13.6 L min^{-1} of green hydrogen was obtained, however the cogeneration of other gaseous products (8%) was reached, such as chlorine and ammonia. Then, the electrochemical treatment of leachate effluent was efficiently achieved with cogeneration of green hydrogen.

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Evaluation of corrosion protection by anodic coatings containing incorporated zeolites and Ce^{3+} ions

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The anticorrosive proprieties of coatings obtained by *Micro-Arc Oxidation* (MAO) on magnesium alloy can be improved through the nanoparticle incorporation of SiO_2 from the electrolyte [1]. In this work, it was proposed to obtain coatings by MAO from electrolyte baths containing dispersed zeolites on magnesium AZ31 alloy and perform the cerium ions incorporation in this coating for anticorrosive protection in a solution that simulates the physiologic medium. The zeolites are microporous aluminosilicates with high silica content and elasticity like human bones, therefore promising for bioimplants [2]. As for cerium ions, they are non-toxic and have been shown to protect Mg-based alloys under physiological conditions [3]. Before the anodization, the cerium ions were incorporated into the coating by immersing for 1 hour in an aqueous solution containing cerium ions. The coating formation containing zeolite and cerium was identified by X-ray Diffraction analysis. Electrochemical Impedance Spectroscopy (EIS) tests were also carried out to evaluate corrosion resistance. The EIS results have shown that in the coating containing incorporated cerium ions, the impedance modulus value at 10 mHz ($|Z|_{0.01\ Hz}$) was 27.9 kΩ cm^2. For the coating without cerium ions presence, the $|Z|_{0.01\ Hz}$ value was 3.8 kΩ cm^2, showing that the presence of cerium ions in these coatings improves the corrosion protection of the magnesium alloy.

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Effect of different cerium precursors on Ce-BiVO$_4$ photoanode electrochemical doping

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The use of the BiVO$_4$ semiconductor to generate H$_2$ via water splitting has recently been studied to attend the global energy demand. However, BiVO$_4$ have some limitations as higher electron hole recombination and poor stability, being necessary studies that aims a better performance. In this scenario, the use of the rare earths as a doping agent should be considered. This work explores the BiVO$_4$ electrochemical synthesis via BiOI film obtention [1] and the effects of the use different cerium precursors on the physical and photoelectrochemical (PEC) properties. Cerium(III) nitrate, cerium(IV) oxide and ammonium cerium(IV) nitrate were evaluated as precursors and added into the plating solution before the electrochemical deposition. The films obtained underwent characterization through XRD, SEM-EDS, and micro-Raman analyses. The results indicate that cerium is incorporated into the BiVO$_4$ structure, and that this incorporation did not alter its monoclinic structure. The PEC films characterization was evaluated by cyclic voltammetry, linear sweep voltammetry and electrochemical impedance spectroscopy, showing a different photoelectrochemical activity that depends on the used cerium precursors. The use of ammonium cerium(IV) nitrate improves the activity of the Ce-BiVO$_4$ films, while the others precursors decrease the activity. Analysis of the UV-Vis reflectance spectra and Mott-Schottky showed the difference between the materials bandgap and conduction band, highlighting that the use of different cerium precursors must be carefully evaluated to obtain a Ce-BiVO$_4$ film with a lower electron hole recombination.

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Electrodeposition of Zn-Ni alloy on 22MnB5 steel for application at high temperatures.

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Safety guidelines for automotive vehicles and ecological aspects related to the reduction of fuel and CO_2 emissions, have been demanding that automotive industries develop lighter steels with greater mechanical resistance. To acquire these mechanical properties, steels with specific compositions such as 22MnB5 require a thermomechanical process denominated Hot Stamping (HS) [1]. The application of Zn-based coatings has been reported in the literature to avoid excessive oxidation, with loss of mechanical properties, and to ensure cathodic protection after HS of these steels [1,2]. In this work, it was proposed to coat the steel with a Zn-Ni alloy by galvanostatic electrodeposition to evaluate the oxidation resistance. These samples were submitted to heat treatment similar to the HS. The Zn-Ni coatings were analyzed by Scanning Electron Microscopy and Energy Dispersive Spectrometry before and after heat treatment. These analyses show that the electrodeposits were not damaged by heat treatment, presenting a thickness of around 10 µm. The element analyses indicated the presence of 2.3 (wt%) of Fe on the Zn-Ni coating surface after heat treatment, demonstrating that the coating inhibited Fe migration from the substrate to the surface.

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New route for the production of lab-made composite filaments based on soybean oil, polylactic acid and carbon black nanoparticles, and its application in the additive manufacturing of electrochemical sensors

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Additive manufacturing electrochemical sensors have gained relevance and prominence in the scientific community due to their usage of sustainable materials, relatively low-cost, and the possibility of large-scale production and customization [1]. In recent years, the production of conductive filaments has attracted interest, allowing the development of different compositions of those filaments [1, 2]. Due to their performance and highly reliable analysis, they have been used for diverse kinds of detections, such as pharmaceuticals, among others. Given this, that work presents a lab-made conductive filament based on carbon black (CB), polylactic acid (PLA) and polypropylene (PP), developed by a new route for the production of that filament, for the detection of Catechol (CTC) in tap water and river water. The analyses were made after polishing but without any electrochemical treatment. As a result, an analytical curve was built in the linear range of 3.0 to 40 µmol L^{-1} and showed the limit of detection of 0.7 µmol L^{-1}. Also, recovery tests presented values between 93.4% and 101.7%, demonstrating that there is no significant matrix effect. Therefore, it is possible to infer that the production of electrochemical sensors from lab-made conductive filaments was successfully reached, demonstrating the applicability of the material since it was possible to determine CTC in sub-micromolar concentration and with no matrix interference.

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Electrocatalytic Activity of Carbon-supported PdFe Nanoparticles Towards the Ethanol Oxidation in Alkaline Media

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The electro-oxidation of ethanol is a topic of significant importance due to its relevance as a renewable biofuel and application in fuel cells. Ethanol is particularly attractive because it can be produced sustainably from biomass and is less toxic compared to methanol. However, the complete oxidation of ethanol to CO_2, which releases 12 electrons, is challenging with conventional noble metal catalysts like platinum. These catalysts often suffer from poisoning by carbonaceous intermediates such as CO and CH_x species, which adsorb strongly on the catalyst surface, thus reducing their efficiency and durability. The need for catalysts that can effectively break the C-C bond and fully oxidize ethanol to CO_2 has driven research towards alloying noble metals with other elements to enhance their catalytic properties. This study focuses on the comparative study of the electrochemical activity of Pd/C and PdFe/C nanoparticles towards the oxidation of ethanol in alkaline media. The nanoparticles were evaluated using cyclic voltammetry (CV) and Tafel analysis to assess the catalytic performance and kinetic parameters. So far, our findings indicate that PdFe/C nanoparticles exhibit superior catalytic activity compared to Pd/C nanoparticles. The CV profiles reveal that the PdFe/C catalysts have a higher peak current density and lower onset potential for ethanol oxidation, suggesting enhanced electrocatalytic properties. The Tafel plots corroborate these results, indicating improved reaction kinetics. The addition of Fe to Pd appears to facilitate the cleavage of the C-C bond in ethanol, promoting the complete oxidation to CO_2 and mitigating catalyst poisoning by intermediates. This study contributes to the understanding of bimetallic catalysts' role in enhancing the activity and stability of the ethanol electro-oxidation reaction.

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Desenvolvimento de célula eletroquímica em 3D com dois eletrodos de trabalho para detecção de Ciprofibrato

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A impressão 3D tem revolucionado a produção de objetos de maneira rápida e eficaz, com baixo custo e grande liberdade na criação de estruturas complexas [1]. Recentemente, filamentos condutores à base de negro de fumo e grafeno têm sido explorados devido à sua alta área superficial, baixo custo e boa condutividade elétrica, combinando materiais condutores com polímeros de forma favorável [2]. No contexto do diagnóstico, o futuro demanda plataformas que ofereçam benefícios como proficiência em multiplexação. Plataformas multiplex apresentam vantagens como detecção *in loco*, análise qualitativa e quantitativa, facilidade de uso, portabilidade, baixa demanda de amostra e economia de tempo ao permitir repetições em um único ensaio [3]. Este estudo foca na produção de uma célula eletroquímica 3D com dois eletrodos de trabalho utilizando o filamento CB/PLA da Protopasta® para a detecção de ciprofibrato. Este medicamento é essencial para controlar altos níveis de gordura no sangue (hiperlipemia), sendo um tipo de proliferador de peroxissomos hipolipidêmicos que inclui compostos sintéticos e contaminantes ambientais [4]. A detecção do ciprofibrato é crucial devido à sua eficácia no controle de colesterol elevado e triglicerídeos. Os eletrodos foram impressos, polidos e ativados em NaOH 0,5 mol L^{-1}, preparando-os para os testes eletroquímicos. O sistema foi caracterizado utilizando ferrocenometanol para calcular a área eletroativa. Por conseguinte, medidas de voltametria de pulso diferencial e voltametria de onda quadrada foram realizadas na presença de ciprofibrato em meio de tampão fosfato (pH 7,0), onde foi observada um sinal de corrente de pico anódica em 1,2 V vs. CB/PLA. A caracterização morfológica foi realizada por microscopia eletrônica de varredura (MEV), enquanto os testes de ângulo de contato foram conduzidos com um equipamento descrito por Silva et al. [5]. Este estudo destaca a viabilidade da célula eletroquímica 3D utilizando o filamento comercial CB/PLA (Protopasta®) para a detecção de ciprofibrato em produtos farmacêuticos.

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Standardizing protocols for electrochemical studies using DES – Application in CO_2 reduction.

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Electrochemical reduction of carbon dioxide (CO_2RR) is an attractive strategy towards conversion convert an environmental liability into value-added products. However, one of the main obstacles limiting the application of the CO_2RR in aqueous electrolytes is the working potential range, that is similar to the hydrogen evolution [1], what makes the use of aqueous electrolytes not attractive in terms of efficiency. Deep eutectic solvents (DESs) are mixtures composed of a hydrogen donor with an acceptor that presents good potential for CO_2RR due to factors as good solubility of CO_2 [2]. Anyhow, the literature did not present a consensus about the working conditions in DES such as the ideal reference electrode. Once we want to avoid adding water to the system, using a typical Ag/AgCl aqueous electrode as a reference may transfer water molecules to the media. Herein, we first evaluated Ag wire as a pseudo-reference electrode in reline (choline chloride + urea) and ethaline (choline chloride + ethylene glycol). We observed that the OCP value (using vitreous carbon as both working and counter electrode) displaced by c. a. 150 and 60 mV in reline and ethaline respectively after 2 hours, not reaching a plateau. This must be more like the silver surface reacting with the chlorides in the media. Thus, aiming for a more stable system, we inserted an Ag wire covered by an AgCl film in a Luggin capillary, and added a solution of the respective DES with 0.1 M of AgCl, this system reached an equilibrium and a stable potential with less than 10 mV of variation. Further, we employed potassium ferricyanide/ferrocyanide as internal standard for correcting the potential. Thus, a CO_2RR test was performed in reline, employing the obtained reference electrode, vitreous carbon as counter electrode and a metallic copper as working electrode. A linear voltammetry was performed in the absence of CO_2 gas, and after the saturation, a peak at -1,2 V vs ferrocyanide was identified, indicating the CO_2 molecule adsorption in the Cu surface for further reduction. In conclusion, we were able to develop more strict conditions for working with CO_2RR using DES, that still will be further evaluated with more complex electrodes.

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Avaliação da performance eletrocatalítica do carbono Printex XE2B modificado com ferroceno para aplicação na reação de redução de oxigênio

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A reação de redução de oxigênio (RRO) via 2 elétrons é um processo eletroquímico que visa a produção de peróxido de hidrogênio (H_2O_2) *in situ*, sendo este oxidante comumente utilizado como precursor para a formação de radicais hidroxila em processos oxidativos avançados eletroquímicos que visam a degradação de poluentes emergentes. Os materiais carbonáceos comerciais do tipo Printex são eletrocatalisadores seletivos para a RRO via 2 elétrons, e sua atividade pode ser otimizada ao serem modificados com nanomateriais metálicos [1]. Assim, neste trabalho avaliou-se a interação provocada pela modificação do carbono Printex XE2B (CPXE2B) com diferentes porcentagens mássicas de ferroceno ($Fe[C_5H_5]_2$), sendo avaliados os fatores físico-químicos que influenciam o perfil eletrocatalítico destes materiais quando aplicados nos processos de RRO visando a eletrogeração de H_2O_2 *in situ*. O CPXE2B e suas composições contendo 1, 2 e 3% (m/m) de $Fe[C_5H_5]_2$ foram caracterizados por difração de raios X, ângulo de contato e microscopia eletrônica de varredura. Realizaram-se os ensaios eletroquímicos em eletrólito de K_2SO_4 0,1 mol L^{-1} (pH 9,0) saturado com $O_{2(g)}$ ou $N_{2(g)}$, utilizando um reator de 150 mL contendo três eletrodos, sendo o eletrodo de referência de Ag |AgCl, contra-eletrodo de Pt e como eletrodo de trabalho o eletrodo de disco-anel rotatório. Sobre a superfície do carbono vítreo do eletrodo de trabalho depositou-se uma microcamada composta por estes eletrocatalisadores, sendo utilizadas as técnicas de voltametria cíclica (VC) e voltametria de varredura linear (VL). Por meio desses estudos verificou-se que a modificação do CPXE2B com 3,0% (m/m) de $Fe[C_5H_5]_2$ apresentou aumento no efeito de hidrofobicidade, menor número de elétrons (2,2) e maior seletividade (~90%) para a eletrogeração de H_2O_2. Por outro lado, quando aplicado somente o CPXE2B nestas mesmas condições, foram obtidos número de elétrons de 2,5 e seletividade de 73,4%. Nos experimentos de cronoamperometria utilizando o eletrodo de difusão gasosa (EDG) produzido com o CPXE2B contendo 2,0% (m/m) de $Fe[C_5H_5]_2$ em pH 9 e potencial aplicado de -2,8 V, uma concentração de H_2O_2 eletrogerado de 974 mg L^{-1} foi obtida após 90 minutos de eletrólise enquanto o EDG composto somente por CPXE2B obteve 320 mgL^{-1}. Desta forma, estes resultados demonstram com sucesso o potencial promissor da modificação do CPXE2B com $Fe[C_5H_5]_2$ para aprimorar a eletrogeração de H_2O_2 *in situ*. Os resultados revelam um sinergismo notável entre os dois materiais, culminando em um aumento significativo das propriedades eletrocatalíticas do CPXE2B modificado.

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Application of indirect electrochemical oxidation in real textile effluents with simultaneous cogeneration of green hydrogen

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Water consumption for industrial activities represents one of the contemporary problems [1]. Industries such as textiles have high water consumption and the with generation of effluents with a high organic load, requiring approaches to make the process more sustainable. In this context, the use of electrochemical technologies can be efficient in degrading pollutants present in effluents and wastewater. The objective of this work consists of the degradation of a synthetic textile effluent containing the dyes Red BG-3B, Yellow BG-4G and Blue DC-2G, individually, and its trichome by electrooxidation approach with cogeneration of green hydrogen. The electrochemical cell used was a type-anion exchange membrane with boron-doped diamond (BDD) as anode and Ni-Fe mesh as cathode, respectively. In the anodic compartment, 200 mL of textile effluents were electrolyzed; while a solution of 1000 mL of 0.5M NaOH was used in the cathodic compartment, both in flow regime, by applying 100 mA cm^{-2} for 10 h. It was observed that after 5 h of electrolysis, 100% of the color was removed from the solutions for all dyes studied. After 10 h of process, the organic load of the water matrices achieved a reduction of around 97.57% for yellow, 97.76% for red and 83.02% for blue while 98.36% for trichomy, according to COD analyses. The production of persulfate in the solution was also observed in all experiments, which promoted the degradation of dyes. Regarding the production of green hydrogen, after 10 h of electrolysis, 7.0 L were produced. According to the results obtained, it was possible to conclude that electrochemical approach is an effective method for removing organic charge and removing trichromy and its respective colors, contributing simultaneously to produce an energy vector.

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Enhanced ORR performance of Graphene Nanoribbons through strategic heteroatom doping

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Graphene nanoribbons (GNRs) are promising for catalytic applications due to their unique properties. This study explored the effects of heteroatom doping on the performance of GNR-based catalysts in oxygen reduction reactions (ORR). A series of N, S, and P-doped and co-doped carbon catalysts were developed using a single GNR matrix and evaluated for their impact on ORR activity and selectivity in different conditions [1]. The doping of GNRs with N, S, and/or P was performed through hydrothermal synthesis. The GNRs were mixed with different combinations of dopants (NH_4OH, $N_2H_6SO_4$, $K_2S_2O_8$, and P_2O_5) and water, followed by heating and stirring. The resulting product was then mixed with water and NH_4OH, heated in an autoclave system, and finally washed by centrifugation. The impact of N, S, and P doping and co-doping on the catalytic ORR activity and selectivity of GNRs in acidic, neutral, and alkaline media was analyzed. The characterizations revealed that doping introduced heteroatoms and depleted oxygen, with nitrogen being the most effective. Electrochemical analysis indicated that heteroatom insertion and oxygen depletion affected ORR selectivity. Nitrogen improved catalytic activity in high pH, while sulfur and phosphorus were effective in acidic media [2]. These findings suggest that doping can adjust the ORR pathway according to the desired product. By modifying the GNR surface chemistry through doping, it is possible to enhance ORR activity and selectivity without compromising structural integrity. This study deepens the understanding of how heteroatom doping affects the electrochemical behavior of carbon-based catalysts, providing a foundation for future research in optimizing electrocatalysts for energy conversion.

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Compósitos baseados em carbono do tipo Printex L6 e Printex XE2B aplicados como eletrocatalisadores para a reação de redução de oxigênio

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Materiais carbonáceos são amplamente utilizados como eletrocatalisadores para a reação de redução de oxigênio (RRO) devido às suas características de estabilidade química, condutividade elétrica, elevada área superficial específica e baixo custo, além de apresentar alta seletividade mecanística para o mecanismo de dois elétrons, devido aos grupos oxigenados presentes em suas estruturas [1]. Apesar de serem eletrocatalisadores promissores, a performance eletrocatalítica destes materiais pode ser melhorada a partir da incorporação de diferentes grupos funcionais em sua estrutura, aderindo maior quantidade de sítios ativos com a função de aumentar a atividade eletrocatalítica durante a RRO por diferentes mecanismos. Nesse sentido, este trabalho avalia o efeito da combinação de diferentes materiais carbonáceos comerciais do tipo Printex L6 (CPL6) e Printex XE2B (CPXE2B) para aplicação como eletrocatalisadores nos processos de RRO. Busca-se a obtenção de efeitos eletroquímicos semelhantes aos modificadores metálicos (como íons metálicos, nanopartículas e compostos organometálicos). CPL6 e CPXE2B foram caracterizados por difração de raios X, ângulo de contato, espectroscopia Raman, espectroscopia de infravermelho e microscopia eletrônica de varredura. Os ensaios eletroquímicos foram realizados em eletrólito suporte 0,1 mol L^{-1} de K_2SO_4 (pH 9,0), saturados com $O_{2(g)}$ ou $N_{2(g)}$. A RRO foi avaliada por voltametria cíclica (VC) e voltametria de varredura linear (VL), sendo esta realizada em diferentes rotações (300-1500 rpm). Estes ensaios foram conduzidos em um reator contendo três eletrodos: eletrodo de referência de Ag|AgCl, contra-eletrodo de Pt e eletrodo de disco-anel rotatório (RRDE) do tipo Pt-GC como eletrodo de trabalho. A tinta composta pela combinação dos materiais carbonáceos foi realizada pela variação de diferentes proporções (20:80, 40:60, 60:40 e 80:20 (m/m)) de CPL6:CPXE2B. Baseado nos resultados obtidos, verificou-se que as composições com maior porcentagem de CPL6 apresentaram maiores seletividade para a RRO via dois elétrons. A composição com 80% (m/m) alcançou seletividade para H_2O_2 de 76% e número de elétrons médio de 2,5. Por outro lado, a composição com 80% (m/m) de CPXE2B apresentou seletividade de 41%, número de elétrons médio de 3,0, além de um deslocamento no início da RRO para potenciais mais positivos. Desta forma, verifica-se que a combinação de dois materiais carbonáceos que possuem propriedades físico-químicas diferentes demonstra efeitos eletrocatalíticos diversos, direcionando a RRO para 2 ou 4 elétrons, em função da sua composição do compósito.

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Electro-oxidation of Ethanol in Different Conditions – A Fundamental Investigating of Sulphate Electrolytes Effects

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Catalytic energy conversion processes, such as the hydrogen evolution reaction (HER) from water, are essential for clean energy production and addressing fossil fuel dependency. However, the water oxidation reaction (WOR) faces significant thermodynamic obstacles, rendering it inefficient. The electro-oxidation of organic compounds, such as ethanol on a polycrystalline platinum (pc-Pt) electrode, presents a viable alternative due to its lower overpotential (0.6 V *vs.* RHE) compared to water (1.23 V vs. RHE). In this context, it is crucial to investigate the effects of different electrolyte ions. Sulfate, in particular, demonstrates significant kinetic control over alcohol oxidation. Furthermore, the introduction of chaotropic species stabilizes interactions between the catalyst and anion. Research results for the electro-oxidation of 1 M ethanol indicate higher current density and favorable electron transfer up to 75% (Na_2SO_4/$NaClO_4$) at pH 13. Similarly, in acidic media, the trend remains apparent, suggesting kinetic and mechanistic changes. These promising data highlight the SO_4^{2-}/ClO_4^{-} system, whose fundamental effects are being explored. Additionally, other pairs of anions are under investigation to extend interactive observations.

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Voltammetric detection of dopamine using a graphite paste electrode modified with a hybrid carbon/silsesquioxane-based composite

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A new composite synthesized from carbon nanotube and a silsesquioxane functionalized with an intervalence complex, silver pentacyanonitrosylferrate (CNTSPAg), which was then characterizes by different spectroscopic techniques. The spectroscopic techniques proved that the nanocomposite was successfully formed. The cyclic voltammograms of the graphite paste electrode modified (CNTSPAg) shows a redox couple with potential ($E^{\Theta'}$) = 0.23 V ± 0.01 ($v=50 mVs^{-1}$, $KClO_3$ 1.0 mol L^{-1}). After electrochemical system optimization, the graphite paste electrode was tested for the detection of dopamine. The composite was sensitive to dopamine concentrations with a linear response in the range of 1.0×10^{-5} to 9.0×10^{-3} mol L^{-1} with a limit of detection of 5.46×10^{-6} mol L^{-1} to dopamine.

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Characterization of different graphite brands through the development of electrochemical sensors for CA detection

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Different synthesis routes can generate a variation in the physical-chemical properties of the products due to disparities in size, shape or even molecular structure. In this context, for the production of electrochemical sensors it is interesting to investigate these variations, because even small differences in material properties can generate irregularities in its surface area or electrical conductivity. Other facts, such as the presence of defects, impurities and emergence of different functional groups can also affect their electrochemical behavior [1]. One of the most utilized material for the production of conductive inks is graphite [2]. Based on this, the present work used SEM, FTIR, XRD and contact angle to assess the sensors surface made with graphite from the brands Fisher Chemical™, Sigma-Aldrich® and Synth®. Other techniques, such as CV and EIS were utilized to evaluate the possible differences in the electrochemical behavior of the proposed devices. Through this analysis, it was possible to observe some structural and electrochemical differences. Furthermore, all the devices were submitted to catechol (CA) detection by CV analysis, which it is a substance present in the list of priority pollutants, needing a constant monitoring in aquatic environments [3]. The most promising device was G_{Fis}-GV which it was submitted to CA detection in SWV analysis. Under optimized parameters a calibration curve was obtained for the linear range of 5.0×10^{-6} to 1.0×10^{-4} mol L^{-1}, in phosphate buffer pH 8.0, with an R^2 of 0.997, with a limit of detection of 1.39×10^{-6} mol L^{-1}. The developed sensor is easy to prepare and the results suggest the viability for the detection of CA in real samples.

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Estudo hidrodinâmico e eletroquímico de um novo projeto de reator em fluxo aplicado para eletrogeração de H_2O_2 in situ

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O tratamento de água enfrenta desafios para decomposição de compostos recalcitrantes, resistentes aos métodos tradicionais. Nesse cenário, reatores eletroquímicos surgem como uma alternativa promissora, oferecendo a produção de peróxido de hidrogênio (H_2O_2) in situ para processos eletroquímicos oxidativos avançados (PEOA). Este estudo apresenta um novo modelo de reator eletroquímico em fluxo contínuo, equipado com eletrodo de difusão gasosa (EDG) para redução de O_2 em H_2O_2. O EDG, composto por carbono Printex L6 e politetrafluoretileno (80/20), foi sintetizado e incorporado em tecidos de carbono, seguido de sinterização a quente. Para avaliar o comportamento hidrodinâmico do reator, a técnica de pulso traçador (KCl) foi empregada em diferentes vazões (12, 24 e 36 L h^{-1}), medindo-se a condutividade ao longo do tempo [1]. A eletrogeração de H_2O_2 nas mesmas vazões permitiu a comparação com o comportamento hidrodinâmico. Os resultados da distribuição de tempo de residência (DTR) revelaram comportamentos distintos em diferentes vazões. Vazões baixas (12 L h^{-1}) apresentaram maior dispersão do traçador e tempo de residência elevado, caracterizando um modelo de tanque. Nessas condições, maiores concentrações de H_2O_2 (~100 mg L^{-1}) foram observadas após a estabilização. Em contrapartida, vazões mais altas (36 L h^{-1}) apresentaram comportamento de reator de fluxo laminar, com menor tempo de residência e concentrações de H_2O_2 (~40 mg L^{-1}) após a estabilização. O reator eletroquímico testado demonstra comportamentos hidrodinâmicos distintos em diferentes vazões, influenciando na concentração de H_2O_2. Modelagem e otimização do sistema podem permitir o controle preciso da concentração de H_2O_2, abrindo caminho para aplicações de tratamento de água utilizando-se de métodos POA.

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Electrocoagulation waste as sustainable modifier for sensors

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Electrocoagulation (EC) is a technique in which wastewater is treated with high efficiency, producing a residue. This residue is mainly composed of aluminum ions, due to the use of sacrificial aluminum electrodes to remove pollutants from water [1]. However, a limited number of applications has been given for this residue, and therefore, we proposed here the use of the EC residue as a sensor modifier to increase the selectivity and sensitivity of Pyrolytic Graphite Sheet (PGS). To do that, PGS electrodes were cut into square pieces of 1 cm^2 and submitted to a drop casting modifying process following a procedure adapted from previous works [2]. The aluminum EC residue for each of the experiments was dissolved into the chitosan solution (1% w/w) and drop casted using 25 µL of the chitosan/HCl/aluminum dispersion and left to dry at room temperature. Cyclic voltammetry shows that the presence of aluminum residue on the electrode surface shifted oxidation and reduction peaks in the cyclic voltammograms (CV) for 1 mmol L^{-1} $[Fe(CN)_6]^{3-/4-}$ in 0.1 mol L^{-1} KCl. An important parameter observed in the CV profiles is the ΔE_{peak}, which is lower for all the modifications, indicating greater conductivity in the modified electrodes. In addition, electrochemical impedance spectroscopy (EIS) wascarried out to measure the resistance to charge transfer (R_{ct}) that these surfacesprovide, showing that all the modified surfaces have a higher conductivity than the unmodified electrode. With this, it is possible to demonstrate an improvement in the conductivity of the PGS electrode after the addition of the residue generated in EC process. The work's future objectives are to test these modified electrodes in the simultaneous detection of interest molecules.

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Electrogeneration of value-added products from the simultaneous degradation of paracetamol, caffeine and olanzapine using a BDD film

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The disposal of pharmaceuticals into the environment has led to their wide presence in wastewater and groundwater, marking them as emerging contaminants. Traditional wastewater treatment methods struggle with these persistent pollutants due to their refractory nature [1]. Advanced Oxidation Processes (EAOPs) allow the transformation of pollutants into more oxidized organic compounds instead of complete mineralization [2]. Among them, electrochemical treatment can generate highly oxidized substances like carboxylic acids, which are common acids identified post-treatment (acetic, formic, propionic, isobutyric, caproic, and valeric acids) [3]. This research focuses on electroproduction of carboxylic acids by treating wastewater contaminated with paracetamol, caffeine, and olanzapine simultaneously using a boron-doped diamond (BDD) anode. Additionally, the study investigated the impact of different supporting electrolytes on this electrochemical process determining the degradation sequence (paracetamol > caffeine > olanzapine). Both electrolytes used in the study enhanced contaminant degradation. H_2SO_4 notably increased olanzapine solubility and degradation. Chromatographic analysis revealed that carboxylic acid production varied with electrolysis duration. Under acidic conditions, higher concentrations of acetic acid and formic acid (11.57 mg L^{-1} and 4.42 mg L^{-1}, respectively) were observed at 30 and 45 min of electrolysis, respectively. Conversely, lower carboxylic acid production, below 2.5 mg L^{-1} even after 120 minutes, was achieved with Na_2SO_4. Then, electrochemical oxidation of effluents serves as a viable method to drive waste conversion into valuable substances like carboxylic acids, positioning it as an electro-refinery approach.

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Electrochemical study of the Al | $NaAlCl_4$(f) interface of Na-Al batteries

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Sodium beta alumina batteries are a class of molten salt batteries that operate at temperatures between 160 °C and 350 °C. They are assembled with molten sodium metal on the negative electrode, a solid electrolyte of Na-β/β"-alumina as separator and ionic conductor of Na^+, and different materials on the positive electrode, such as sulfur for the Na-S battery or a mixture of powdered Ni, NaCl and a molten salt of $NaAlCl_4$ for the Na-Ni battery [1]. Currently, it is proposed to use aluminum in molten $NaAlCl_4$ on the positive electrode, thus forming a Na-Al battery [2,3]. So, this work presents an electrochemical study of the Al electrode in molten $NaAlCl_4$. For the investigations, the $NaAlCl_4$ salt was synthesized in the laboratory from the sublimation of $AlCl_3$ followed by reaction with NaCl at 185 °C, obtaining a yield of 92 %. The $NaAlCl_4$ salt was also obtained from the oxidation of an Al according to the equation $Al(s) + 4NaCl(s) \rightarrow 3Na(f) + NaAlCl_4(f)$, using the cell Na(f) | Na-β/β"-Al_2O_3(s) | $NaAlCl_4$(f), NaCl(s) | Al(s). Both salts were characterized by melting point, X-ray diffractometry and Raman spectroscopy. Electrochemical studies of an Al working electrode in molten $NaAlCl_4$ at 250 °C were carried out using an Al(s) | $AlCl_4^-$(f) reference electrode. Processes associated with the reversible half-reaction $Al(s) + 4Cl^-(f) \rightleftharpoons AlCl_4^-(f) + 3e^-$ and also the half-reaction $Al(s) + 7AlCl_4^-(f) \rightleftharpoons 4Al_2Cl_7^-(f) + 3e^-$ at 300 mV more positive potentials were observed. Al electrodeposition occurred in a dendritic structure with a large area, generating a cross-over between the direct and reverse scans in voltammetric curves. From these results, the Al redox processes in molten $NaAlCl_4$ will be discussed.

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Polyol-synthesized Cu/C catalyst for CO_2 electroreduction in alkaline medium

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Currently, there is a global trend to reduce carbon dioxide emissions caused by industries and the large fleet of automobile vehicles, as this gas is mainly responsible for global warming and severe climate change around the world [1]. Therefore, new CO_2 capture technologies are highly investigated to mitigate excess carbon dioxide and prolong life on Earth. In this sense, copper is the most efficient metal for reducing CO_2 in addition to CO and generating products with C-C coupling of commercial interest, such as ethanol and ethylene glycol. Herein, we use the polyol method coupled to microwave heating to prepared copper nanoparticles on Vulcan carbon [2]. We prepare it using two different precursors, copper sulfate, and chloride. Initial cyclic voltammetric analyzes ranging from -1.0 V and 0.6 V vs Ag/AgCl were investigated in 0.1 M potassium carbonate as supporting electrolyte. Catalyst prepared from copper sulfate as precursor showed better activity showing the Cu redox peaks at -0,22 and 0,6 V vs Ag/AgCl. Thus, this catalyst was chosen to investigate the effect of the supporting electrolyte (KOH, $NaHCO_3$, $KHCO_3$ and K_2CO_3). Reduction of CO_2 was observed in all electrolytes, nevertheless, potassium carbonate and potassium hydroxide were more active. The onset potential for CO_2 reduction was -0,8 V vs Ag/AgCl and -0,9 V vs Ag/AgCl, respectively for carbonate and hydroxide electrolytes. Based on these results, we are running electrolysis with a gas diffusion electrode to verify the products formed and whether we can modify the copper catalyst, aiming to obtain C2-reduction products with greater added value.

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Mineralization and Proposed Sulfamethoxazole Degradation Route by photo electro-Fenton Process

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Electro-Fenton (EF) is one of the most efficient Electrochemical Advanced Oxidative Process (EAOP) in which organic compounds are oxidized in the presence of a solution with eletrogenerated *in situ* hydrogen peroxide (H_2O_2) and irons ions (Fe^{2+} /Fe^{3+}) acting as a catalyst [1]. Moreover, this process can be enhanced by irradiation with ultraviolet (UV) light, referred to as photo electro-Fenton (PEF). Advantages of EF and PEF compared to traditional Fenton and photo-Fenton AOPs is mainly the H_2O_2 to be generated *in situ,* without the need to handle and storage this agent [2]. In this way, this work focused on the degradation and mineralization of Sulfamethoxazole (SFX), an antibiotic, using both EF and PEF process (assisted by UVA light). The degradation of 10 mg L^{-1} SFX was carried out in a three-electrode bench cell in 0.05 mol L^{-1} K_2SO_4 (pH 2.5) as the support electrolyte applying 50 mA cm^{-2} in 0.5 L min^{-1} O_2 flow during 120 min. Iron salts were added to this system to give 0.25 mmol L^{-1} of Fe^{2+}, and H_2O_2 was electrogenerated by cathodic reaction of oxygen reduction using an efficient gas diffusion electrode (GDE) made with carbon Printex L6 supported on carbon cloth. Liquid and gas chromatography coupled with mass spectrometry (LC-MS and GC-MS) and ions chromatography (CI) was employed to investigate the by-products formed. As result, the PEF process was the most efficient, since it shows complete degradation of antibiotic in only 10 min and a partial mineralization of ~56% SFX. The low mineralization rate was explained by the accumulation of stable byproducts. By means of LC-MS it was possible to proposed three degradation routes, one of which involves the formation of polymeric compounds during the PEF process. The CI analyses showed an increase in the concentration of short-chain carboxylic acids, such as acetic and oxalic, which form stable complexes with Fe^{2+} and minimize degradation kinetics.

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Synthesis and modification of graphene oxide-cubic silsesquioxane matrix with Prussian Blue analogue for electrocatalytic detection of the pesticide Diuron

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The present work describes the preparation and chemical modification of the graphene oxide (OG) platform with cubic octa(aminopropyl)silsesquioxane (POSS), called OGS, and iron hexacyanoferrate (OGSFeH) for electrocatalytic detection of Diuron, a pesticide widely used and reported in the literature as genotoxic and carcinogenic [1]. The aforementioned nanostructured hybrid materials were characterized by different techniques: Raman Spectroscopy, Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM). It was possible to observe, through the bands referring to the condensation of cubic silsesquioxane to graphene oxide, the modification of the matrix, as well as characteristic indicators of Prussian Blue analogs, such as absorption bands in the infrared region and peaks in the diffractograms. Additionally, through the obtained micrographs, a substantial morphological alteration was observed in which the blocks, which are the aggregates of hexagonal carbon layers in graphite that, after chemical procedures and subsequent exfoliation, converted into graphene oxide sheets, followed by the appearance of cubic structures on the surface of the layers, due to the surface modification of GO with POSS, and with the subsequent formation of metal complex nanoparticles on the surface of GOS, corroborating the synthesis of GOSFeH proposed in this work. The cyclic voltammogram of the system composed of the graphite paste electrode modified with OGSFeH exhibited two distinct and well-pronounced redox couples, with average potentials $E_I^{\theta'} = 0.24$ V and $E_{II}^{\theta'} = 0.94$ V (KCl 1.0 mol L^{-1} ; $v = 5$ mV s^{-1} ; pH = 7.0; 10% w/w): the first refers to the redox process of the $Fe^{(II)}/Fe^{(III)}$ couple, and the second to the $Fe^{(III)}Fe^{(II)}(CN)_6$ /$Fe^{(III)}Fe^{(III)}(CN)_6$ redox couple of the metal complex formed [3]. The electrocatalytic behavior in the face of diuron detection was investigated using the Differential Pulse Voltammetry technique, with a detection limit of 4.96×10^{-9} mol L^{-1}, relative standard deviation of ± 1% (n = 3) and amperometric sensitivity of 30.59 A/mol L^{-1}. The Diuron is the target of investigation in the waters of rivers, lakes, seas and even in urban supply systems, therefore tests were conducted and indicated the low interference of cations and anions possibly present in these media in concentrations up to ten thousands folds greater than the substance of interest, therefore suggesting the inclusion of the compound OGSFeH as a material with the potential to be used in the manufacture of modified electrodes for the detection of Diuron in water samples.

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Development of screen-printed electrode with conductive ink based on commercial glue and graphite powder for sensing dopamine

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The disposable screen printed electrochemical sensors (SPCEs) presents advances such as low cost, sample volume reduction, miniaturization, mass production and portability, becoming prominent in Electrochemistry [1, 2]. Thus, in this work, was developed a new self-manufactured conductive ink from graphite powder as a conductive material, and a commercial glue based on polyvinyl acetate (PVA), originally used for craft purposes, as a polymeric vehicle, with the addition of solvents, acetone, ethyl acetate, and glycerin as additives. The substrate used for this for the production of the sensor was the aromatic aliphatic copolyester EcoflexTM due to its flexible characteristic. SEM, FTIR, contact angle and voltammetry, carried out the morphological characterizations. Thereby, the developed electrochemical sensor was applied for detection of dopamine, using square wave voltammetry in the range from 3.0 to 100 μmol L^{-1} and with detection limits of 0.078 μmol L^{-1}. The sensor was suitable for rapid detection of dopamine in artificial sweat with recovery between 98.3 and 111%. Therefore, it was possible to develop a low-cost screen-printed electrochemical sensor flexible with satisfactory analytical response using a lab-made conductive ink based in materials unusual for this proposes as a glue craft.

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Chemometric Mapping of the Nanoparticle Position in a Nanotube Matrix Simulated by Finite Element Method

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Porous materials coated with catalyst nanoparticles have had fundamental importance to promote hydrogen and oxygen evolution reactions. However, little is discussed about how relevant the disposition of this catalyst inside de porous matrix can be. In this work, it was built a model to describe the effect of the position of single iridium nanoparticle deposited on the inner wall of a titanium nanotube. The model is based on a Secondary Current Distribution, which consists of the Laplace equation and the Butler-Vomer equation [1]. The first one describes the electronic conductivity through the titanium and iridium and the ionic conductivity through the electrolyte, whereas the second one describes the charge transfer process at the Ti|electrolyte and Ir|electrolyte interfaces. It was assumed a conductivity of $2.05 \times 10^6\ S/cm$ for Ti, $2.13 \times 10^5\ S/cm$ for Ir and $0.2\ S/cm$ for the electrolyte, which is a typical conductivity for $0.5\ mol/l\ H_2SO_4$, and the exchange current density for the hydrogen evolution reaction for the same electrolyte composition is $1.54 \times 10^{-5}\ mA\ cm^{-2}$ at Ti and $0.20\ mA\ cm^{-2}$ at Ir [2]. It was admitted a cylindrical nanotube $100\ \mu m$ long under a constant current density of $-100\ mA\ cm^{-2}$, and the response is the overpotential due to the charge transfer process and the ohmic drop through the metals and the electrolyte. Four variables were chosen to be investigated in 5 different levels: nanoparticle radius (r_{NP}) from 88.88 to $133.34\ nm$, nanotube radius (r_{NT}) from 133.34 to $200.00\ nm$, nanoparticle position (z'_{NP}) from -95 (bottom) to $-5\ nm$ (top) and the nanotube density (ρ_{NT}) from 1 to $4\ tubes/\mu m^2$ under a multivariate perspective by using a central composite design with star points [3]. As a way to introduce the experimental error, a random contribution was added to each level within a range of $\pm 5\%$. It enables the selection of main and cross effects as well as the construction of response surfaces that expands the range of conditions originally investigated. Figure 1A shows the Pareto chart where the main and cross effects are represented, it can be seen that the most significant effect is the ρ_{NT} because the greater the pore density, there is more active catalyst surface through which charge transfer can occur, and therefore the lower the overpotential. The z'_{NP} is the third most important effect, and according to its relative contribution (not showed here) when the position of the nanoparticle is changed from $-5\ \mu m$ (closer to the surface) to $-95\ \mu m$ (further from the surface), it implies an 11% more cathodic overpotential on average, which means that the nanoparticle at the bottom of the tube is less active and it requires an additional amount of applied potential to yield the same current density. Additionally, the response surface in Figure 1B shows the combined effect of the ρ_{NT} and z'_{NP} on the overpotential, and the better condition is found when both variables are in their highest levels.

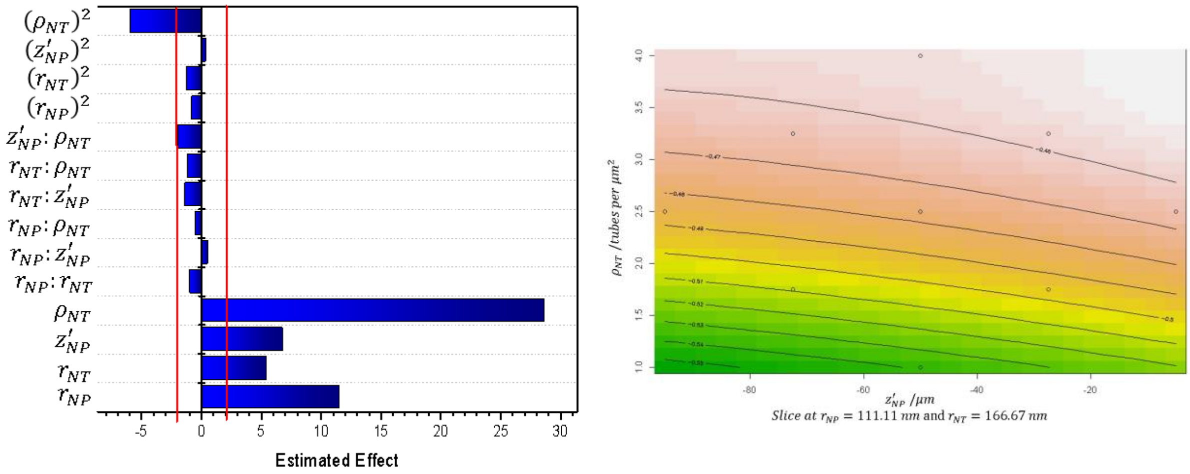

Figure 1. A) The Pareto chart showing the main and cross effects and B) a response surface which represents the behavior of the overpotential as a function of both ρ_{NT} and z'_{NP} for fixed values of $r_{NP} = 111.11\ \mu m$ and $r_{NT} = 166.67\ \mu m$.

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Electrosynthesis of Polyaniline Copolymers for Organic Electrochemical Capacitors

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Organic supercapacitors represent an exciting frontier in energy storage technology, offering a promising alternative to traditional batteries. Their high electrical conductivity and rapid charge and discharge capabilities are ideal for applications requiring high peak power. Using conductive polymers as electrodes improves energy efficiency and introduces flexibility into the design of electronic devices. The synthesis of copolymers, such as polyaniline, allows the creation of materials with adjustable electrical properties, opening a range of possibilities for innovation in diverse fields, from consumer electronics to regenerative medicine [1].

The electrodeposition synthesis process, controlled by cyclic voltammetry, is a precise method that guarantees the formation of uniform copolymer layers on transparent FTO. This technique allows for rigorous control over the morphology and chemical composition of the material, which is crucial for optimizing charge storage properties. Subsequent electrochemical characterization in acidic solutions provides valuable data on the copolymer's energy storage capacity and stability during repeated charge and discharge cycles [2, 3]. In this work, the use of aniline (Ani), ortho-methoxy aniline (oMA), and 3-aminobenzene sulfonic acid (SAn) as precursors of copolymers was studied, and the proportion of these was varied to analyze their effects on the polymer obtained and its response on capacitance. The results revealed a strong influence with capacitances ranging from 40 to 280 F g^{-1} depending on the proportion used in electrosynthesis. These results can be related to the major modification of the electronic structure of these materials due to the addition of ring substituents with different characteristics on the amino-substituted aromatic ring. This research underscores the great potential of these materials in composites for energy storage.

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Screen-printed electrode based on *Apis mellifera* beeswax modified with Printex 6L carbon@chitosan/Au for sensing of folic acid

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Abstract

The present work reports the development of a highly sensitive electrochemical platform, constructed using screen printed electrode based on *Apis mellifera* beeswax (SPWE) – employed as substrate – modified with Printex 6L Carbon (P6LC) at Chitosan (Chi) plus electrodeposited Au, and its application for Folic acid (FA) detection and recovery and real samples, since it is a crucial vitamin for regular physiological functions, as well as, is essential for DNA and RNA formation [1]. The beeswax utilization emerges from the necessity of ecofriendly materials and the possibility of beeswax reutilization for SPWE manufacturing, making it an interesting alternative for application in species detection systems [2]. The electrode was named as SPWE/P6LC@Chi/Au. For the electrodes production, the beeswax is melted on plates with thickness of 0.1 cm, being the graphite ink, applied on its surface. The graphite ink was studied by cyclic voltammetry (CV) with 1.0 mmol L^{-1} (KCl 0.1 mol L^{-1}) Fe $(CN)^{3-/4}$ in different proportions of graphite powder (G)/varnish glass (VG) (w/w), being: 50% G/50% VG, 60% G/40% VG and 70% G/30% VG. Also, a study varying the number of graphite ink layers (two, three and four layers) was carried out by CV. The same wax was reused three times, and tests made by CV were carried out, resulting in electrochemical profiles that demonstrated stability, with a decay of 6,2% in current. The electrochemical characterization was performed by CV with 1.0 mmol L^{-1} (KCl 0.1 mol L-1) $Fe(CN)^{3-/4}$ as supporting electrolyte for the reversibility parameters. The electrode with 3 ink layers and a proportion of 70% graphite:30% varnish, obtained the best results in terms of reversibility parameters, reaching

values of electroactive real area of 90.8%; i /i= 1.04; ΔEp= 139.3 mV e K0=1.68x10^{-3} cm s^{-1} and therefore was used for further electrochemical analysis. The kinetic study was made by CV in the potential range of 5 to 100 mV s^{-1}, obtaining R^{2} values of 0.99271 and 0.98993 for the anodic and cathodic linearity, respectively. The stability of the system was tested by chronoamperometry for 4.5 h, presenting a current deviation of 0.16 µA. For FA detection (2x10^{-4} mol L^{-1} at BR buffer, pH 6.0) at SPWE/P6LC@Chi/Au sensor, DPV was carried out and the obtained current response was 7.8 µA. This sensor, when compared with other platform (SPWE), had an increase in the electrochemical sensitivity of 84%. An analytical curve was realized in the linear range of 0.025 – 0.75 µmol L^{-1} of FA at BR buffer pH 6.0, and LOD= 0.00945 µmol L^{-1} was obtained, with mean recovery rates of FA in female urine of 101.7%, demonstrating the detectability, sensibility and accuracy of SPWE/P6LC@Chi/Au, making it a viable alternative for FA detection in real samples.

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Arduino sensing system for real-time monitoring of textile effluent degradation

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The use of Arduino-based sensing systems for real-time monitoring of the degradation of textile effluents represents an innovative and economical approach to environmental management. These systems allow for continuous data collection, facilitating the rapid identification of changes in the composition of effluents, which is crucial for implementing effective corrective measures. Therefore, this work focused on integrating water quality monitoring sensors into a single platform to monitor photodegradation through the discoloration of textile effluents, which generally have a very high color load. Thus, using sensors such as turbidity (ST100), color (RGB TCS34725), conductivity (ZW-TDS102 TDS), temperature (DS18B20 and DHT-11), and luminosity (photodiode), it is possible to map the factors that are effective in treating bodies of water. The system allowed real-time observation of ambient temperature, sunlight power, and effluent parameters such as temperature, color change, turbidity, and conductivity. Although these are not the only parameters to be observed, monitoring water quality and the evolution of oxidative processes in real-time is possible, allowing one to take action for immediate remediation. Furthermore, more sensors may be added to make this monitoring increasingly precise and assertive.

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Hydrodynamic characterization, CFD simulation and application of flow reactor with DSA in anodic oxidation

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Introduction

Electrochemical advanced oxidation processes (EAOP) have been highlighted due to their advantages such as environmental compatibility, ease of operation, high efficiency, rapid reaction rate and usage time. At laboratory scale, magnetically stirred batch reactors configuration, although simple, makes it difficult to transport mass from the bulk of the solution to the electrode surface. Then, new designs of electrochemical reactors have been proposed, with electrodes arranged according to the structure of the electrochemical reactor, which is also categorized by type of flow [1]. In this study, a Ti/$TiO_2RuO_2IrO_2$ DSA was used to investigate the impact of the geometry of a flow recirculation reactor with rectangular parallel plates, on mass transfer performance under different inlet rates, as well as on removal efficiency of an organic pollutant (Norfloxacin drug).

Materials and methods

The experiments were carried out using a batch reactor with flow recirculation, where the Ti/$TiO_2RuO_2IrO_2$ anode and Ti cathode in rectangular shape were arranged in parallel position. Hydrodynamic linear sweep voltammetry measurements were carried out in a solution of 0.005 mol L^{-1} $K_3Fe(CN)_6$] and 0.01 mol L^{-1} $K_4[Fe(CN)_6]$ in 0.5 mol L^{-1} NaOH to determine the diffusion coefficient. The value of the experimental k_m was determined by the diffusion limit current technique, from which the dimensionless numbers of Re, Sh and SC were estimated, and mathematically correlated to obtain the equation that governs the proposed hydrodynamic system [2]. The flow rate distribution inside the reactor was verified by Computational Fluid Dynamics (CFD) simulation, with Simcenter 3D software [3]. The best operational conditions were used for degrading Norfloxacin (NOR) drug.

Results and discussion

The results showed that the experimental k_m values were strongly dependent on the flow rate; high fluid velocities in the reaction chamber favor the mass flow of species towards the electrode surface, reducing the thickness of the Nernst diffusion layer as well as improving the electron transfer kinetics, consequently increasing the reaction yield. The values of the dimensionless numbers (Sh, Sc and Re) confirmed the relationship between the increase in mass transport and the increase in flow; the mathematical correlation of dimensionless numbers indicated that the flow of the hydrodynamic system operates predominantly in a turbulent regime, regardless of the applied flow; the Computational Fluid Dynamics (CFD) simulation confirmed the increase in mass transport at high flow rates, due to the formation of turbulent zones; and the optimal condition for using the proposed reactor was at a flow rate of 500 L h^{-1}, which allows a $\mathrm{k_m}$ of 1.9×10^{-4} m S^{-1}. The efficiency of the proposed reactor with $Ti/TiO_2RuO_2IrO_2$ anode, operating under current limit conditions, was confirmed in the electrochemical oxidation of 2.5×10^{-4} mol L^{-1} of NOR drug, which promoted a removal of 92% of its concentration in 6 h. The use of DSA as an anode material in reactors for application in EAOP is a viable alternative, favoring the increase in k_m

Conclusions

The association of high flow (higher km) with turbulent mass transport resulted in an efficient performance of the reactor, whose operation is substantially improved by working in conditions of limiting current density, which promotes an acceleration effect degradation efficiency. This study provides a better understanding of the hydrodynamic behavior of parallel plate electrochemical reactors and the rectangular shape of the plates is a potential alternative for removing organic pollutants from water.

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Investigation of the synergistic effect of $Fe_{1.9}V_{0.1}O_3$ nanoparticles as modifiers of Printex L6 Carbon to oxygen reduction reaction for H_2O_2 Electrogeneration

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Hydrogen peroxide (H_2O_2) is an oxidizing compound that has many applications, particularly in the treatment of industrial effluents [1]. Electrochemical technology has been developed to produce H_2O_2 *in situ* through the oxygen reduction reaction (ORR) via 2 electrons pathway, using carbonaceous materials such as Printex L6 carbon (PL6C) [2]. In order to improve the high selectivity and efficiency of PL6C for H_2O_2 electrogeneration, this work studies the synthesis, structural and electrochemical characterization of $Fe_{1.9}V_{0.1}O_3$ nanoparticles (NPs) and used as modifier of PL6C in order to increase the efficiency of ORR via 2 electrons. The NPs were obtained using the Fe^{2+}, Fe^{3+} and V^{5+} salts by co-precipitation method. These materials were characterized by X-ray diffraction (XRD), Raman spectroscopy, scanning electron microscopy (SEM-FEG) and transmission electron microscopy (TEM) to determine their crystal structures and morphological analysis. The X-ray photoelectron spectroscopy (XPS), electrochemically active area (EQSA) and the zero charge point (pH_{ZPC}), as well as the contact angle were also determined to understanding the interaction between the NPs and the PL6C. In addition, PL6C modified with $Fe_{1.9}V_{0.1}O_3$ NPs was analyzed by Cyclic Voltammetry (CV) and Linear Voltammetry (LV), with a rotating ring-disk electrode system (RRDE). These analyses were carried out at pH 3.0 and 9.0 to study the electrocatalytic activity, determine the average selectivity for H_2O_2 formation via ORR and the number of average electrons involved in the process. The results of the structural analysis indicate the presence of two oxide phases: maghemite (Fe_2O_3) and hematite (γ-Fe_2O_3), with an average size of 8.0 mm (± 0.5) and spherical morphology, similar to PL6C. The surface interactions indicate a greater interaction of the modified carbon with the electrolyte and the charges present in the medium, based on the decrease in contact angle (from 113º to 99º) and increase of specific capacitance (from 75.6 $\mu F.cm^{-2}$ to 109.2 $\mu F.cm^{-2}$) of the 98% PL6C@2% $Fe_{1.9}V_{0.1}O_3$ catalytic coatings. In addition, this composition showed an increase in selectivity for H_2O_2 production by ORR when compared to PL6C, at both pH (6.2 and 16.2%, respectively).

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ESTUDO ELETROQUÍMICO DE COMPLEXOS DE RUTÊNIO(II)-ARENO-TRIARILFOSFINAS

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Complexos de Rutênio(II)-areno têm sido explorados como potenciais metalofármacos e alguns complexos, como RM175 e RAPTA-C, estão em ensaios clínicos. O ligante areno coordenado ao Rutênio(II) atua como uma plataforma para o desenvolvimento de novos complexos, devido a versatilidade na troca de ligantes e capacidade de estabilizar o estado de oxidação do Ru^{2+} sob condições fisiológicas, que é o estado ativo no meio biológico. Dentre as possibilidades de ligantes utilizados, as triarilfosfinas mostraram-se promissoras na inibição do crescimento de células cancerígenas.

Este trabalho descreve o estudo eletroquímico de uma série de complexos de Rutênio(II)-areno-triarilfosfinas de fórmula geral $[Ru(\eta^6\text{-p-cimeno})PAr_3Cl_2]$ por meio da voltametria cíclica. Os complexos apresentaram um perfil eletroquímico semelhante, com um processo redox quasi-reversível na região anódica, centrado no par RuII/RuIII. Os complexos com hidroxila como substituinte da triarilfosfina, apresentaram um processo de oxidação irreversível em maior potencial atribuído à formação de um radical fenóxi originado pela perda do átomo de hidrogênio. As varreduras na região catódica mostraram dois processos irreversíveis independentes da oxidação, correspondentes às reduções do centro metálico (RuII/RuI e RuI/Ru0).

A diferença entre os valores de potencial de pico anódico e potencial de pico catódico fornece a faixa de estabilidade da janela de potencial do rutênio no estado de oxidação (2+), quando coordenado ao ligante η^6-p-cimeno. Para os complexos dessa série os valores foram entre 3,15 a 2,89 V, estando em uma faixa de potencial mais ampla quando comparada à fisiológica (+1V a -1V vs. EPH), sugerindo que os complexos da série sintetizada mantenham o rutênio em seu estado de oxidação 2+ in vivo.

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Eletro-oxidação de moléculas modelo de ligninas sobre eletrodos de Ni

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In a context of climate crisis, sustainable development and the environment have been frequently discussed nowadays, especially regarding fossil fuels applications. In this sense, biofuels are a promising alternative, however, the application of biomass residues from that fuels' generation has still room for improvement since their main use is still the burning for thermoeletricity. Lignin is the main source of aromatic carbons in biomass, following cellulose and hemicellulose. This macromolecule has, as monomers, phenolic structures and is an interesting renewable aromatic chemicals source. However, due to the complexity of the monomer connections, the depolymerization of lignins is still not optimized and this reaction mechanism is still under debate. Electrocatalysis was selected as the main tool for this work because it is an easy to control approach that can be applied for the lignin depolymerization by oxidation under ambient conditions, or close to them, and can easily provide kinetic and mechanistic information. For substrate simplification, in this work we proposed to use of model molecules based on the phenolic monomers structure containing a carbonic chain in the *para* position with sizes between one and three carbons, and equivalents containing a metoxyl group in *meta* position were selected as model molecules to comprehend the electrochemical behavior of lignins during oxidation processes. As electrocatalyst, Ni plate electrodes were selected due to the low cost and stability in alkaline conditions.

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Nanoestruturação eletroquímica de eletrodos de Au intensificados para eletroquímica intensificada por plasmônica

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In search of new energy technologies, explore redox reactions to convert earth-abundant renewable feedstocks into value-added fuels, became an important study. This is because renewable energy sources are dependent on weather conditions, such as PV and wind energy. Because of this, energy storage is essential to generate. In this sense, the use of plasmonic assisted water electrolysis, using nanostructured Au electrodes, can be used to produce hydrogen as a solar fuel, a way to chemically store renewable energy. Plasmons are the result of the interaction of some metal nanostructures with electromagnetic waves at resonant frequency. This resonance creates oscillating localized electrons (plasmons – localized surface plasmon resonance – LSPR) and by such movements, these electrons are excited and generate the so-called hot charge carriers. In the present work, we propose the use of electrochemical processes, that allows high level of control by the electric variables, such as current and potential, and it is a low-cost process. We investigated the nanostructuration of Au bulk electrodes, using a standard three-electrode electrochemical cell, a platinized titanium net as the counter electrode and a encapsulated H_2 bubble as the reference. The electrolyte was sulfuric acid, 0.5 mol L^{-1}.

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Towards environmentally friendly catalysts for H_2O_2 electrogeneration: obtaining and using amorphous carbon from waste as support for Pd-single atoms

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Hydrogen peroxide (H_2O_2) is one of the most important chemical compounds in modern society, being used in diverse applications such as a bleaching agent, medical disinfection, and environmental oxidant.[1] The prevailing method for its production involves anthraquinone reduction, constituting over 90% of the global production.[1] Due to challenges in storage and the need for limited quantities in certain applications, the in situ electrogeneration of H_2O_2 has garnered interest.[2] Typically, carbonaceous materials are employed in electrochemical H_2O_2 generation, either in their pristine state or as a support for metal catalysts. Commercially, the carbon used is derived through the unsustainable and environmentally unfriendly incomplete combustion of heavy petroleum products at temperatures as high as 2000 ºC. In this study, we propose a sustainable approach by utilizing carbon derived from renewable sources as a support for Pd single-atom dispersion. Three diverse sources were chosen: sewage sludge, a by-product of biological wastewater treatment plants with limited reuse due to high impurity levels; sugarcane bagasse, a residue generated in significant quantities (180 Mt in Brazil in the 2022/2023 crop); and tannin/cellulose, low-cost biomasses with potential for transformation into high-value functional materials. The methodology for obtaining carbon materials from sewage sludge and sugarcane bagasse involved impregnation followed by pyrolysis, varying temperature, holding time, and activator type/concentration. For tannin-cellulose, a xerogel route using HNO_3 was employed. Subsequently, Pd single-atom modification was achieved through a wet impregnation process using $Pd(acac)_2$ precursor. Results revealed distinct physical morphologies in the obtained amorphous carbon, particularly in structure, total surface area, and functional groups. Nonetheless, the modification using Pd single-atoms successfully presented Pd dispersion in the sub-nanometer scale. Electrochemical results showed that the onset potential taken at 0.1 mA cm^{-2} variated from 0.527 V vs. RHE when the material was supported on the carbon obtained from sewage sludge until 0.700 V vs. RHE for the single atoms dispersed over the carbon obtained from tannin-cellulose biomasses. Selectivity across all materials ranged from 80-90%, slightly lower than Pd over Vulcan carbon.[3] In conclusion, carbon materials obtained from waste resources exhibit promise as support for metals in H_2O_2 electrogeneration, enhancing the sustainability and environmental friendliness of such applications. While the materials in this study demonstrated overall positive results, the synthesis route for this application is in its early stages, necessitating further optimization. The use of lower temperatures (400-900 ºC, holding time max 2h), lower concentration of activating agents (0-20 %m/V), and utilizing near-zero-cost residues make this synthesis route highly attractive, holding potential for significant attention in the coming years.

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Selective Bioelectrooxidation of Seawater using Abiological Copper-containing Proteins

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Seawater is widely considered an abundant resource for water splitting reaction to produce molecular hydrogen (H_2) and oxygen (O_2). However, its practical application is hampered due to chlorine-related competing reactions at the anode. We overcome the practical limitation of bioelectrooxidation of seawater using apo-reconstituted proteins with abiological copper(II) metal centers dispersed in heteropolysaccharide polymer shell. Our strategy showed significant advances for bioelectrooxidation of water both at pH 9 and in seawater (pH 7.6), with Cu(II)Myo having onset potential for OER ~10 mV lower compared to solely Cu(II)P, concomitantly to the track of reaction products using online differential electrochemical mass spectrometry (DEMS). The data was also supported by the monitoring of the electronic structure of the redox states Cu^{+2}/Cu^{+1} by in situ XANES absorption spectroscopy and electrochemistry.

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Activated carbon synthesis using sugarcane bagasse for electrochemical applications

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Hydrogen peroxide (H_2O_2) is one of the most used oxidants [1]. Electrochemical technology allows the synthesis of H_2O_2 in an aqueous medium, but it is necessary to study the electrodic material to direct the reduction of O_2 by 2-electron mechanism. In this sense, the present work aimed to study the synthesis of active carbon, for application in the generation of H_2O_2, using sugarcane bagasse with activator KOH and NaOH. For the synthesis of carbon, the process of calcining sugarcane bagasse was used in the presence of an activating solution (different proportions KOH:NaOH) at temperatures ranging from 400 °C to 900 °C for up to 120 minutes. Each material was electrochemically characterized using the porous micro-layer technique on a rotating disc-ring electrode using linear sweep voltammetry in 0.05 mol L^{-1} K_2SO_4 electrolyte pH 3 saturated with O_2, according to the methodology described in the literature. The best materials were characterized by XPS, FEG, DRX and BET. The main results demonstrated that the electrochemical activity of the synthesized materials is associated with the influence of the temperature associated with the presence of the activating solution, achieving maximum electrochemical activity for the generation of H_2O_2 (detection on the Pt ring) at 650 °C for 60 minutes using 10% activation solution. Regarding activation, the results demonstrated that the combined use of NaOH and KOH (in different proportions) did not promote significant improvements in the materials, with the material using 100% NaOH presenting the best results for H_2O_2 generating.

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Glucose and 5-hydroxymethylfurfural adsorption at Pt(111) in alkaline media

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The electrochemical oxidation of derivative biomass molecules offers a greener route to produce added-value molecules without the use of fossil fuel [1]. Glucose is an important saccharide with large applications and can be dehydrated in acidic conditions to 5-hydroximethilfurfural (5-HMF), an important platform molecule in the industry, since it can be oxidized to furan dicarboxylic acid (FDCA), a bio-based monomer that is a precursor to furandicarboxylate polyethylene (PEF), that has been considered to replace the terephthalate or isophthalic acids to produce polymers [2]. In this scenario, the electrochemical oxidation of glucose and 5-HMF at Pt(111) was studied in alkaline media focusing on adsorption and surface poisoning at concentrations from 10^{-6} to 10^{-1} M. Glucose was able to interact with Pt(111) and be oxidized at both oxide-free and (hydro)oxide-covered surfaces, the current being dependent on the glucose concentration. The presence of adsorbed hydroxyl ions (Pt(111)-OH) facilitates the glucose oxidation reaction leading to the lowest surface poisoning along the experiments. Contrary, the 5-HMF strongly interacts with Pt(111) and although oxidation currents at Pt(111)-OH region increase with 5-HMF concentration, inhibitory effects are observed for concentrations higher than 10^{-4} M. According to the literature, the presence of (hydro)oxides species is essential for furanic compound oxidation after its adsorption. Therefore, the 5-HMF acts as its own poison. In this way, glucose presents a higher activity at Pt(111) than 5-HMF, even in higher concentrations.

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Carbon nanotubes and silver nanoparticles as electrode modifiers for the determination and electro-remediation of sulfamethazine

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The contamination of water resources with emerging contaminants is a global problem, and new water treatment methodologies must be developed in order to guarantee the supply of drinking water and reduce environmental impacts [1]. Sulfamethazine (SMZ) is an antibiotic found in surface and groundwater that can cause impacts for the environment and the population even at low concentrations [2,3]. Therefore, it is essential to develop innovative approaches for the SMZ determination and degradation. This work focuses on the modification of a glassy carbon electrode (GC) for the determination and electro-remediation of sulfamethazine (SMZ) in natural waters using multi-walled carbon nanotubes (MWCNT) and silver nanoparticles (AgNPs) as electrode modifiers. The proposed GC electrode modifier was characterized morphological and electrochemically demonstrating that the silver nanoparticles was incorporated onto the MWCNT. Voltammetric parameters of the SMZ oxidation process were optimized to improve the response in the analysis. A linear response range was obtained from 0.3 to 5.0 µmol L^{-1}, and the limit of detection and limit of quantification obtained was 0.19 µmol L^{-1} and 0.63 µmol L^{-1}, respectively. This electrode was used for SMZ quantification in natural waters and interferents were used in a selectivity study. Finally, the GC/MWCNT-AgNPs was applied for the remediation of SMZ using chronoamperometry with +1.5 V for 2.5 h, decreasing 62.04% of the antibiotic. As a result, MWCNT-AgNPs were found to be an excellent option for the effective determination and remediation of the SMZ.

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Development of a Low-Cost Sensor for Methylparaben Detection in Natural Waters

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Methylparaben (MePa), commonly used in personal care products and foods as a preservative, is increasingly detected in natural waters, raising significant environmental and health concerns due to its endocrine-disrupting capabilities [1,2]. This study presents a novel approach to the real-time monitoring of MePa through the development of a disposable electrochemical sensor that is both cost-effective and efficient. This work aims to develop a disposable sensor modified with biochar and copper nanoparticles (BC-CuNPs) for detecting MePa in natural water samples. For the sensor's construction, a conductive ink was crafted and optimized using 810 mg of Graphite (GR) as the conductive material, 5 mg of Sodium Alginate (AS) as a binder, and 2.5 mL of ultrapure water to ensure the ink's viscosity. An adhesive mask was applied to a pre-sanded and cleaned PET substrate, outlining the regions for the working, auxiliary, and reference electrodes, which were then filled with the GR-AS conductive ink using a spatula. The sensors were dried in an oven at 50 °C for 30 minutes. The resulting Disposable Sensor (DS) sensor was characterized by Electron Microscopy (FEG-SEM), X-ray Dispersive Spectroscopy (EDS), Infrared Spectroscopy (FTIR), and electrochemically through Cyclic Voltammetry (CV) and Impedance Spectroscopy (EIS). After the characterization steps, the sensor was modified with Biochar (BC) and Biochar-Copper Nanoparticles (BC-CuNPs) materials, where the performance of the different sensors in detecting MePa was compared. The DS/BC-CuNPs sensor exhibited the best voltammetric response for the electro-oxidation of 0.01 mol L^{-1} of MePa, presenting an Anodic Peak Potential (E_{pa}) = +835.1 mV *vs.* Graphite. The sensor also showed a 2.86-fold increase in Anodic Peak Current (I_{pa}) compared to the unmodified sensor, being an advantage for future analyzes of environmental interest.

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Green Hydrogen Production Assisted by Ethanol Electrooxidation in Alkaline Media using Ni-B Intermetallic Materials

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The generation of green hydrogen, that is hydrogen produced without carbon dioxide emissions, is one of the most significant tasks of the energy transformation. Electrochemical ethanol reforming in alkaline media can be considered as a viable method for green H_2 production in Brazil, based on a renewable and abundantly available feedstock, ethanol. Nonetheless, there is a need to develop effective and inexpensive HER catalysts for this process to be economically feasible. This work focuses on the preparation of Ni–B intermetallic catalysts for the HER since they are more affordable than the platinum-based catalysts. Nickel-boron (Ni-B) catalysts' physical properties were examined by X-ray diffraction (XRD) which displayed the formation of the Ni_3B phase when the sample was heated at 600ºC and 980ºC. The electrochemical measurements indicated that the catalytic activity for HER enhanced in the presence of ethanol, which supported the notion that ethanol electrooxidation enhanced the H_2 production. Also, to assess the stability of the Ni-B catalysts, the cyclic voltammetry test was performed and it was observed that the catalysts were quite stable for use in ethanol electrochemical reforming cells.

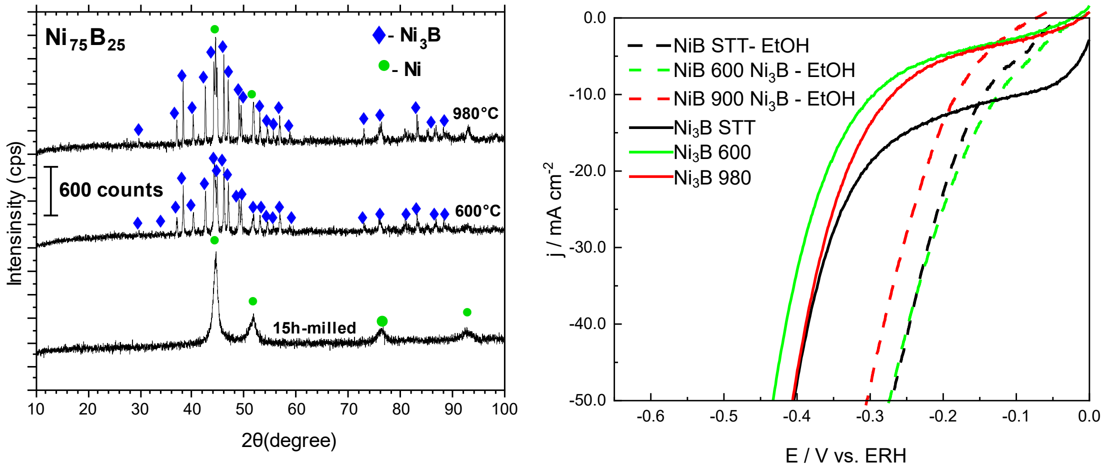

Figure 1: (a) XRD of Ni-B alloys at different temperatures, highlighting Ni_3B formation. (b) Polarization curves of catalysts in 4M KOH + 1M ethanol at 85°C.

Thus, the Ni-B intermetallic catalysts prepared in this work can be considered as promising for green hydrogen generation from ethanol in the basic environment. The findings of the study can therefore be useful in the creation of improved and sustainable H_2 production systems with a great implication on the Brazilian energy sector. The future work will be devoted to the further enhancement of the composition and porous structure of Ni-B catalysts for achieving higher activity and durability of the catalyst.

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Production of NH3 through electrochemical reduction of N2 using GDL cathode modified with MoS2 and NiFe alloy.

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Electrochemical N_2 reduction (NRR) is an alternative to produce ammonia at room temperature and atmospheric pressure compared to the traditional Harber-Bosch Process [1]. However, most catalysts do not have efficient nitrogen fixation activity [2]. In this sense, this work synthesized MoS_2-FeNi materials as a promising catalyst for NRR under ambient conditions. Physical characterization showed the amorphous nature of MoS_2 and FeNi was well distributed over the surface of MoS_2. In 0.1 mol L^{-1} Na_2SO_4 applying -0.75 V vs Ag/AgCl, the MoS_2-FeNi material showed a production rate of 0.78 µmol h^{-1} cm^{-2} and faradaic efficiency (FE) of 6.3%. This value is low, however is comparable our better that other electrocatalysts reported in literature, showing the material can be competitive in optimized conditions [3,4]. At all potentials, there was no formation of N_2H_4, indicating the only ammonia is produced from NRR. The lower efficiency is due hydrogen evolution, which occurs simultaneously with the NNR. These initial results are promising compared with other catalysts reported at literature and could be improved optimizing the synthesis conditions and amount of catalysts.

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Evaluating the impact of contaminants on the electrochemical performance of chemical vapor deposition (CVD) graphene for field-effect transistor-based biosensors

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Graphene, a two-dimensional material, has been employed in a wide range of applications due to its exceptional mechanical and electrical properties. Monolayer graphene was first obtained in 2004 through mechanical exfoliation, demonstrating excellent electrical conductivity [1]. Although mechanical exfoliation remains valuable for producing high-quality graphene for research purposes, its limitations in scalability, uniformity, reproducibility, and cost hinder its application in commercial and industrial settings [2]. Chemical vapor deposition (CVD) is a widely used method for producing high-quality, large-area monolayer graphene for microelectronics applications. This method offers several advantages, including obtain large areas, reproducibility, and versatility, making it suitable for both industrial and research applications. However, one of the challenges in integrating CVD graphene in CMOS (complementary metal oxide semiconductor) technology lies in the transfer process from the metal precursor substrate (commonly copper), to the target substrate, keeping the quality of the graphene. Wet transfer methods, such as copper etching and electrochemical delamination, are commonly used to transfer CVD graphene to the target substrate. In this way, we address an evaluation study in the spectroscopy, morphological, and electrochemical properties of CVD graphene, in the function of the transfer method used, such as copper etching and electrochemical delamination, aiming to select the more suitable transfer method to achieve high quality graphene field-effect transistor-based biosensor. Large area (9 x 11 cm) CVD graphene was synthesized using a furnace, TCVD-50B dual furnace halogen graphene square. CVD graphene areas of 1 by 1 cm^2 were coated with polymethyl methacrylate polymer (PMMA) as a support layer and transferred to SiO_2 (300 nm)/Si substrates using the convection wet transfer method (based on Marbles' reagent) and electrochemical delamination. Our preliminary results show that the transfer method strongly affects graphene's structural and morphological properties. The CVD graphene transferred by convectional wet transfer method (employing marble's reagent) showed a higher presence of D bands in the Raman spectra, related to defects in the material's structure and the higher presence of wrinkles. The following steps of this work aim to evaluate the presence of contaminants from the transfer process (metal particles and residues of PMMA) in the electrical and electrochemical performance of the CVD graphene.

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Development of an Electrochemical Biosensor for the Detection and Quantification of the Hormone Melatonin in Extra-Pineal Organs

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Melatonin (*N*-acetyl-5-metoxytryptamine) is a hormone synthesized in mammals primarily in the pineal gland in response to darkness [1]. Among the wide range of beings that synthesize it, there are many functions attributed to this hormone. Studies report its utilization by repressing AKR1C1 and AKRC3 protein levels, which are cancerous cells [2], besides being utilized in neurodivergent individuals such as those on the autism spectrum, with depression, Alzheimer's disease, and schizophrenia, among others [3]. The present study seeks to detect and quantify melatonin present in extra-pineal organs, such as the liver, intestine, gonads, and ovaries. For this, Wistar rats will be used, which will be euthanized following outlined ethical guidelines. These organs will be removed and dried using liquid nitrogen. Melatonin present in these organs will be purified and transformed into powder, to be added to electrochemical cells containing phosphate saline buffer (PBS) 0.01 M, pH 7.4, coupled with a μAutolab FRA 2 Type III (Metrohm, NL) potentiostat, using NOVA 2.1.7 software. Cyclic voltammetry (CV) experiments will be conducted to characterize the electrodes, square wave voltammetry (SWV) to detect the current signaling the presence of melatonin in the system, and Electrochemical Impedance Spectroscopy (EIS) as a complementary technique to SWV, analyzing the impedance presented by melatonin in the utilized setup.

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Evaluation of the electrocatalytic activity of silver phthalocyanine supported in Printex-L6 carbon for the oxygen reduction reaction

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Hydrogen Peroxide (H_2O_2) is an oxidizing agent used in chemical and biological processes, however, its industrial production from the anthraquinone reduction requires the use of big amounts of organic solvent, temperature and pressure, in addition to the risks associated with transportation and storage[1]. The electrogeneration of H_2O_2 *in situ* from the Oxygen Reduction Reaction (ORR) is a viable and advantageous alternative. The Printex-L6 carbon (PL6C) has been applied as an ORR electrocatalyst because it has carboxyl and carbonyl groups in its structure that favors the O_2 gas adsorption resulting in high selectivity for formation of H_2O_2 through the $2e^-$ transfer[2]. The electrocatalytic activity of PL6C for ORR can be enhanced by modifying it with silver phthalocyanine (AgPc). That way, the ORR studies in electrolyte (0.1 mol L^{-1} K_2SO_4) acid (pH= 3) and alkaline (pH= 9) were realized using a rotating ring-disk electrode (RRDE GC/Pt), Pt counter-electrode and reference electrode Ag/AgCl. In the RRDE surface was deposited the electrocatalyst matrices compound by PL6C unmodified and modified with 0.5, 1 and 1.5% of AgPc (w:w %). Cyclic and linear voltammetry were used to evaluate the behavior of the electrocatalysts and whit the data obtained were calculated the values of the selectivity for H_2O_2 formation and electrons number. The modification of PL6C with 1% de AgPc increase the selectivity of 90% to 97.5% for the ORR in alkaline medium. Already in acid medium the better modification was equivalent at 0.5% of AgPc increasing the selectivity of PL6C of 75% for 90.3%. In these conditions, the electron number was equivalent at 2.0 and 2.2 respectively for the ORR in alkaline and acid medium, establishing that the modifications made the PL6C more selectivity for H_2O_2 formation in both media. The morphology of the electrocatalysts were evaluated by electron microscopy techniques (SEM and TEM-EDS).

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Synthesis of TiO_2 Nanotubes on Ti Substrates for Implant Applications: Synthesis parameters and Controlled Substance Release Systems

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Bone implants have been widely investigated to increase the success and adhesion of prostheses. In particular, the use of pure commercial titanium stands out due to its biocompatibility and mechanical properties. In this way, modifying the surface of the material can help the osseointegration process by altering characteristics such as morphology, wettability, and crystalline phase [1]. The synthesis of TiO_2 nanotubes on the surface of these materials can help the adhesion process and act as a compartment for the immobilization of drugs, thus acting in the controlled release process. The nanostructures are synthesized mainly using anodic oxidation, in a potentiostatic regime, using a solution containing fluoride ios, derived from ammonium fluoride, NH_4F. The process initially consists of forming a barrier oxide film, then dissolving the film in contact with fluoride ions, consequently forming nanostructures [2]. In this study, the pure commercial titanium samples underwent a pre-treatment process, using #600, #1200, #1500, #2000, and #3000 grit sandpaper and polishing using 6 μm, 3 μm, and 1 μm diamond pastes, respectively, ending with cleaning with neutral detergent and acetone. The prepared samples underwent the potentiostatic anodizing process, using a range of 20 and 30 V for 1200 seconds, also varying the electrolyte temperature in the 30, 40, and 50 °C ranges. The electrolytic cell, consists of a voltage source, a platinum counter electrode, and the working electrode, the titanium sample. The electrolytic solution used in the process consists of 90% v/v ethylene glycol, 10% milli-q water, and 0.75 m/m NH_4F. Although the synthesis of TiO_2 nanotubes is still being refined, the upcoming weeks are expected to confirm the emergence of these nanostructures through the scanning of experimental conditions. This will allow the work to progress to the loading, immobilization, and drug release testing stages.

Acknowledgments:

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Environmental remediation of contaminated water and sustainable development of catalysts based on reduced graphene oxide

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Sulfanilamide is an antibiotic, resistant to biodegradation, that inhibits the formation of folic acid by a competitive mechanism, preventing crucial reactions for bacterial growth, and can be found in water bodies [1]. This substance is an emerging pollutant due to its widespread use, which allows for large-scale detection in the environment. Based on recent scientific evidence, advanced electrochemical oxidation processes (EAOPs) effectively remove various organic pollutants. Under certain operating conditions, EAOPs can almost completely mineralize pollutants present in water [2]. In this work, we propose the use of reduced graphene oxide (rGO) synthesized from cellulosic biomass and iron as catalyst for the degradation of SNM through heterogeneous electro-Fenton (EFH). The catalysts were synthesized using 1 g of cellulosic biomass and different amounts of iron-based catalyst (0,1 g a 0,3 g), where it was subjected to approximately 300°C in a muffle furnace for 30 minutes, obtaining the catalysts called rGO1, rGO2 e rGO3, (0,1g;0,2g;0,3g) respectively. An SNM solution (0,50 mmol L^{-1}, pH 5,86) was treated with the three catalysts for the EFH reaction. Carbon-PTFE with air diffusion and Pt were used as cathode and anode, respectively, in 0,05 mol L^{-1} Na_2SO_4 and 60 mA cm^{-2} current density. The catalyst concentration was set at 100 mg L^{-1}. For comparison purposes, degradation by homogeneous EF was also performed. It was possible to indicate that the increase in the proportion of Fe enabled greater removal of total organic carbon, after 240 minutes of treatment.

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Enhanced Electrochemical Ammonia Synthesis from N_2 using Metal Phosphide Nanoparticles on 3D Porous Graphene

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Ammonia is an essential chemical commodity and a candidate as a green energy vector. However, industrial manufacturing relies on the Haber-Bosch process. The conventional H-B method requires high energy density and generates millions of tons of CO_2 annually [1]. Electrochemical ammonia synthesis driven by renewable energy sources is a promising approach to replace the conventional H-B process due to operating in mild conditions. Despite the advantages, the ENRR still faces challenges such as low faradaic efficiencies, uncompetitive yield production, and slow kinetics. In this scenario of early-stage application [2], developing efficient electrocatalysts is essential for achieving favorable performances. Transition metal phosphides are interesting materials for several reduction/hydrogenation reactions. The present work reports the investigation of Fe and Co-based metal phosphides as catalysts for the electrochemical N_2 reduction to ammonia. For the nitrogen reduction reaction, nanoparticles of individual metal phosphides of Fe and Co- and FeCo-supported graphene were synthesized via a biomass-hydrogel method followed by thermal reduction. The synthesized materials were evaluated for their catalytic activity in the electrochemical reduction of N_2 in 0.1 M Na_2SO_4 at a constant potential electrolysis from -0.20 to -1.2V vs RHE. The FeCo phosphide presented a 3-fold increase in the NH_3 production rate compared to the individual metal phosphide, suggesting a synergistic effect of the Fe, Co bimetallic active sites.

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Characterization of Nanocomposite Based on Bacterial Nanocellulose Produced From a Waste, Carbon Nanotubes and Silver Nanoparticles in the Modification of Electrochemical Sensors for Determination of Emerging Contaminants

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Carbon nanotubes (CNT) possess physical-chemical characteristics that are highly advantageous for the advancement of electrochemical sensors, including excellent electrical conductivity and a large surface area. However, their low dispersion significantly impacts the electrical signal conductivity [1,2]. Bacterial nanocellulose (BNC) is a biocompatible polymer with high purity, surface area, and pores that can function as a matrix for the immobilization of nanoparticles [2], such as silver nanoparticles (AgNPs), which are also biocompatible and outstanding conductors with significant potential for sensor applications [3]. CNT, AgNPs, and BNC produced using brewery waste in a culture medium were utilized as electrode modifiers. BNC and functionalized multi-walled CNTs (MWCNT) were prepared with three different proportions of BNC (10, 20, and 30%) and AgNPs (30 and 40%) for each 20 mg of MWCNT. The modified glassy carbon electrodes (GCE) were morphologically and electrochemically characterized, and the incorporation of AgNPs onto the BNC/MWCNT composite was observed. Experiments were conducted using cyclic voltammetry and differential pulse voltammetry to determine the optimal configuration of MWCNT/BNC and MWCNT-BNC/AgNPs ratios. The configuration that exhibited the highest peak currents and reversibility of redox probes was found to be 20% BNC and 40% AgNPs. Parameters related to voltammetry and pH were also optimized, and the proposed electrode was employed to detect furosemide (FUR) in PBS 0.2 mol L^{-1} pH 5. A linear relationship between the concentration of 1.0 μmol L^{-1} and 6.0 μmol L^{-1} was established. Consequently, BNC derived from waste has proven to be a promising nanoscale biopolymer when combined with MWCNT/AgNPs for electrochemical sensors.

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Evaluation of phenazines as cathode for application in aqueous Zn ion batteries

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Zinc-ion batteries (ZIBs) have garnered attention in the scientific community, due to the well-established chemistry of zinc and its impressive theoretical energy capacity of 820 mA h g^{-1}. The abundant reserves of zinc position ZIBs as a promising candidate for environmentally friendly aqueous batteries. However, successful ZIB development hinges on understanding cathode material roles. Despite extensive research on inorganic cathodes, they are plagued by challenges including structural instability, low electrical conductivity, and restricted ion diffusion coefficients. These issues must be addressed to fully harness the potential of ZIBs.[1]

Organic materials, specifically those featuring carbonyl and imine bonds, have surfaced as potential candidates for cathodes. One such example is phenazine, which has garnered significant interest.[2] Although the organic synthesis of phenazine poses certain challenges, it's noteworthy that phenazine can also be obtained naturally from the secondary metabolites of microorganisms. This alternative method paves the way for more sustainable production routes [3], further enhancing the appeal of phenazine as a cathode material.

Therefore, our study aimed to evaluate phenazine compounds as cathodes for ZIBs to develop more efficient and sustainable energy storage devices.

Electrodes were prepared using the following mass percentages: 65% active material (phenazine), 20% active carbon (Ketjen), and 15% binder (PVDF). In addition to phenazine (PZ), other substituted compounds derived from PZ were investigated, such as phenazine-1-carboxylic acid (PCA), phenazine-1-ol (POH), phenazine-1-carboxamide (PCM), and 1-methoxyphenazine (PMO).

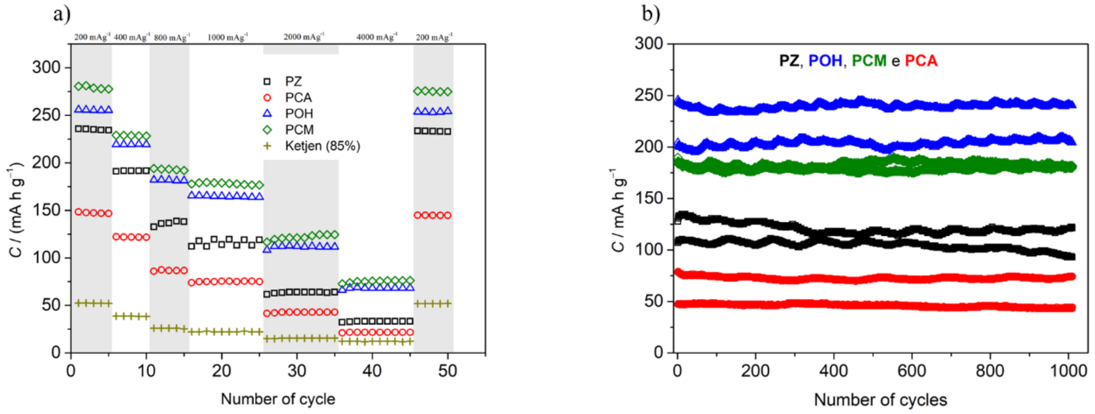
To investigate the performance of substituted phenazines, galvanostatic charge-discharge (GCD) tests were conducted within the potential range from 0.3 to 1.4 V *vs.* Zn/Zn^{2+} at distinct current densities. The experiments utilized a 2-electrode Swagelok cell with glass fiber as separator and 110 µL of $ZnSO_4$ 2M as electrolyte.

As can be seen in Figure 1a, the specific capacity decreased continuously for high current densities applied; however, the initial values were recovered when 200 mA g^{-1} were used. Remarkably, under all tested current density conditions, there was no dissolution or oxidation of the active material. Such stability behavior is unprecedented in the literature.

Lastly, to investigate the retention of the specific capacity for prolonged assays, tests were conducted at 1 A g^{-1} for 1000 cycles. As shown in Figure 1b, all tested active materials exhibited slight fluctuations during the same experiment, but the specific capacity remained constant throughout experiment. This reinforces the hypothesis that the active materials are not undergoing dissolution or degradation. Additionally, the Faradaic efficiency remained at 100% in all measurements.

In summary, it has been confirmed that phenazine and its substituted compounds are an interesting option to be used as cathode in ZIB, since high stability and specific capacity (specially POH and PCM) were obtained. In addition, these compounds can be synthesized through biological.

Figure 1 - Specific capacity using different active materials as a function of the number of cycles during GCD a) at different mass current densities and b) duplicates for prolonged tests (1000 cycles) at 1 A g^{-1}.



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Direct growth of MoS_2/Ni_3S_2 composite on nickel foam using Microwave-Assisted synthesis

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The emergence of green hydrogen has sparked significant interest in both scientific and industrial spheres, marking a pivotal milestone in the global transition towards more sustainable energy sources. Produced through electrolysis methods powered by renewable energy sources such as solar and wind, this fuel holds immense potential for mitigating carbon emissions and addressing the challenges of climate change. In this context, the study and improvement of the Hydrogen Evolution Reaction [1] are indispensable for the evolution of these technologies. This study presents a method for synthesizing the MoS_2/Ni_3S_2 composite [2] in a single step, achieving rapid growth, by using microwave radiation. We utilized Ni foam pieces (0.6 cm x 1 cm) as a substrate, initially subjected to a pre-cleaning process involving immersion in a 1 mol/L hydrochloric acid solution for 10 minutes within an ultrasonic bath to eliminate surface oxides. For microwave reaction, a solution consisting of 3 mL of ammonium tetrathiomolybdate at a concentration of 10 mg/mL was introduced into a silicon carbide reaction tube alongside the nickel foam, followed by heating to 240°C for a duration of 30 minutes in a microwave reactor (Anton Parr). X-ray diffraction analysis revealed the formation of the crystalline phase of Ni_3S_2. No molybdenum sulfide phase was observed. However, Raman analysis exhibited the E_{2g} and A_{1g} modes corresponding to the crystalline phase of MoS_2. Scanning electron microscopy (SEM) analysis revealed a significant change in the morphology of the Ni foam, displaying porous structures of nanoparticles and nanorods, likely corresponding to the phases of MoS_2 and Ni_3S_2. Linear sweep voltammetry (LSV) analysis presented an overpotential of 188mV at 10 mA/cm^2 and a good stability in a chronoamperometric experiment in a 0.5 mol/L H_2SO_4 solution.

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Machine Learning Predictions of Onset and Oxidation Potentials for Alcohols: Model Development, Experimental Validation, and Implementation.

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The onset and oxidation potentials of electrochemical reactions are pivotal in assessing catalytic energy efficiency, with applications spanning various domains, including sustainable energy generation. However, predicting these potentials presents a complex and uncharted challenge. By leveraging the capabilities of Machine Learning techniques, we initiate an innovative endeavor to construct a comprehensive pipeline that, for the first time, facilitates the creation of predictive models for the onset and oxidation peak potentials of electrochemical reactions tied to the oxidation of methanol and ethanol using existing scientific literature data. We assessed the performance of different regression models: Linear, Random Forest, and XGBoost, achieving a prediction error of 0.169 V for oxidative peak potential and 0.175 V for onset potential.

The work has three pasts, as illustrated in Figure 1. First it was developed a machine learning model, using the database that we created from the scientific articles. 312 papers were meticulously selected, considering that several of them featured multiple electrodes (different materials) or varying reaction conditions for alcohol oxidation. As a result, a comprehensive database comprising 681 entries was established. Each entry within this dataset represents a distinct electrochemical scenario associated with the oxidation of either methanol or ethanol. These distinctions encompass various factors, such as the employed working electrode, deposited materials, pH, electrolytes, or the analytes under assessment.

Next, it was explained an experimental validation that compares the predicted outcomes with the actual results of methanol and ethanol oxidation experiments conducted in a chemical laboratory, utilizing Platinum, Gold and RuO_2/FTO electrodes. Finally, we developed an app using Streamlit plataform to make the model accessible to the community. Thus, our aim was to highlight the use of ML in the prediction of electrochemical data with a focus on curtailing resource utilization and decreasing the time spent on the experimental step to develop more efficient materials.

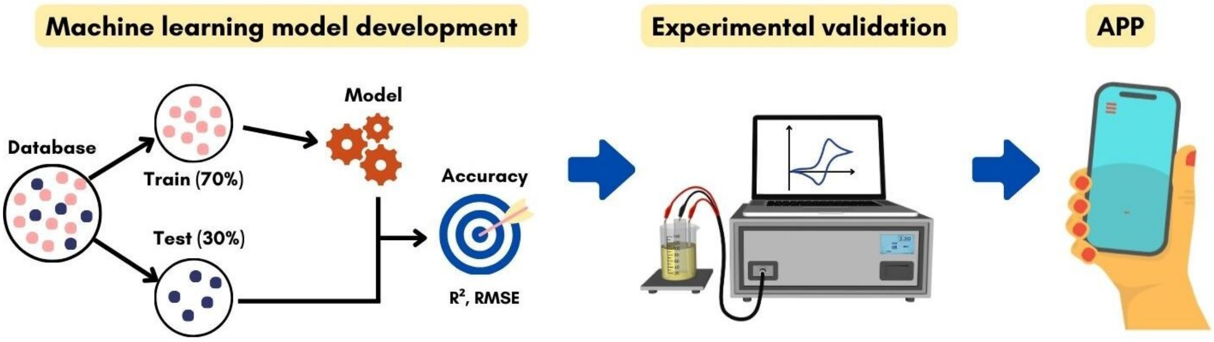

Figure 1: The three parts of the work: Machine learning model development; Experimental

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Energy-saving electrochemical green hydrogen production coupled with lignin biomass upgrading with NiMoP-modified electrode

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Electrocatalytic oxidation (EO) of lignin, a rich renewable aromatic resource that can provide high-value compounds, is a potential process for converting this biomass feedstock into high-value fuels and chemicals under mild operating conditions with simultaneous green hydrogen production [1,2]. In this study, NiMoP coating was electroplated on stainless steel for anodic electrotransforming lignin (500, 1000, and 1500 mg L^{-1} of lignin concentrations) by applying 40, 70, and 100 mA cm^{-2} into carboxylic acids and green hydrogen production at the cathode, using an anion-exchange membrane water electrolyzer . This material was characterized by SEM/EDX, cyclic voltammetry, and polarization curves analysis. NiMoP films contained many ramified cracks on its substrate surface because of the internal stress generated by molybdenum co-deposition that alters the crystallographic structure of nickel. Voltammetric curves reveal that the incorporation of Mo into the NiP matrix decreases the onset potential of the hydrogen evolution reaction. From the point of view of the EO of lignin, formic and acetic acids were generated. Carboxylic acids concentrations remain constant after 30 minutes of electrolysis for all the current densities and lignin concentrations investigated, achieving 4.82 and 43.76 mg L^{-1} for formic and acetic acids, respectively, at 70 mA cm^{-2} with 1500 mg L^{-1} of lignin after 240 minutes of electrolysis, as the best performances.

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Conductive rPETg filament for electrochemical detection of uric acid

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Recent literature on sensors for healthcare using additive manufacturing electrochemistry predominantly uses conductive PLA, which is not ideal for widespread industrial application [1]. In contrast, poly (ethylene terephthalate glycol) (PETg) stands out for its excellent thermal, mechanical and chemical properties for additive manufacturing. In this study, we present an innovative electrically conductive filament made of recycled PETg (rPETg) enriched with carbon black, multi-walled carbon nanotubes and graphene nanoplatelets as conductive fillers. We systematically characterize the fabrication process, physicochemical properties and electrochemical performance. We optimize the activation of electrodes made of this material after printing and achieve a significantly improved electrochemical performance with a k0 value of 1.03×10^{-3} cm s^{-1} compared to non-activated electrodes. The Relative Standard Deviation (RSD) was calculated for multiple measurements using the same electrode and different electrodes (n=3) with $[Fe(CN)_6]^{-3/-4}$, resulting in RSD values of 1.80% and 5.25%, respectively. With regard to the critical need for sterilization in biomedical applications, we show that treatment with UV light treatment makes these electrodes reliable and distinguishes them positively from their PLA counterparts [2]. When tested with synthetic urine, differential pulse voltammetry and chronoamperometry techniques show impressive sensitivity and low limits of detection (LOD) for uric acid (25.7 µA µmol L^{-1}, 0.27 µmol L^{-1}). In addition, we evaluate the reusability of the electrodes for uric acid detection and confirm their efficacy over ten cycles before detecting a significant drop in performance. This research highlights the potential of the new conductive rPETg material to advance additively manufactured sensors in healthcare. Its sterilizability, reusability, minimal solution intrusion and ability to address the issues of cost and plastic waste in healthcare highlight its promising role in the future development of sensors [3].

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Green electrochemical sensors using CO_2 laser pyrolysis on green leaves substrates for paracetamol detection

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The use of green leaves as a substrate for the fabrication of electrochemical sensors is an innovative approach that provides a sustainable and environmentally friendly alternative to conventional sensor substrates. In this context, the CO_2 laser represents an attractive area of research that has the potential to revolutionize the way sensors are manufactured. CO_2 laser pyrolysis enables the fabrication of electrodes on various substrates, contributing to the development of high-performance, environmentally friendly, and economical sensors. The green leaf sensor was optimized and standardized according to the adjustment possibilities of the laser cutting printer, such as laser distance, potential, and burning speed. In this sense, the leaves used for the fabrication of the electrochemical sensors stem from *S. macrophylla* and *P. americana*, tree species widely and easily found in Brazil [1,2]. The system was used for the detection of paracetamol (PAR), an analgesic and antipyretic medication, being one of the most prescribed for the relief of fever, headaches and pain in general [3]. A study was conducted using CV in the absence and presence of 1.0 mmol L^{-1} PAR in 0.2 mol L^{-1} PB buffer (pH 7.0) and an analytical curve was constructed in the concentration range of 5.0 to 100.0 µmol L^{-1} using SWV. The obtained SWV voltammograms in the presence of PAR presented a peak around 0.33 V, and the analytical curve exhibited excellent linearity ($R^2 = 0.999$), in the explored concentration range. The calculated values for the LOD and LOQ were 0.23 and 0.76 µmol L^{-1}, respectively. The ratio of the cathodic and anodic peak currents was 0.97, and the ΔEp was 0.68 V, indicating an irreversible behavior. The electrode had an electroactive area of 0.17 cm^2 and a geometric area of 0.23 cm^2. The proposed sensor was applied for PAR electrochemical determination as a proof of concept and values of 10.8 ± 0.7, 24.3 ± 0.9, and 72.4 ± 1.8 µmol L^{-1} PAR were obtained for the 10, 25, and 75 µmol L^{-1} solutions, demonstrating the electrode's ability to analyze pharmaceutical samples containing PAR. Therefore, the work proposes a new system prepared with dehydrated green leaves substrate using CO_2 laser pyrolysis for the detection of paracetamol.

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Eletrodo impresso descartável a base de goma laca chinesa e carbon black para detecção de melatonina

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Os eletrodos serigrafados (SPE – do inglês screen printed electrode) se destacam como uma tecnologia inovadora no campo dos sensores eletroquímicos, oferecendo diversos benefícios como, facilidade de produção, fabricação em larga escala e possibilidade de replicação[1] . Além disso, os SPEs viabilizam a fabricação de grandes quantidades de eletrodos de carbono, de relativo baixo custo e descartáveis. A escolha de diferentes materiais para a confecção de eletrodos permite a personalização desses sensores, adaptando-os a diversas necessidades especificas, sendo isto, possível pois são fabricados a partir de uma tinta condutora [2]. Devido a essas vantagens, eles estão cada vez mais presentes em variadas áreas, como medicina, farmácia entre outros[3]. Em vista disso, o sensor apresentado é preparado à base de uma tinta composta por carbon black, goma laca chinesa e acetona, para produzir um dispositivo eletroquímico. Para mostrar o potencial analítico do SPE proposto ele foi utilizado para determinação de melatonina, um hormônio produzido pelo corpo onde sua principal função é regular o ciclo do sono [4]. A curva analítica, foi obtida a partir da voltametria de onda quadrada, foi construída na faixa de 1 a 100 µmol L^{-1} com limite de detecção de 0,1 µmol L^{-1}. Além disso, análises de recuperação foram realizados na amostra de urina sintética com os valores obtidos entre 86,7% e 110%. A partir dos resultados obtidos podemos concluir que a tinta condutora desenvolvida apresentou característica desejáveis para a confecção dos eletrodos impressos e o dispositivo analítico apresentou resultados satisfatórios para detecção de melatonina.

Palavras-Chave: eletrodo impresso descartável, melatonina, tinta condutora, goma laca chinesa e carbon black

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Carbon functionalization: A strategy to improve Pd catalysts for fuel cells

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The high cost and susceptibility to carbon monoxide (CO) adsorption limit the commercial viability and efficiency of Pd nanoparticles in fuel cell anodes [1]. The choice of support material for Pd nanoparticles is essential for reducing costs and enhancing their catalytic performance [2]. Hence, we investigated the impact of carbon support functionalization on the catalytic activity of Pd nanoparticles for ethanol, glycerol, and CO oxidation in alkaline media. Vulcan XC-72 carbon was functionalized using KOH and HNO_3 treatments, and Pd nanoparticles were deposited via chemical reduction with Sodium Borohydride. FT-IR analysis indicated increased hydrophilicity in functionalized Vulcan carbon. The presence of Pd was confirmed in all catalysts by XRD, with C_{KOH} and C_{HNO_3} supports yielding smaller crystallite sizes due to higher BET surface areas (C_{KOH}: 41.94 m^2g^{-1}, C_{HNO_3}: 37.21 m^2g^{-1}) compared to non-functionalized carbon (25,03 m^2g^{-1}). Pd/C_{KOH} and Pd/C_{HNO_3} catalysts displayed lower CO, ethanol, and glycerol oxidation onset potentials than Pd/C. The onset potential for CO oxidation decreased by 70 mV and 40 mV for Pd/C_{KOH} and Pd/C_{HNO_3}. For ethanol oxidation, the reductions were 40 mV and 80 mV, while both catalysts exhibited 40 mV lower onset potentials for glycerol oxidation compared to Pd/C. Pd/C_{KOH} showed the highest specific activities for ethanol and glycerol oxidation, 1.84 and 2.68 times greater than Pd/C. Our results suggest that support functionalization enhances the activity of Pd nanoparticles by reducing crystallite sizes and providing oxygenate species, thereby facilitating the oxidation of adsorbed alcohol and intermediate molecules.

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Desenvolvimento de sensor eletroquímico por impressão 3D para determinar ácido gálico

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A tecnologia de impressão tridimensional está sendo utilizada para produção de sensores eletroquímicos para detecção de diversos analitos para auxilio das áreas de medicina, farmacêutica, pesticidas e matriz alimentares [1]. Desta forma, essa alta aplicabilidade dos sensores eletroquímicos mostram-se aliados à sociedade e suas demandas. Os dispositivos eletroquímicos destacam-se por relativo baixo custo, boa condutividade, produção em alta escala e uso de materiais sustentáveis em sua produção. Junto a isso, há possibilidade de personalização de sensores a partir de diferentes composições de materiais [2]. O ácido gálico é encontrado em folhas de chá, frutas, sementes de frutas e bebidas e possuem benefícios como atividades anti-inflamatórias, anti-carcinógeno, antimutagênica e proteção hepatológica [3] Neste sentido, este trabalho foi realizado a partir da produção de filamentos a base do negro de fumo (CB, do inglês *Carbon Black*), polipropileno (PP), ácido polilático (PLA) e óleo de coco. O filamento desenvolvido foi então impresso em 3D e os parâmetros de impressão foram ajustados conforme necessidade. Os eletrodos impressos foram tratados eletroquímicamente, com hidróxido de sódio 0,1 mol L^{-1} e polidos com lixa para expor o material condutor, o negro de fumo, retirando o excesso de polímero. Foram utilizadas duas proporções de polipropileno e ácido polilático a fim de otimizar o filamento para obtenção de uma melhor resposta ao analito de interesse. Posteriormente foram feitas voltametrias cíclicas obtidas para o ferrocenometanol 1,0 mmol L^{-1} em KCl 0,1 mol L^{-1} com velocidade de varredura 50 mV s^{-1}. Foi realizado o estudo de área eletroativa com voltamogramas cíclicos obtidos para ferrocenometanol 1,0 mmol L^{-1} em KCl 0,1 mol L^{-1} em diferentes velocidades de varredura (10 a 100 mV s^{-1}) que acusaram corrente de pico anódica em 0,3V *vs.* carbono. Sendo assim, o trabalho desenvolvido mostra a produção de um sensor eletroquímico 3D usando filamento *lab-made* de CB/PP/PLA/Óleo de coco para futura detecção de ácido gálico.

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Modified Carbon Paste Electrode for Theobromine Detection

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This work [1] presented consists of the construction and application of an electrochemical sensor using a carbon paste electrode (CPE) chemically modified with a Schiff base complex $[V^{II}O(salen)]$ for the determination and quantification of theobromine in chocolate powder samples. The main objective is to provide a simple, economical, and environmentally sustainable method for agile and efficient analysis in the food and pharmaceutical industries.

An AFM evaluation of the modified working electrode revealed notable surface modifications, including increased thickness and roughness that encourage analyte contact. The amperometric sensitivity for CV was 2.91 (±0.10) μmol L^{-1}/μA for the anodic peak and 18.66 (±0.44) μmol L^{-1}/μA for the cathodic peak on the CPE@$[V^{II}O(salen)]$ electrode, with LODs of 0.22 (±0.01) μmol L^{-1} and 0.38 (±0.01) μmol L^{-1}, respectively. For SWV, the amperometric sensitivity values were equal to 5.38 (±0.17) μA/μmol L^{-1} and 2.60 (±0.05) μmol L^{-1}/μA, with LODs of 0.40 (±0.01) μmol L^{-1} and 0.41 (±0.01) μmol L^{-1} for the anodic and cathodic peaks, respectively.

An analysis of the chocolate powder samples by HPLC-DAD confirmed the presence of theobromine, with a sensitivity comparable to electrochemical analysis, whose linear range of quantification of the analyte varied from 4.93 (±0.02) μmol L^{-1} to 14.94 (±0.02) μmol L^{-1}. The research presented highlights the efficacy of CPE modified with $[V^{II}O(salen)]$ for the sensitive and selective electrochemical determination of theobromine. The electrode offers significant advantages over traditional methods of analysis, including a shorter analysis time, a lower relative cost, and environmental sustainability. Ultimately, its successful application in chocolate powder samples demonstrates its suitability for industrial quality control.

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Application of TiO_2 nanotubes modified with copper MOFs in photocatalytic degradation of ciprofloxacin in water

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Metal-organic frameworks (MOFs) are compounds made of porous materials in which a coordination network between a metal cluster (or ion) and organic ligands has potentially empty cavities. MOFs have specific characteristics such as permanent microporosity, high crystallinity, low density, high surface area values, and good physical and chemical stability. The stability of the MOFs in water is a current challenge to be applied in the aqueous system. In addition to these characteristics, it is possible to use different strategies for synthesizing and functionalizing this type of material to apply it in various areas [1]**.** Among the techniques used to produce MOFs, solvothermal, hydrothermal, sonochemical, electrochemical and microwave-assisted synthesis are the most used. This work aims to synthesize copper MOFs (Cu-MOFs) stable in an aqueous solution via the solvothermal method[2] to apply in photocatalytic processes. To reach this objective, two parameters of the Cu-MOF syntheses were investigated: organic ligands (terephthalic and trimesic acids) and solvents (ethanol, water and dimethylformamide (DMF)). Subsequently, the more stable Cu-MOF was used to modify TiO_2 nanotube oxides for the degradation of ciprofloxacin using a photocatalytic process. To characterize the Cu-MOFs the following techniques were used: powder X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FT-IR), Thermogravimetric Analysis (TG/DTA), specific surface area calculation (BET) and water stability test. The synthesized MOFs presented morphological differences such as planar and octahedral structures. Powder X-ray diffraction analyses identified the position of the peak characteristics of Cu-MOFs prepared. For the spectra analysis, the bands indicated the formation of MOFs through copper linked to the oxygens of the organic ligands. Thermogravimetric curves indicated the stability of the Cu-MOFs at approximately

250ºC and 300ºC. High surface area values were obtained for all Cu-MOFs synthesized. The MOF Cu-BTC was chosen for application in photocatalysis due to the highest yield, largest surface area, best synthesis conditions, high stability in water, and well-defined structure. These properties made the MOF suitable for modification of TiO_2 nanotubes and their application in photoelectrocatalysis. This modified material decreased the degradation time of the ciprofloxacin in water by up to 50%. This study shows that conjugated material applied in photoelectrocatalysis is an excellent alternative for treating emerging contaminants, mainly pharmaceuticals, in the aquatic environment.

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Evaluation of electrodeposited Ni-S films as catalysts for HER: a chemometric approach

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In the past years, the search for new catalysts for the hydrogen evolution reaction (HER) has been a great challenge to minimize the energy demand of the electrolysis process and to achieve more stable materials that can efficiently produce hydrogen for long periods of time [1]. In this context, transition metal sulfides, particularly Ni-S for alkaline electrolysis, are being studied as promising materials due to their versatility - they can be synthesized through various routes, can form different structures, have high electrical conductivity, and the Ni-S bonds present favorable thermodynamics for hydrogen reduction [2]. In this work, the electrodeposition method was chosen to produce Ni-S electrocatalysts, following a Plackett-Burman experimental design to evaluate which parameters affect the overpotential for HER and other electrochemical properties of the electrodes in 1 M NaOH solution. The variables used in the experimental design included the concentrations of Ni and S precursors, anionic species in solution, substrate, pH, current density, and deposition temperature. The best result obtained from the saturated design was an overpotential of 189 mV at 10 mA cm^{-2}, a Tafel slope of 93 mV dec^{-1}, a charge transfer resistance of 6 Ω cm^{2} at an overpotential of 200 mV, and an electrochemically active surface area of 200 cm^{2}. The copper substrate, the highest Ni and S precursor concentrations, the alkaline pH, and the presence of $SO4^{2-}$ anions in solution were responsible for 85% of the contribution to the improved overpotential response.

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Replacing oxygen evolution reaction in water splitting process by electrochemical energy-efficient production of formic acid with co-generation of green hydrogen

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Glycerol is one of the main biomass-derived platform molecules to produce high-value species such as glyceraldehyde, dihydroxyacetone, glyceric acid, tartronic acid, glycolic acid, oxalic acid and formic acid [1, 2]. Formic acid is one of the proposed products for the efficient and effective use of glycerol, as it can be used as a fuel for fuel cells [3]. Therefore, this study aims to electroconvert glycerol into formic acid using DSA anode and promote the simultaneous production of green hydrogen. Electrochemical oxidation of glycerol (0.1 mol L^{-1}) was carried out at three different current densities, 30, 60 and 90 mA cm^{-2} with DSA anode (20.8 cm^2) for 360 minutes, using 0.5 mol L^{-1} Na_2SO_4 as supporting electrolyte, at room temperature. Under these conditions and higher current density, 80.4 mg L^{-1} of formic acid was electroproduced. Different concentrations of the supporting electrolyte (0.1, 0.5 and 1.0 mol L^{-1} of Na_2SO_4) were also analyzed to evaluate the influence on the glycerol transformation process by applying 90 mA cm^{-2}, achieving the best performance of production of formic acid (189 mg L^{-1}) when 0.1 mol L^{-1} of Na_2SO_4 was used. Meanwhile, green H_2 production in the experiment produced 7.6 L of dry gas, which can be used as an energy source in various processes. With the results presented, it is clear that it is possible to convert this residue into products of greater economic value.

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Solução tamponante a base de etanol CMR.

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RESUMO

Palavras-chave: Etanol, Ejl, pH

Introdução: A medição de pH é um método criado para medir o potencial do íon H+ em soluções aquosas, o etanol é considerado um solvente orgânico por ter uma quantidade muito pequena de água (6,2 - 7,5%(m/m), por esse motivo é necessário o desenvolvimento de uma Solução Tamponante para que haja uma calibração do eletrodo de uma maneira que o potencial de junção elétrica seja mínimo.

O etanol é o biocombustível mais utilizado, devido ao seu baixo custo, alto rendimento e baixo potencial poluidor comparado aos demais combustíveis fósseis. O pH é o parâmetro da qualidade do etanol, da refinaria até o consumidor final. Este parâmetro é dado e exigido pelas normas, Brasileira **NBR10891 DE 11/2017** ([1]) e internacionalmente **ASTM-D6423:2008-EAC** ([2]). A normatização se dá pelo fato que a utilização de etanol hidratado combustível (**EHC**) fora das especificações pode favorecer à corrosão do motor e de suas peças. O pH do etanol é medido utilizando o sistema de medição potenciométrico (potenciômetro+eletrodo de vidro combinado (**EVC**)), de modo simplista, o eletrodo irá medir a diferença de potencial gerada entre as interfaces interna e externa ao eletrodo de membrana de vidro que contém uma solução aquosa, saturada, em cloreto de potássio **(KCl)**, no eletrodo de referência externo, oriunda da diferença nas atividades da atividade dos íons H^+, interna e externa ao bulbo de vidro. O potenciômetro converte a diferença de potencial gerada nas interfaces em valor de pH. No entanto, o **EVC** foi desenvolvido para a medição do pH em água para $I \leq 0{,}1\ mol\ L^{-1}$.

A partir das considerações anteriores, os eletrodos empregados para medir o pH em etanol fornecem medidas que refletem, exatamente, o pH do etanol? Para atender a indagação foi preparado um **EVC** com nova arquitetura contendo etanol em todos os compartimentos. No entanto esses sistemas de medição precisam, ainda, ser calibrados com **MRC** aquoso, o que faz presente o potencial de junção líquida **(Ejl)**, nos diafragmas. Esse potencial pode ocorrer em situações em que as espécies iônicas presentes nas soluções possuem diferentes valores de mobilidade iônica, ou, quando se trata dos mesmos eletrólitos, porém com concentrações diferentes. Essa diferença gera a separação de cargas na interface, gerando assim, o potencial de junção líquida **(Ejl)**. O desenvolvimento deste potencial, que pode assumir valor positivo ou negativo, é somado ao potencial global da célula, comprometendo os resultados da medição, causando um falseamento nos resultados. O trabalho já está em estado avançado, utilizando a solução para a calibração do eletrodo e efetuando a medição em amostras comerciais demonstrando ótimos resultados referentes a calibração do eletrodo, o método utilizado neste trabalho possui algumas exigências, como por exemplo manter a temperatura constante e sem interferência de ondas de rádio, pois podem interferir nos resultados. Concordante com a literatura (**GALSTER, 1991**) [3] o potencial de junção líquida do sistema sob a leitura em sulução etanolica, possui um erro de leitura de pH ±0,43 (erro qual já era esperado). Calibrando o eletrodo em 6,75 (valor estudado) com a sulução Tamponante em desenvolvimento, temos uma diminuição muito consideravel do erro chegando a uma variação de ±0,015 do pH.

Assim, a objetiva deste trabalho é a preparação de um candidato a material de referência (**CMR**), a base LiAc/HAc em **EHC** (acetato de lítio e ácido acético em etanol combustivel), para a calibração do sistema de medição potenciométrico, fazendo com que haja uma taxa de quase zero no erro das análises.

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Characterization and Electrochemical Evaluation of Polyaniline-Modified Screen-Printed Electrodes in Microbial Fuel Cells

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Screen-printed electrodes (SPE) modified with polyaniline (PAni), reduced graphene oxide (rGO), and their hybrid (PAni-rGO) were developed by serigraphy[1], characterized, and evaluated as anodes in H-type microbial fuel cells with *Saccharomyces cerevisiae*[2]; SPE/PAni-rGO was used as the cathode. The materials were characterized using scanning electron microscopy (SEM), Raman spectroscopy, attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR), and X-ray diffraction (XRD). The SPEs were fabricated with conductive ink made of graphite and alkyd resin, incorporating 2.5% by mass of the modifying materials. Electrochemical evaluations were conducted using cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) in a redox system. The bioelectrochemical system (BES) comprised an H-type biofuel cell (BFC) with Britton-Robinson buffer (pH = 7.0), *Saccharomyces cerevisiae*, glucose, and methylene blue in the anode compartment, and potassium ferricyanide in the cathode compartment, operated at room temperature. This system was analyzed using EIS, Linear Sweep Voltammetry (LSV), and polarization curves. Characterization results confirmed the successful chemical polymerization of PAni, the production of rGO via the modified Hummers method, and the synthesis of the hybrid material. Electrochemical results showed that the modification of the SPE with PAni/rGO maintained the voltammetric profile of PAni, increased current intensity, and, based on complex plane diagrams and equivalent circuit fittings, reduced charge transfer resistance for SPE/PAni-rGO compared to other electrodes. Both SPE/PAni and SPE/PAni-rGO enhanced current intensity in polarization curves compared to unmodified SPE, with a further reduction in charge transfer resistance (EIE) after bioelectricity generation.

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Comparação de sensores eletroquímicos descartáveis fabricados com uso de diferentes tintas condutoras obtidas comercialmente para a determinação de L-dopa

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No âmbito científico e industrial, novas maneiras de produzirem sensores eletroquímicos cada vez mais eficientes, de baixo custo e confiáveis, vêm sendo pesquisadas devido à grande versatilidade que diferentes materiais podem fornecer quando aplicados em determinados métodos [1]. Um exemplo é o uso de materiais condutores para produção de tintas condutoras utilizadas em diversos campos, tal como na impressão de sensores descartáveis por serigrafia (do inglês, *Screen-Printed Electrodes,*SPE), visto que apresentam grande potencialidade para produzir dispositivos completos e otimizados [1, 2]. Diante das vantagens da simplicidade de produção e também das análises eletroanalíticas, a demanda, em áreas como a da saúde para análises clínicas; vem aumentando significativamente, fazendo com que seja atrativo para as empresas os produzirem em escala industrial. Nesse sentido, o presente trabalho apresenta uma comparação entre sensores descartáveis fabricados com duas tintas condutoras distintas, produzidas comercialmente, denominadas C1 e C2, para a determinação de L-dopa em medicamentos, uma vez que é um fármaco fundamental no tratamento da doença de Parkinson. Os SPEs foram caracterizados por voltametria cíclica, os quais foram empregados com duas sondas redox, o ferrocenometanol e o hexaminrrutênio. Além disso, o analito também foi avaliado pela voltametria de onda quadrada (SWV) e voltametria de pulso diferencial (DPV). A superfície dos sensores foi caracterizada por microscopia eletrônica de varredura (MEV). Valores de pico anódico foram observados em 0,45 V *vs*. Ag, com a corrente de pico em 1,5 µA (eletrodo C1), enquanto que na tinta C2 o pico foi observado em 0,25 V *vs*. Ag, com a corrente de pico em 2,0 µA, valores próximos aos relatados na literatura [2]. Diante desta comparação, conclui-se que o eletrodo C2 apresenta uma melhor performance, pois seu pico de oxidação e potenciais são maiores e mais próximos da literatura. Estudos de otimização foram realizados com o sensor C2, o qual será utilizado para próximas etapas. Deste modo, é possível inferir que os sensores propostos são boas alternativas para a detecção do analito em questão, os quais são produzidos de forma fácil, rápida e simples.

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Insights into *In Situ* electrogeneration of H_2O_2 in urine matrix using gas diffusion electrodes

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The quest for new technologies and catalytic materials capable of producing chemical oxidants has been a topic of considerable interest. Among these, the *In Situ* production of hydrogen peroxide (H_2O_2) offers significant advantages, particularly in decentralized treatments of complex matrices, by reducing constraints related to storage, transportation, and management of this reagent. H_2O_2 is a powerful and versatile chemical reagent that can act both as a reducer and an oxidizer. The *In Situ* generation of this oxidant can be achieved through the Oxygen Reduction Reaction (ORR) involving the transfer of 2 electrons [1]. Carbon materials have prominently featured due to their ability to selectively promote reactions. A notable example is the carbon-based Printex-L6 matrix, distinguished by its large surface area and functional groups acting as active sites [2,3]. These characteristics impart a hydrophobic nature to the material, making it a promising choice for various applications. In this study, gas diffusion electrodes (GDEs) were synthesized using Printex-L6 carbon (P-L6) for in situ generation of H_2O_2 aimed at enabling the production of this oxidant for subsequent use in degrading organic components of synthetic urine, specifically urea, uric acid, and creatinine. Electrogeneration of H_2O_2 was evaluated under different current densities (25, 50, and 100 mA cm^{-2}) in the inorganic salts present in synthetic urine and compared with electrogeneration in Na_2SO_4 at the same ionic strength. Experiments were conducted in a glass electrochemical cell equipped with a Ag/AgCl reference electrode, a dimensionally stable anode used as a counter electrode, and the GDE prepared as the working electrode positioned at the cell base. O_2 flow was fixed at 0.05 L min^{-1} and directly injected into the GDE. Quantification of H_2O_2 was performed by UV-Vis absorption spectrophotometry ($\lambda = 350$ nm) using ammonium molybdate. All experiments were performed in duplicate over 120 min, under mechanical stirring and constant temperature of 20 °C. Results revealed that, overall, the highest H_2O_2 generation occurred at a current density of 100 mA cm^{-2}, reaching a maximum concentration of 1253 mg L^{-1} in Na_2SO_4 medium. However, under the same conditions, H_2O_2 generation in salts from synthetic urine was 94.314 mg L^{-1}.

Comparing this electrogeneration among the supporting electrolytes, approximately 75% of H_2O_2 consumption in urine matrix salts can be attributed to the compositional complexity of this matrix. Besides Na_2SO_4, the synthetic urine composition included NaCl, Na_2HPO_4, and KCl, which significantly interfered with H_2O_2 generation and stability. Chloride ions (Cl^-) may catalyze H_2O_2 decomposition, leading to the formation of chlorine gas (Cl_2) and water; sulfate ions (SO_4^{2-}) may react with H_2O_2 to form persulfate (HSO_5^-); and phosphate ions (HPO_4^{2-}) may catalyze H_2O_2 decomposition, resulting in the formation of water and oxygen. These interactions can reduce H_2O_2 concentration in the solution. Moreover, the simultaneous presence of various ion types may result in secondary reactions that also affect H_2O_2 stability.

Thus, these results indicate that in situ generation of H_2O_2 in complex matrices such as synthetic urine is significantly influenced by the presence of inorganic components. Despite the high efficiency observed in Na_2SO_4, the presence of NaCl, Na_2HPO_4, and KCl in synthetic urine drastically reduces H_2O_2 concentration due to decomposition reactions. This study opens new frontiers for optimizing H_2O_2 generation systems, underscoring the importance of developing materials and experimental conditions that can mitigate the influence of inorganic interferents in complex matrices.

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Study of caffeine degradation by UVC/electrogenerated H_2O_2 based process using GDE composed of Printex L6 carbon

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Caffeine (CAF) is an indicator of short-term, untreated anthropogenic inputs. Overall, the presence, behavior, and sources of organic micropollutants in the urban water cycle is indicators of anthropogenic impacts. Processes, such as ozone-based, are alternatives due to their ability to treat organic compounds in aqueous media for CAF degradation [3], but the high toxicity of the by-products formed limits their application in real treatments systems. In this sense, the use of electrogenerated hydrogen peroxide (H_2O_2) as an oxidant in indirect electrochemical processes stand out promising as an alternative to high the degradation and mineralization of compounds as CAF. Considering the H_2O_2 production by electrochemical method, an excellent system to it, , are process that using gas diffusion electrode (GDE). In this case, an advantage of the GDE system is the absence of a catalyst that promotes the generation of an exact quantity of oxidant species. It is also easy to access and competitive because of the low costs. In this work, the effect of UVC/H_2O_2 in differents conditions as pH and currently density (*j*) was investigated for the CAF degradation and mineralization. The influence of the *j* and pH was analyzing on the UVC/H_2O_2 process. The optimal conditions are being doing (CAF concentration and degradation/mineralization of several matrices of water). On the other hand, in all measures the total (100%) elimination of the CAF was reached in the 20 min of treatment and TOC removal, mineralization with 60%, after 90 min of electrolysis. Overall, the fabricated Printex L6 carbon-based reach high concentrations of H_2O_2, favored by the increase pH as well as the UVC/H_2O_2 process allowed the total degradation and 60% mineralization in short period time. Finally, pos-treatment the samples will be evaluated by *Lactuca sativa L* method to show the absence or ausence of toxicity of the by-products

formed and exhaustive CAF oxidation/mineralization during 5h will be done on the study.

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Avaliação do desempenho eletrocatalítico do $La(OH)_3$ na seletividade para H_2O_2 via reação de redução de oxigênio

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O peróxido de hidrogênio é um produto químico de grande importância global, mas seu principal método de produção, o processo de antraquinona, enfrenta desafios logísticos, ambientais e de segurança. A síntese eletrocatalítica através da redução de oxigênio molecular é uma rota promissora para a produção de H_2O_2. No entanto, essa redução é cineticamente difícil, exigindo eletrocatalisadores estáveis com alta atividade e seletividade para a reação de transferência de 2 elétrons. Neste estudo, sintetizamos e avaliamos a seletividade do $La(OH)_3$ para a produção de H_2O_2 via RRO. A síntese das nanopartículas de hidróxido de lantânio ($La(OH)_3$) foi realizada a partir do sal $La(NO_3)_3 \cdot 6H_2O$, utilizando uma solução de sal de metal em ácido cítrico (AC) na proporção 1:50, aquecida a aproximadamente 80°C com agitação constante. Etilenoglicol foi adicionado gradualmente até atingir a proporção 1:5:40 (M:AC:EG). A mistura foi aquecida para remover o excesso de solvente, formando uma resina que foi tratada termicamente a 400°C por 4 horas em atmosfera de Ar ou N_2, com uma taxa de aquecimento de 5°C/min. Após a pré-calcinação, o material foi moído e posteriormente calcinado a 800°C por 4 horas em Ar ou N_2, com uma taxa de aquecimento de 10°C/min, para produzir os hidróxidos de lantânio. Microscopia eletrônica de varredura e difração de raios X (XRD) foram usadas para analisar a estrutura dos materiais após as diferentes condições térmicas. Os resultados mostraram uma mudança estrutural no catalisador com a variação da temperatura. O XRD das amostras produzidas a 400°C não apresentou picos significativos, enquanto os materiais produzidos a 800°C, tanto em ar quanto em N_2, apresentaram picos bem definidos. A caracterização eletroquímica das nanopartículas de $La(OH)_3$ sintetizadas revelou diferentes comportamentos em relação ao substrato de carbono vítreo e às condições de temperatura e atmosfera de calcinação. As amostras calcinadas a 400°C e 800°C mostraram variabilidade na densidade de corrente com a mudança de potencial, destacando a influência da temperatura de calcinação e da atmosfera sobre as propriedades catalíticas do material. A seletividade para a produção de H_2O_2 também foi influenciada pelas condições de síntese, com os catalisadores produzidos a 800°C alcançando uma seletividade média superior a 90% na faixa de potencial estudada. Os resultados indicam que a síntese de $La(OH)_3$ sob diferentes condições térmicas e atmosféricas resulta em materiais com características eletrocatalíticas distintas. A otimização desses parâmetros é essencial para o desenvolvimento de catalisadores eficientes para a produção eletroquímica de H_2O_2 via RRO, oferecendo uma alternativa viável para superar as limitações do método tradicional, com maior segurança e eficiência no processo industrial.

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Integrated sensor system for monitoring electrochemical hydrogen generation

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Hydrogen emerges as a promising renewable energy source due to its ability to be produced through methods that do not emit greenhouse gases. The electrolysis of water, using energy from renewable sources, allows the production of hydrogen without carbon emissions, making it a clean and sustainable energy vector, green hydrogen (H2g) [1]. This process is fundamental for decarbonizing industrial and transport sectors, which are large energy consumers and contribute significantly to global emissions [2]. However, accurately measuring the hydrogen generated is a considerable technical challenge. Traditional methods, such as volume measurement, are notoriously inaccurate due to the influence of environmental variables, while gas chromatography, despite its accuracy, is prohibitive in terms of cost and accessibility. This work highlights the importance of technological innovations in overcoming barriers to adopting renewable energies. Using Arduino as a prototyping platform and sensor modules for hydrogen, voltage, current, relative humidity, and temperature. These sensors were then coupled to a variable stabilized voltage source and a two-electrode electrochemical cell. Current and voltage applied to the system were simultaneously measured while the quantity of hydrogen generated, and its quality (water contamination, for example) were recorded in real-time. Finally, they were compared with the gas chromatograph to validate the results obtained. This proves to be sensitive and enables low-cost real-time monitoring coupled with different systems, quantifying the amount of hydrogen produced by calculating the faradaic efficiency in real-time.

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Study of the fulvic acid interference in the electro-oxidation of tetracicline

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Pharmaceuticals were a central role in the pollution of water bodies and can interact with humic substances present in rivers, forming stable complexes that are difficult to degrade [1]. This study aimed at the electrochemical degradation of tetracycline (TEC) in the presence of fulvic acid (FA). An anodic electro-oxidation was carried out using a filter press cell, with a flow system, and a commercial mixed metal oxide (MMO) anode with an area of 14.81 cm^2. The optimized conditions were: current density 40 mA cm^{-2}, pH 3.0, and time 240 min. The degradation products were analyzed by high-performance liquid chromatography (HPLC) and UV-Vis spectrophotometry. It was observed that the removal of TEC was 97% in 15 min, while the removal of FA was about 25% in 60 min. For the combined TEC and FA (TECFA), the removal was 54% in 60 min, as shown in Fig. 1a. After 60 min of TECFA degradation, an increase in the HPLC signal with a band shift was noted. This behavior was also observed in the UV-Vis for the 357 nm band, where the concentration decay was only until 60 min, and after this time, a new band at 290 nm, characteristic of complex formation, was noted, as shown in Fig. 1b. Thus, it can be concluded that the degradation of tetracycline in the presence of fulvic acid is hindered but is a feasible process with good results.

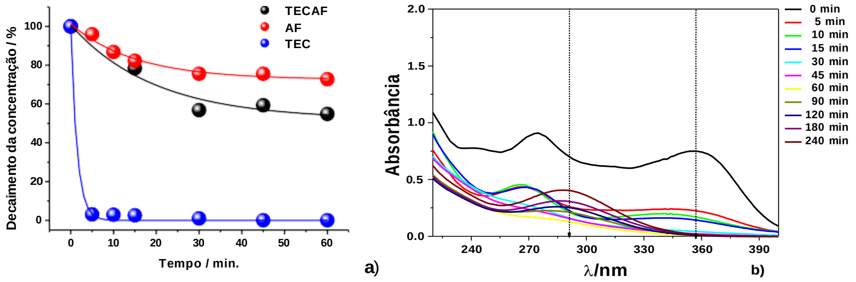

Fig. 1: a) decay of TEC, AF, and TECAF concentrations b) Time-dependent UV-Vis spectrum evolution of TECAF during electrolysis

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Combined process of adsorption, desorption and electrochemical oxidation of antibiotics in ethanol medium

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Wastewater treatment is crucial due to increasing environmental pollution from pharmaceuticals, that threaten water resources. Electrochemical oxidation is a viable method for removing these pollutants but struggles with low-concentration media due to diffusion challenges, necessitating pre-concentration[1,2]. This study examines tetracycline (TeC) degradation through electro-oxidation using a commercial mixed oxide electrode (MMO). It involves an initial pre-concentration step with activated carbon adsorption, followed by desorption in ethanol. Adsorption isotherms for two carbon types showed a better fit to the Freundlich model, Fig. 1a e b, with maximum adsorption capacities of 186.10 mg/g, Fig. 1d,for CAG-Synth carbon and 226.65 mg/g, Fig. 1c. for filter-carbon. In the ethanol desorption process, TeC concentration increased after 50 minutes. Electro-oxidation of TeC achieved high removal rates of 87.57% in water and 84.48% in ethanol after 6 hours of electrolysis. This confirms the effectiveness of activated carbon pre-concentration and electro-oxidation in alcoholic media, suggesting a promising wastewater treatment strategy that could be enhanced by combining with photolysis.

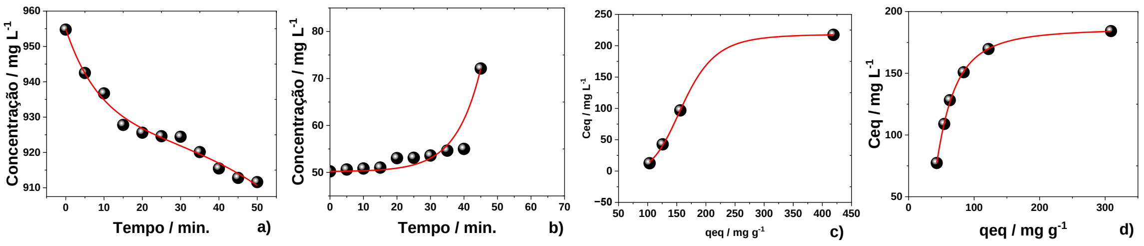

Fig 1. a) Adsorption test for TeC in water and b) Desorption of TeC using ethanol c) Freundlich isotherm for filter carbon d) Freundlich isotherm for CAG-Synth

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Sensores eletroquímicos comerciais utilizados na determinação da serotonina.

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Os eletrodos descartáveis serigrafados (do inglês, Screen – Printed Electrodes – SPEs), produzidos através de tintas condutoras têm se mostrado uma ótima alternativa para realização dos mais diversos tipos de análises eletroquímicas [1]. Pelo fato de serem simples e apresentarem um baixo custo, se tornam uma ótima alternativa para a realização de diferentes técnicas de análise para obtenção de dados sobre uma determinada amostra, pois permite que eles sejam utilizados apenas uma vez e descartados posteriormente, evitando interferências no material [1] [2]. A partir disso este trabalho busca comparar dois sensores eletroquímicos descartáveis comerciais produzidos com tintas diferentes para a determinação da serotonina, inicialmente a voltametria cíclica foi realizada em velocidades de varredura entre 10 mVs^{-1} até 120mVs^{-1}, utilizando a sonda redox ferri-ferro para a caracterização dos eletrodos e estimativa da área eletroativa, que resultou em uma média de 0,13093 cm^2 para o sensores 1 testados e 0,14492 cm^2 para o sensores 2, além disso, dando ênfase nas medidas realizadas a 50 mVs^{-1} foi possível identificar uma corrente de pico tanto para oxidação, quanto para redução, onde o delta EP encontrado após calcular a média foi de aproximadamente 310 mV e 307 mV para os sensores 1 e 2 respectivamente. Posteriormente os testes de voltametria cíclica (CV), voltametria de pulso diferencial (DPV) e voltametria de onda quadrada (SWV) foram realizados nos eletrodos submetidos a uma solução contendo serotonina a uma concentração de 0,1 mmol L^{-1} em tampão fosfato em pH=6, com isso foi identificado correntes de pico referentes ao analito em um potencial de aproximadamente 0,75V para a DPV e SWV nos 2 sensores. Para averiguar a superfície dos eletrodos, o MEV (Microscopia Eletrônica de Varredura) também foi realizado. Desse modo, conclui-se que, apesar de algumas diferenças, ambos os sensores demonstraram ser uma ótima alternativa para realização de análises, pois foram eficientes na determinação da serotonina, e ao passarem pelos testes, apresentaram resultados muito semelhantes aos que se é possível encontrar na literatura.

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On-site electrochemical techniques to stainless steel tanks inspection

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The food-grade storage tanks are widely used by food and beverage manufacturing industries in order to stock their final product and, based on this feature, the storage tank require a high aseptic level in the industrial process [1]. Storage tanks usually have large dimensions, and they are constructed directly at the permanent installation site into a cold chamber framework that operate from -20 ºC to 0 ºC. The industrial maintenance reports demonstrated a close relation between biofilm formation and localized corrosion on AISI 316L stainless steel surface applied in aseptic process. The food-grade product is microbiological contaminated when this phenomenon occurs, and a maintenance project is required to repair the surface to the aseptic quality. However, the maintenance protocols are limited to the liquid penetrant examination and average roughness (Ra) measurements as technique to evaluate the adequacy of the surface to the aseptic applications. The ASME Bio Processing Equipment (ASME BPE) code suggests to use the on-site cyclic polarization measurements to assess the surface in product contact to validate its aseptic properties[2], [3].

This study aims to elucidate the application of the on-site open circuit potential (OCP) and cyclic polarization (CPP) measurements obtained during a maintenance project of the orange juice aseptic tank. This tank showed biofilm formation and localized corrosion and the electrochemical techniques were used to support the decision regarding the aseptic properties of the surface.

The surface defects were detected applying liquid penetrant examination and then they were repaired using a TIG welding procedure. After repair all defects, the surface is re-polished until the average roughness (Ra) be inferior to 0.76 microns. A chemical passivation treatment was executed applying a 15% (w.) nitric acid aqueous solution according to the ASTM A-380. These procedures are not the focus of this paper and so they will not be detailed.

A customized portable electrochemical mini-cell was used to obtain on-site measurements regarding the level of passivation of the aseptic tank. OCP and CPP measurements were performed in 3.5% NaCl solution to evaluate the level of passivation and pitting corrosion resistance. The test script considered an open circuit potential measuring time of 30 minutes, and after that an anodic polarization scan was performed at a sweep rate of 1.67 mV s^{-1}. The anodic scan was reversed at a current density of 1 mA cm^{-2}, and the scan proceeded in the cathodic direction until -200 mV vs. OCP. The electrochemical measurements were performed in two different conditions: (1) As-contaminated surface condition; (2) Surface repaired condition.

The level of passivation of AISI 316L was evaluated and the passivity and corrosion resistance parameters were quantified through on-site cyclic polarization measurements. As-contaminated surface generated an open circuit potential of -110 mV/Ag|AgCl|KCl3mol/L, and the pitting potential (Epit) was +400 mV with a positive hysteresis after reversing the potential scan, meaning that the stainless steel surface was not able to be re-passivated and the localized corrosion is still in progress.

The repaired surface showed the higher level of passivation and suitable to the AISI 316L for aseptic applications, given that the open circuit potential registered was +180 mV/Ag|AgCl|KCl3mol/L, and the pitting potential was not observed and a negative hysteresis was registered after reversing the potential scan, which means that stable pits were not nucleated in the tested conditions. The passivated surface reached an average potential of +500 mV in the passive region. Other relevant comparation between tested surfaces is regarding passivation current density (i_{pass}). Considering the potential of +0.3 V, the i_{pass} to the repaired surface was 92% lower, indicating that the susceptibility to corrosion was drastically reduced.

The onsite electrochemical inspection procedure was efficacy to assess and differentiate a degraded surface from a repaired surface, in terms of corrosion resistance. It was concluded that this procedure was able to quantify the level of passivation as suggested by ASME BPE code and it can be useful to validate the on-site maintenance services focused in repairing aseptic surfaces.

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Desenvolvimento de eletrodo serigrafado multiplex com tinta de negro de fumo e betume para detecção de Catecol

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O desenvolvimento de sensores eletroquímicos é uma ótima alternativa para analises ambientais devido a seu excelente desempenho analítico, aliada à simplicidade e ao relativo baixo custo de produção. Para este fim, foi desenvolvido um sensor multiplex, com dois eletrodos de trabalho, que permite detecções simultâneas, resultando em custos reduzidos e maior número de análises em menos tempo [1]. Para a produção em larga escala de eletrodos, foi selecionada a técnica de serigrafia, conhecida pela sua rapidez e simplicidade, e o substrato utilizado foi o acetato [2]. Uma nova tinta condutora foi produzida, formulada com negro de fumo (CB, do inglês carbon black), um composto carbonáceo com excelente condutividade e custo baixo relativo, e betume, um polímero viscoso derivado de hidrocarbonetos e nunca usado na literatura para esses fins. Desta forma, o sistema serigrafado desenvolvido foi aplicado para o catecol (CA), um composto fenólico utilizado na indústria farmacêutica, cosmética e fotográfica, que gera resíduos tóxicos significativos, representando um risco ambiental. Sua detecção é de extrema importância para o controle ambiental, por exemplo [3]. Nesse contexto, foi realizada a caracterização eletroquímica do sistema utilizando hexaminrutênio $1,0 \times 10^{-3}$ mol L^{-1} como sonda eletroquímica em KCl 1,0 mol L^{-1}, com voltametria cíclica e velocidade de varredura entre 10 e 100 mV s^{-1}. O cálculo da área eletroativa resultou em $0.062 \pm 0.0141 cm^2$ para o eletrodo de trabalho 1 e 0.061 ± 0.0203 cm^2 para o eletrodo de trabalho 2. Para a detecção do CA foram realizadas medidas de voltametria cíclica e de onda quadrada com velocidade de varredura de 50 mV s^{-1}, na qual observou se um pico catódico em torno de 0,1 V, em solução de $1,0 \times 10^{-4}$ mol L^{-1} CA. Para caracterização morfológica foi utilizada microscopia eletrônica de varredura. Desta forma, este estudo destaca a formulação de uma nova tinta condutora à base de betume e CB, além da fabricação de um sistema serigrafado multiplex para detecção de CA, contribuindo para o avanço das técnicas analíticas voltadas à preservação ambiental e ao controle de efluentes industriais.

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PREPARAÇÃO DE MATERIAIS A BASE DE NIÓBIO APLICADOS A FOTOCATÁLISE E A BATERIAS.

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O nióbio (Nb) desempenha um papel crucial na economia e no desenvolvimento industrial do Brasil. Como o maior produtor mundial, o Brasil detém mais de 90% das reservas globais de nióbio. Desta maneira esse elemento químico estratégico contribui de modo significante para as exportações brasileiras, gerando receitas substanciais. No entanto, a produção atual é suficiente para suprir a demanda mundial de nióbio, mesmo considerando um crescimento estimado de 10% ao ano. Portanto, é estratégico desenvolver novas aplicações para o uso do Nb. Assim, este trabalho descreve o esforço de produção de diferentes materiais, baseados no uso do Nb, visando a aplicação como fotocatalisador na degradação de poluentes e como ânodo em baterias de íon sódio. Foram produzidos, principalmente pela técnica hidrotermal, um niobato de zinco ($ZnNb_2O_6$) para aplicação fotocatalítica, além de um niobato alcalino ($KNbO_3$) e um polioxoniobato ($Na_{14}[H_2Si_4Nb_{16}O_{56}]$) para aplicação em baterias de íon sódio.

A avaliação da ação fotocatalítica do $ZnNb_2O_6$ para a descolorização do Azul de Metileno produziu 95% de remoção de cor após 40 min de irradiação. Já os materiais $KNbO_3$ e $Na_{14}[H_2Si_4Nb_{16}O_{56}]$ foram analisados como ânodo em baterias de sódio. Esses materiais apresentaram capacitâncias especificas relativamente baixas de 20 e 40 $mAhg^{-1}$, respectivamente, entretanto ambos apresentaram alta estabilidade a ciclagem mantendo cerca de 90% da capacitância especifica, após 2000 ciclos. Os materiais preparados apresentaram bom desempenho quando comparados a diferentes sistemas divulgados na literatura.

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Development of a carbon paste electrochemical sensor modified with multi-walled carbon nanotubes for the detection of fipronil

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Brazil has emerged as a leading consumer of pesticides globally and is the largest importer of these substances in 2021, importing substantial quantities of active ingredients, including fipronil (FIP). FIP is a phenylpyrazole insecticide that is used to control various pests. It poses environmental and human health risks and is unstable in aquatic habitats[1]. Consequently, it is crucial to develop methods for detecting pesticides, such as FIP, that are simple to prepare, highly sensitive, and accurate at low concentrations. Electrochemical sensors are ideal for this task because of their sensitivity, reproducibility, ease of miniaturization, simplicity, and low cost. Modifying electrodes with nanomaterials is an effective method to enhance the performance of electrochemical sensors for pesticide detection. These nanomaterials amplify the signal through strong adsorption on a large surface area, exhibiting high specificity and increased electron transfer rate. Among these nanomaterials, multiwalled carbon nanotubes (MWCNTs) are particularly effective in accelerating electron transfer between electrodes and detection molecules, owing to their exceptional electrical properties[2]. The objective of this study was to develop a sensor for the detection of FIP using a carbon paste electrode that had been modified with MWCNT. To create the carbon paste electrode (CPE), a mixture of 83% w/w graphite and 17% w/w mineral oil was prepared and homogenized in a mortar with a pestle for 20 minutes. CPE10CNT was prepared by adding 10% w/w MWCNT into the carbon paste mixture, which contained 75% w/w graphite and 15% w/w mineral oil. CPE20CNT was prepared using a similar methodology, but with a higher concentration of MWCNT (20% w/w). The pastes were then inserted into the electrode's 3 mm orifice. A three-electrode electrochemical cell was used for the detection of FIP, which included a working electrode, a reference electrode consisting of Ag|AgCl (KCl 1 M), and a counter electrode made of platinum wire. Electrochemical characterization and detection of FIP were performed using a PalmSens potentiostat and PSTrace 5.9 software. For electrochemical characterization, a solution of 5 mM $K_3Fe(CN)_6$ in 0.1 M KCl was used, and the Cyclic Voltammetry (CV) measurements were performed at different scan rates (25 – 400 mV s^{-1}). To observe the electrochemical behavior of the FIP, an aliquot 2.50 µM was used with 0.1 M Britton-Robinson (BR) buffer solution (pH 10) and CV measurements were conducted at different scan rates from 25 – 400 mV s^{-1}. The optimized parameters for FIP (pH 10) in SWV were an amplitude of 50 mV s^{-1}, frequency of 100 Hz, and step potential of 2 mV s^{-1}. The Bare CPE, CPE10CNT and CPE20CNT electrodes demonstrated reversibility, with an I_{pa}/I_{pc} ratio of 0.95 ± 2.7, 1.01 ± 0.09 and 1.17 ± 0.16, respectively. Moreover, the peak potentials (ΔE_p) decreased: 0.28 ± 0.28 V for Bare CPE, 0.26 ± 0.05 V for CPE10CNT, and 0.16 ± 0.02 V for CPE20CNT. Additionally, the electroactive area increased (0.039 ± 0.005 cm^2 for Bare CPE and 0.059 ± 0.014 cm^2 for CPE20CNT), which was calculated using the Randles–Ševčík equation. The Bare EPC and CPE20NTC electrodes displayed current peaks for FIP at approximately 5.3 and 10.4 µA an irreversible electrochemical process at 0.4 V s^{-1} in FIP solution, demonstrating that the MWCNTs significantly enhanced the detection signal. The interaction between the FIP, an aromatic molecule, and the MWCNT surface occurs through hydrophobic, electrostatic, and π–π stacking interactions, facilitating the oxidation of FIP molecules on the electrode surface. Under optimal conditions (pH 10), the detection FIP measured by SWV varied linearly ranging from 0.05 to 6.5 µM, with a detection limit of 0.315 µM and a quantitation limit of 0.945 µM ($y = 3{,}35x + 0{,}966$ and $R^2 = 0{,}991$). The CPE electrodes demonstrated better performance in FIP detection, with CPE20CNT displaying superior results compared to the Bare CPE and CPE10CNT. The hydrophobic nature of both the FIP and MWCNT, combined with the increased electroactive area of the electrode (0.059 ± 0.014 cm^2), contributed to the adsorption of the molecule on the electrode surface.

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Electrosynthesis of acetate from ethanol using a BDD electrode

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Primary alcohols are susceptible to various reaction paths during electrochemical oxidation (EO), which may results in the generation of carboxylic acid (acetic acid) or aldehyde (acetaldehyde) rather than CO_2. Since these products are commodities for many industrial sectors, they are highly desirable from a commercial perspective. Among the primary alcohols, ethanol stands out as a compound with low toxicity, easy to store, safe to transport, carbon neutral and it can be obtained from renewable sources, such as biomass. Pt anodes have been used in studies on the EO of ethanol. However, the production of CO can poison this type of electrode. Therefore, other anode materials have been studied. Among them, boron doped diamond (BDD) films electrodes result to be attractive materials. In addition, it has a wide potential window, leading to the formation of hydroxyl radicals and ozone in aqueous media, favoring ethanol electrochemical conversion process. The aim of this study was to evaluate the EO of ethanol (300 mg L^{-1}) in a basic medium to selectively produce acetate as the priority product, using BDD electrode. To this end, different concentrations (0.05 and 0.5 mol L^{-1}) of the support electrolyte (NaOH) and different current densities (20, 40 and 80 mA cm^{-2}) were studied. It has been demonstrated that when 0.05 mol L^{-1} NaOH was employed, the ethanol electroconversion efficiency decreased from 40% (20 mA cm^{-2}) to 6% (80 mA cm^{-2}) as the applied current density rose. This decrease in efficiency could be the result of the mineralization of electrosynthetized acetate due to the production of oxidants, or the synthesis of other organic compounds such ethyl acetate and acetaldehyde. The ethanol electroconversion efficiency into acetate was approximately 42% for all current densities applied when a concentration of 0.5 mol L^{-1} of the support electrolyte was utilized, suggesting that electrode surface saturation had occurred.

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On the electrochemical behavior of modified electrodes based on deep eutectic solvents-carbon black films

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Deep eutectic solvents (DES) are considered green solvents, since they display remarkable features such as biodegradability and low toxicity. In addition, the ease of preparation and low cost are highlighted [1, 2]. In this context, DES have been studied in the most diverse applications including green catalysis, extraction of bioactive compounds, synthesis of advanced materials and electrochemistry [2]. In this work, the use of different DES (Reline, Ethaline and Glyceline) as well as carbon black (CB) in the preparation of modifying films of glassy carbon electrode (GCE) was evaluated using as electroactive model analyte the neurotransmitter dopamine (DA). The results obtained by cyclic voltammetry (CV) and differential pulse voltammetry (DPV) showed the characteristic redox peaks of DA. Among the studied modified electrodes, the one prepared with CB and Ethaline (ETA-CB/GCE) presented a three times higher signal intensity when compared to the bare GCE. Furthermore, evaluating the peak-to-peak potential separation (ΔE_p), it was observed that the same modified electrode provided a smaller value of 54.0 mV (at 50 mV s^{-1}), indicating a more reversible process. From CV studies carried out at different scan rates, it was possible to calculate the respective heterogeneous electron transfer rate constants (k^0) by applying the Nicholson-Shain method. The proposed ETA-CB/GCE presented the largest k^0 value (4.7×10^{-3} cm s^{-1}). The set of results shows the effectiveness of DES and CB as electrode modifiers in the manufacturing of low cost and sustainable electrochemical sensors, with emphasis on the influence of the type of DES on the electrochemical behavior of the respective films.

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Synthesis of a polypyrrole hydro-sponge and recycled MnO2 composite from spent alkaline batteries

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Recently, the scientific community has been dedicated numerous efforts in the development of ecological, sustainable and renewable energy sources. In this context, efficient and low-cost electrical energy storage systems are demanded. This work presents the synthesis and electrochemical characterization of a nanocomposite between polypyrrole hydro-sponge and recycled manganese dioxide from the cathodes of spent Zn-MnO_2 batteries for application in supercapacitors. The synthesis was made by cyclic voltammetry in two steps: (1) synthesis of polypyrrole hydro-sponge and (2) deposition of MnO_2 onto polypyrrole surface. The electrochemical characterization was performed using a three-electrode system in KCl 0.1 mol L^{-1} aqueous solution. It was observed that the composite showed a specific capacitance of 102.42 F g^{-1} at 10 mV s^{-1}. This value is very close to that reported for polypyrrole hydro-sponge in the literature [1][2]. However, our studies have been indicated that the manganese dioxide obtained from spent batteries improves the electrochemical stabilities of the hydro-sponge over several voltammetric cycles: after 100 cycles, the capacitance retention of the composite and polypyrrole hydro-sponge were 86.47% and 77.64%, respectively, under the same conditions. The results indicate the feasibility of using recycle manganese dioxide from spent batteries in the formation of nanocomposites with polypyrrole. The obtained material showed high potential for application in supercapacitor.

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Metodologia exploratória de sensor eletroquímico para análise e detecção de *Salmonella enterica* em ambientes diversos

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Eletrodos quimicamente modificados oferecem a vantagem de produzir filmes uniformes e altamente aderentes, os quais podem ser ajustados através de parâmetros como a concentração de metais, varreduras de potencial e solventes, entre outros. As doenças transmitidas por alimentos (DTA) são uma preocupação significativa devido à presença de microrganismos contaminantes, muitas vezes resultantes de falhas na higienização dos alimentos. Uma abordagem promissora para detectar esses contaminantes é o uso de aptâmeros, que são oligonucleotídeos de fita simples com alta afinidade para moléculas orgânicas, embora sua aplicação ainda seja limitada para bactérias Gram-negativas.

Este estudo explora o uso de um sensor eletroquímico baseado em eletrodo impresso de carbono (SPE) para a detecção rápida de *Salmonella enterica* em ambientes relevantes. A sonda redox foi preparada com ferrocianeto de potássio $K_4[Fe(CN)_6]$ a uma concentração de 6 mmol/L em KCl 1M. Os aptâmeros foram suspensos em água e testados em concentrações variadas, de 100 nM a 1000 nM, seguidos por ensaios microbiológicos com *Salmonella enteritidis* em ágar Verde Brilhante.

A modificação do SPE foi realizada através da eletrodeposição controlada de íons Fe(III), utilizando um volume de 60 µL na superfície do eletrodo de trabalho, a análise foi feita por voltametria cíclica (VC) utilizando um micro-potenciostato µDrop@sens. Os resultados mostraram uma resposta adsortiva na superfície do SPE, evidenciada pelas variações nos voltamogramas cíclicos de corrente capacitiva.

Embora os resultados sejam preliminares, eles indicam um potencial promissor para futuros estudos e aplicações práticas. Este trabalho contribui para o desenvolvimento de tecnologias eficientes de monitoramento microbiológico em processos alimentares. O estudo foi possível graças ao apoio e financiamento do Laboratório de Fotoquímica-UNESP, Embrapa Instrumentação, Famerp e NanoChemTech Solutions.

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Nb_2O_5 electrodes prepared by different methods: photoelectrochemical properties and photocatalytic performance.

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Niobium pentoxide (Nb_2O_5) has attracted a great amount of interest due to its high performance and applications in photoelectrochemical devices, catalysts, and water splitting [1]. Despite its usage in photocatalysis in powder form, Nb_2O_5 is still under-researched in the electro photocatalysis field as a photoanode for the degradation of organic molecules. Due to its large bandgap of 3.4 eV, Nb_2O_5 is adequate for redox reaction due to its absorption in the UV region. Depending on the conditions of the synthesis, it is possible to obtain different structures of Nb_2O_5, with the transition from amorphous to orthorhombic, pseudohexagonal, and monoclinic phases possible with the changes in temperature [2]. Amongst the characteristics of this material, there is high stability which makes it a good candidate for photoelectrochemical studies. This research aims to obtain and characterize different Nb_2O_5 films via different synthetic routes and treatments. Their photoelectrochemical properties via photocurrent measurements were analyzed, and films used for degradation of a selected organic compound.

The two methods employed were the Pechini and anodization followed by calcination treatment. The Pechini method consisted of the application of niobium resin layers in a titanium substrate followed by a heat ramp and calcination at 500°C. The anodizing process was realized in a Nb plate via the application of 40mA cm^{-2} in H_2SO_4 0,1 mol L^{-1} solution, followed by calcination at 600°C in an inert atmosphere. Both electrodes produced were further treated via cathodic treatment by application of current in H_2SO_4 0,1 mol L^{-1} solution. The characterization of such films was done via X-ray diffraction (DRX), scanning electron microscopy (SEM), Mott-Schottky, and linear sweep voltammetry (LSV) in the presence and absence of light. There was an increase in the photocurrent of the electrodes obtained post-cathodic treatment compared to those without said treatment, with both before and after showing high stability in an acidic solution. The Nb_2O_5 electrode chosen was used for degradation of 2,4-D and has demonstrated promising results: in one hour of photoelectrochemical degradation, 52.1% removal was identified without optimization, showing promise and deserving further studies.

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Enhanced glyphosate detection using laser-induced graphene electrodes modified with copper phthalocyanine/gold nanoshells

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Glyphosate (N-(phosphonomethyl) glycine), one of the most widely used herbicides, poses significant health and environmental risks despite ongoing debates about its carcinogenicity. Prolonged and extensive glyphosate use has been linked to various adverse health effects, including miscarriage, reduced fertility, thyroid disease, cardiopulmonary disorders, muscular issues, neural disorders, neoplasia, and fetotoxic effects [1]. Given its widespread use and associated toxicity, detecting glyphosate residues in environmental and food samples is crucial for ensuring both environmental and food safety. Conventional detection methods struggle due to glyphosate's ionic nature, low volatility, high water solubility, and low molar mass. Thus, a simple, efficient, and highly selective detection method is needed [2]. In this study, we introduce an environmentally friendly and cost-effective electrochemical sensor. This sensor is crafted from laser-induced graphene electrodes modified with copper phthalocyanine and gold nanoshells. The sensor leverages the affinity between glyphosate's phosphate, amine, and carboxyl groups and Cu^{2+} ions, forming a glyphosate–Cu^{2+} complex that inhibits current response [3]. Moreover, the incorporation of gold nanoshells enhances the sensor's electrochemical response. Characterization techniques such as cyclic voltammetry, Raman spectroscopy, scanning electron microscopy with field emission gun (SEM-FEG), and X-ray photoelectron Spectroscopy XPS) confirmed the synthesis and modification of the carbonaceous material on the electrode. Glyphosate detection was achieved using square wave voltammetry at an oxidation potential of 0.25 V. This methodology shows great potential for pesticide detection and can be applied to monitor glyphosate in fruits and tap water.

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Electrochemical synthesis of nanoporous nickel electrode applied as a cathode for water electrolysis

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The development of a clean and sustainable energy matrix is one of the most important topics of the 21st century. Hydrogen (H_2) is seen as the ideal substitute for fossil fuels since its combustion does not release pollutants. Producing H_2 through water electrolysis allows energy storage in the H_2 molecule, which can be restored in fuel cells[1]. A good electrocatalyst for this system must have high physical-chemical stability, low production cost, low overpotential, and high exchange current density [2]. Thus, this work shows the development of a nanoporous nickel (NiNPs) through electrochemical synthesis to be applied as a cathode for water electrolysis. NiNPs films were prepared cronoamperostatically in a tree-electrode cell on Ni substrate as working electrode, Pt as counter electrode, and $Ag/AgCl_{KCl(sat)}$ as reference electrode, applying 0.5, 1.0, and 2.0 A cm^{-2} for 50, 150, and 300 s. The precursor solution was composed of $NiCl_2{\cdot}6H_2O$ 0.1 M, NH_4Cl 1.0 M, and NaCl 1.0 M. The NiNPs electrodes were characterized by SEM, EDS and DRX. The electrochemical characterization was performed using KOH 1.0 M through a polarization curve at 5 mV s^{-1}, to extract the overpotential and Tafel parameters. In addition, EIS and capacitance experiments were realized to extract the charge transfer resistance (R_{ct}) and ECSA values. The optimized electrode showed a nanoporous morphology with elevated ECSA, resulting in lower overpotential, R_{ct} and Tafel slope values, showing to be a good candidate to substitute Pt electrode for water electrolysis.

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Imunoensaio eletroquímico para detecção de anticorpos específicos de hanseníase utilizando peptídeo sintético

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A hanseníase é uma doença infecciosa crônica causada pela bactéria *Mycobacterium leprae* e, até o momento, seu diagnóstico é feito majoritariamente por exame clínico [1]. Assim, neste trabalho foi desenvolvido um imunoensaio baseado em peptídeo sintético para detecção de anticorpos específicos anti-*M. leprae* em soro humano. O peptídeo foi utilizado juntamente com partículas magnéticas (PMs) para a captura seletiva dos anticorpos. Ademais, à uma das extremidades do peptídeo, adicionou-se cisteína, de modo a orientar sua ancoragem às PMs via ligação covalente com maleimida, garantindo resultados mais específicos e minimizando impedimento estérico. Por fim, adicionou-se ao imunoensaio anticorpos anti-IgG marcados com a enzima *horseradish peroxidase* (HRP), obtendo o bioconjugado final PM/pep/anti-*M. leprae*/anti-IgG-HRP. A detecção eletroquímica foi baseada em eletrodos serigrafados de carbono totalmente descartáveis (SPCE) e a técnica de voltametria de pulso-diferencial (DPV), tendo como solução de detecção H_2O_2 e hidroquinona (HQ). A concentração do peptídeo foi otimizada na faixa de 5,0 a 40,0 μg mL^{-1}, obtendo-se um valor ideal de 10,0 μg mL^{-1}. Além disso, a concentração de etanolamina, usada no bloqueio de sítios não específicos, também foi estudada com valor ótimo de 1,0 mmol L^{-1}. Adicionalmente, avaliou-se o uso de soro de cabra na etapa de captura do anticorpo na amostra e os resultados indicam minimização do efeito matriz, garantindo melhor seletividade à detecção. O imunoensaio apresentou elevada precisão intermediária (DPR = 5,31%) e repetibilidade (DPR = 3,92%) nas condições experimentais otimizadas. Ademais, foi capaz de diferenciar com eficiência pacientes com hanseníase multibacilar dos indivíduos saudáveis, apresentando alta seletividade e especificidade. Deste modo, apresenta-se como alternativa viável e de baixo custo para auxiliar no diagnóstico de hanseníase.

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Zeolitic imidazolate framework-Zn structure applied as catalyst of the Printex L6 carbon for H_2O_2 electrogeneration by using a Gas Diffusion Electrode

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In recent years, water treatment and purification focus on developing new, on-demand and decentralized technologies. In this sense, electrochemical flow reactors (EFR) using systems that contain gas diffusion electrode (GDE) for the electrogeneration of oxidants like hydrogen peroxide (H_2O_2) is promising. However, work that reports the investigation of novel electrode materials remains critical for improving efficiency and stability [1]. This study proposes the modification of carbon-based GDE with zeolitic imidazolate framework-Zn (ZIF-Zn) structure to optimize the oxygen reduction reaction (ORR) towards H_2O_2 production. We employed a previously established method for GDE synthesis to incorporate 0.5-2.0 wt% of a ZIF-Zn into a commercial carbon matrix Printex-L6 (CPL6). The modified GDE served as the cathode, while a commercial DSA®-Cl_2 electrode was used as anode in the EFR system studied elsewhere [2]. A semi-batch system with a 1L reservoir connected to the EFR was employed to evaluate the effects of varying pH (3 - 10) and cell potential (2.5 - 15.0 V) on H_2O_2 production performance. Additionally, reaction kinetics, current efficiency, and energy consumption were investigated. To understand the differences in surface properties between the unmodified and modified GDEs, electrochemical impedance spectroscopy (EIS) was performed. The investigation of ZIF-Zn content within the CPL6 revealed that 1.0 wt% of this catalyst offered the optimal modification for H_2O_2 production, compared to both higher (2.0 wt%) and lower (0.5 wt%) ZIF-Zn loadings. Among all test conditions, the ZIF-Zn/CPL6(1%) exhibited the

highest H_2O_2 concentration after 60 min. of electrolysis. At a cell potential of 10.0 V, the modified electrode achieved a superior current efficiency (23.9%) compared to the unmodified CPL6-GDE (12.3%). The current density experiments at 100 mA cm^{-2} revealed a plateau in H_2O_2 production for both electrodes (CPL6 and ZIF/CPL6(1%)) after 3 h. Prior to this plateau, a linear increase was observed, allowing for the definition of a kinetic system for H_2O_2 production. The apparent rate constant derived from the slopes of linear curves were 16.4 and 12.2 mg L^{-1} min^{-1} for ZIF-Zn/CPL6-GDE(1%) and CPL6-GDE, respectively. The modified GDE showed better performance at all tested pH values. Interestingly, the variation of H_2O_2 concentration by change the initial pH during electrolysis was minimal for both electrodes. The repeatability of the H_2O_2 production process by using ZIF-Zn/CPL6(1%)-GDE was confirmed by testing three electrodes prepared from the same batch, being an RSD of 13.6%. The EIS analysis, by Nyquist plots, indicated a porous material profile for both electrodes, as expected for GDEs. The Bode plot for the CPL6-GDE exhibited three distinct phase changes, which can be attributed to the ORR proceeding through multiple pathways. The first likely involves a two-electron transfer, generating H_2O_2, while the second can be the H_2O_2 reduction into H_2O. The third phases might correspond H_2O reduction, since this phase occurs at the same frequency for both electrodes. This multi-step process could explain the lower efficiency observed for the CPL6-GDE compared to the ZIF-Zn/CPL6(1%). In conclusion, this study demonstrates the successful application of ZIF-Zn for modifying commercially available CPL6 matrices to enhance the efficiency of GDE to H_2O_2 production via ORR. This represents a novel approach in GDE modification for this specific application, as evidenced by the lack of prior other studies. The findings contribute significantly to our understanding of how material modifications can influence for the H_2O_2 electrogeneration using GDE. This knowledge provides a valuable foundation for future research efforts aimed at optimizing electrocatalysts for improved performance and efficiency.

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Electrochemical genosensor for rapid and simplified detection of zika virus DNA using safranin as DNA genomic intercalator

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Zika virus (ZIKV) has been recognized as a significant threat to global public health due to its association with severe conditions such as Guillain-Barré syndrome and microcephaly. The World Health Organization emphasizes the urgent need for accurate and timely diagnostics to control the spread of this virus, particularly in tropical regions where it is most prevalent [1]. Current diagnostic methods, such as Polymerase Chain Reaction (PCR), are limited by their high cost and the requirement for trained personnel, which poses challenges in resource-limited settings [1]. Biosensors, particularly electrochemical genosensors, offer a promising alternative due to their cost-effectiveness, ease of use, and high sensitivity. These devices combine biocomponents like DNA probes with technological platforms to provide precise measurements of analytes in biological samples [2]. Electrochemical genosensors are especially suitable for clinical diagnostics due to rapid response and minimal equipment requirements [3].

This study presents an electrochemical genosensor designed for the rapid and simplified detection of Zika virus DNA, using the biological dye safranin as a genomic DNA intercalator. The genosensor utilizes a gold-printed electrode modified with a bilayer formed by cysteamine and glycine-functionalized graphene quantum dots, which facilitates the immobilization of oligonucleotide probes specifically designed for the Zika virus. Electrochemical detection is carried out using differential pulse voltammetry (DPV), monitoring the peak current of the DNA intercalator, in this case, safranin. The peak current results from the reduction of safranin intercalated into the double strand of the immobilized oligonucleotide probe. The biosensor demonstrated detection limits for Zika virus DNA concentrations in the range of 10^{-13} mol. L^{-1}. Modifications made to the sensor were monitored by scanning electron microscopy (SEM) and atomic force microscopy (AFM).

The surface modification of the electrode was confirmed by SEM and AFM analyses. SEM images showed interconnected circular structures on the electrode surface after cysteamine modification, indicating significant morphological changes. AFM analysis corroborated these findings, showing changes in the surface topography that facilitate biomolecule adsorption.

FTIR spectroscopy was employed to analyze the bilayer of cysteamine and graphene quantum dots (GQDs). The FTIR spectra revealed characteristic peaks of both cysteamine and GQDs, indicating their presence and interaction within the bilayer. This analysis confirmed the effective interaction between cysteamine and GQDs, essential for stabilizing and functionalizing the electrode surface.

Electrochemical detection using DPV monitored the reduction peak of safranin around -0.6 volts, proportional to the concentration of safranin intercalated in the DNA double strand. This relationship allowed for the precise quantification of the target DNA. A calibration curve was constructed using Zika virus DNA concentrations ranging from $5x10^{-11}$ to $5x10^{-13}$ mol. L^{-1}, demonstrating a clear linear relationship between target concentration and the electrochemical response.

The genosensor showed an impressive detection limit of $5x10^{-13}$ mol. L^{-1}, indicating high sensitivity. This low detection limit is crucial for early diagnosis of Zika virus infections, enabling timely therapeutic interventions. The simplicity and minimal training required for result interpretation make this genosensor ideal for use in resource-limited settings, enhancing its applicability in various environments, including public health initiatives.

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Electroreduction of nitrate to ammonia through Co_3O_4 nanowires supported on TiO_2

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Ammonia (NH_3) is widely used in industry as a raw material for synthesis of reagents, drugs and fertilizers. Furthermore, due to its high volumetric energy density (4.32 kWh L^{-1}) and carbon-free composition, it has the potential to be a carrier of renewable energy.[1] In search of alternatives to the well-established Haber-Bosch process, the electrochemical synthesis of ammonia from the nitrate reduction reaction (NO_3RR) has attracted interest, because it use clean and renewable energy, exploring the mitigation of nitrate contamination in water resources, without emitting pollutants. To minimize the energy cost of NO_3RR, catalysts with performance similar to that of noble metals are being developed.[2] Therefore, in this work, Co_3O_4 nanowires on TiO_2 were synthesized as a catalyst using a methodology consisted of cobalt electrodeposition followed by calcination. The performance of the catalyst was evaluated in an H-type electrochemical cell in Ar-saturated $NaNO_3$ 20 mmol L^{-1} and NaOH 1.0 mol L^{-1}. Cyclic voltammetry results of the catalyst immersed in electrolyte without the presence of nitrate shows activity characteristic of the hydrogen evolution reaction at potentials near to -0.3 V vs RHE. However, with the catalyst immersed in electrolyte containing nitrate, the current density increases at less negative potentials, associated with nitrate reduction. To evaluate the catalytic performance, potentiostatic electrolysis was carried out, followed by product quantification. The maximum faradaic efficiency reached 66.0 ± 5.77% at -0.3 V vs RHE, and in relation to the yield rate, the maximum was reached at 0.0 V vs RHE (6.69 ± 0.818 µmol h^{-1} cm^{-2}), indicating that the catalyst has a low overpotential for NO_3RR. Given the considerations above, Co_3O_4 nanowires on TiO_2 have demonstrated to be promising catalysts for NO_3RR.

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Amperometric Method For Determination of Venlafaxine Using A BIA System And BDDE

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Venlafaxine (VEN) is an antidepressant whose main function is to increase neurotransmitters. The technique of Batch Injection Analysis (BIA) coupled with an electrochemical detector using a boron-doped diamond electrode (BDDE) has been widely used to study different substances [1,2]. This work aimed to develop a quick and sensitive electroanalytical method to quantify VEN in drug samples and synthetic urine. The standard VEN solutions were prepared at a concentration of 0.10 mol L^{-1} and subsequent dilutions were made, the Britton-Robinson (BR) buffer solution 0.04 mol L^{-1}, pH = 2.0, was used as the supporting electrolyte. The reference electrode used was Ag/AgCl (KCl 3.0 mol L^{-1}), the auxiliary electrode was a platinum wire and the BDDE used has an area of 0.19 cm^2. The electrochemical measurements were performed on the PalmSens 4 potentiostat. Initially, the electrochemical parameters of the system were evaluated. Subsequently, the parameters of the analytical curve were obtained in a linear concentration range of 0.03 to 1.00 μmol L^{-1} with a straight-line equation of I_p (μA) = 0.1748 + 0.848029 [VEN] and a calculated detection limit value of 3.70 nmol L^{-1}. Intra and inter-day repeatability studies were carried out, where the relative standard deviations obtained were less than 11%. The interference study was carried out with substances prepared in different proportions, there was no significant interference of these substances in the quantification of VEN. Addition and recovery studies were carried out with synthetic urine, for the analysis of VEN 0.50 μmol L^{-1}, a recovery of 97.4% was obtained, indicating no significant interference from the sample matrix in the quantification of VEN. Two pharmaceutical samples were analyzed by the proposed method and by a comparative method, using spectrophotometry, the results obtained were concordant to a level of 95%. The proposed electroanalytical method is quick, samples to execute, precision, and has low reagent consumption, there is no need for complex sample preparation and use of organic solvents.

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Xylene biodegradation and simultaneous bioelectricity production in a microbial fuel cell using mixed culture present in mangrove sediments

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Xylene is present in oil and its derivatives, and as they are extremely toxic, genotoxic and mutagenic, even at low concentrations, causing serious damage to the environment and the human health[1]. A very attractive alternative for biodegradation of this compound is the use of microbial fuel cells (MFC), which simultaneously produces clean energy. This study aims to develop an MFC to degrade xylene and simultaneously generate electricity. For this, a MFC (50 mL) was constructed. The anode was a carbon fiber brush (3.0 cm × 2.0 cm) and the cathode a carbon cloth modified with 40% Pt (15 cm^2) hot-pressed into Nafion® proton exchange membrane. The voltages generated by the MFC were recorded, each 15 minutes, using an Arduino Mega 2560 microcontroller board programmed in the C++ language. To obtain the biofilm, a mixed microbial culture from mangrove sediment was used (Latitude: − 20°16’45.3’’S; Longitude: − 40°18’27.4’’W). A volume of 50.0 mL of a suspension at 20.0% (m/v) composed of sediment and Lovley and Philips culture medium was transferred to the MFC. As the primary substrate for microorganisms, sodium acetate at 1.0 g L^{-1} was used, being gradually replaced by xylene. An external resistance of 1000 Ω was connected between the anode and cathode. As the biofilm growth increased the output voltage of the MFC to 320 mV and a maximum power density of 63.5 mW m^{-2}. The biofilm reduces the internal resistance (Rint) from 2177 Ω (abiotic condition) to 435 Ω. The MFC degraded all contaminant (xylene) present in the medium (324.0 mg L^{-1}) in less than 100 hours with simultaneous production of electricity. The evaluation of the microbial community showed a distinct microbial diversity of the initial mangrove sediment and after fed with sodium acetate. The results obtained suggested that biofilm formation greatly improves the electronic transfer process, making the developed MFC an excellent, promising and attractive alternative for xylene biodegradation and simultaneous energy production.

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Simultaneous detection of paraquat and carbofuran through electrochemical technique using a modified polymeric electrode

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Water contamination is a major problem in contemporary society. In this context, there is a need to develop simple, portable, and selective detection analysis methods in water. Therefore, this work presents a flexible and biodegradable sensor (g-PBAT). The substrate was modified using the Layer-by-Layer (LbL) technique, alternating poly(allylamine hydrochloride) (PAH) and carbon nitrite dots (CND). The functionalized surfaces were used to detect paraquat (PQ) and carbofuran (CBF) in water using electrochemical methods. Electrochemical characterization was performed using cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS) and differential pulse voltammetry (DPV) to detect PQ and CBF. The electrochemical characterization showed good reproducibility (RSD 14%), repeatability (RSD 8.8%) and stability, with the current signal peak increasing by only 8.5% in relation to the initial cycle. Among the analytical parameters determined, sensitivity values of $3.69E^{-5} \pm 5.94E^{-7}$ A cm^{-2} M^{-1} were found with LOD 0.63 μM and LOQ 2.09 μM for PQ and sensitivity values of $2.51E^{-7} \pm 2.09E^{-8}$ A cm^{-2} M^{-1} with LOD 7.14 μM and LOQ 23.8 μM for CBF. The addition–recovery methodology was used, with recoveries ranging from 89.3% to 105.0% to PQ and 92.1% to 105.9% for CBF, indicating that the sensor presents good sensitivity for the real sample presenting a stable RSD, ranging from 4% to 7.5%. The sensor demonstrates peak effectiveness in detecting pollutant molecules such as PQ and CBF in contaminated water.

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