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Electro-synthesized composite of polyaniline and gum Arabic for colorimetric ammonia vapor detection

Composto eletrossintetizado de polianilina e goma arábica para detecção colorimétrica de vapor de amônia

Compuesto electrosintetizado de polianilina y goma arábiga para detección colorimétrica de vapor de amoníaco

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Abstract
Polyaniline (PAni) is a conductive polymer with colorimetric properties, which can be combined with biopolymers to form novel composite materials. These materials can be used as a colorimetric sensor/indicator in the identification of different kinds of analytes. The present study aimed to develop a film of PAni and gum Arabic (GA) by electrochemical method to evaluate its potential use in a colorimetric sensor for ammonia vapor (NH_3). The composite PAni/GA was prepared by electrochemical polymerization on a gold surface using the cyclic voltammetry technique. The stability of the films was evaluated in acid (HCl 0.1 mol L^{-1}) after 25 imaging cycles. Strongly acidified gum arabic solution (pH 0.0) and a sweep speed of 10 mV s^{-1} caused higher current density in the film formation. The morphology of the films was observed by SEM and the presence of cracks in its structure can favor the entry of gases. The response of the composite in the presence of NH_3 was measured using a colorimeter. The PAni/GA composite is a promising and rapid tool for use in colorimetric sensors in the detection of NH_3.
Keywords: PAni; Polysaccharide; Electrochemical synthesis; Composite.

Resumo

A polianilina (PAni) é um polímero condutor com propriedades colorimétricas, que pode ser combinada com biopolímeros para formar novos materiais compósitos. Esses materiais podem ser usados como sensor/indicador colorimétrico na identificação de diferentes tipos de analitos. O presente trabalho teve como objetivo desenvolver um filme de PAni e goma arábica (GA) por método eletroquímico para avaliar seu potencial de uso em um sensor colorimétrico para vapor de amônia (NH_3). O compósito PAni/GA foi preparado por polimerização eletroquímica em superfície de ouro utilizando a técnica de voltametria cíclica. A estabilidade dos filmes foi avaliada em ácido (HCl 0,1 mol L^{-1}) após 25 ciclos de imagem. A solução de goma arábica fortemente acidificada (pH 0,0) e uma velocidade de varredura de 10 mV s^{-1} causaram maior densidade de corrente na formação do filme. A morfologia dos filmes foi observada por MEV e a presença de trincas em sua estrutura pode favorecer a entrada de gases. A resposta do compósito na presença de NH_3 foi medida usando um colorímetro. O compósito PAni/GA é uma ferramenta promissora e rápida para uso em sensores colorimétricos na detecção de NH_3.

Palavras-chave: PAni; Polissacarideo; Síntese eletroquímica; Compósito.

Resumen

La polianilina (PAni) es un polímero conductor con propiedades colorimétricas, que puede combinarse con biopolímeros para formar nuevos materiales compuestos. Estos materiales se pueden utilizar como sensor/indicador colorimétrico en la identificación de diferentes tipos de analitos. El presente trabajo tuvo como objetivo desarrollar una película de PAni y goma arábiga (GA) por método electroquímico para evaluar su potencial uso en un sensor colorimétrico para vapor de amoníaco (NH_s). El composite PAni/GA se preparó mediante polimerización electroquímica sobre superficie de oro utilizando la técnica de voltamperometría cíclica. La estabilidad de la película se evaluó en ácido (0,1 mol L^{-1} HCl) después de 25 ciclos de formación de imágenes. La solución de goma arábiga fuertemente acidificada (pH 0,0) y una velocidad de barrido de 10 mV s^{-1} provocaron una mayor densidad de corriente en la formación de la película. La morfología de las películas fue observada por SEM y la presencia de grietas en su estructura puede favorecer la entrada de gases. La respuesta compuesta en presencia de NH_3 se midió utilizando un colorímetro. El compuesto PAni/GA es una herramienta prometedora y rápida para su uso en sensores colorimétricos en la detección de NH_3.

Palabras clave: PAni; Polisacárido; Síntesis electroquímica; Compuesto.

1. Introduction

Polyaniline (PAni) is a conductive polymer with excellent electrical and mechanical properties, low cost, and good environmental stability. According to the variation of the oxidation state, PAni can exist in different forms, known as leucoesmeraldine (PAni-L) in yellow color; pernigranillin (PAni-P), in purple; and emeraldine (PAni-E) in green color (when salt is formed) and blue color (when it forms a base) (Oliveira, et al., 2019; Macdiarmid, et al., 1987). PAni has a potential for application in the area of colorimetric indicators, due to its very fast and visually perceptible doping and dedoping characteristics, in addition to good chemical stability and conductivity after doping (Kumar, et al., 2016; Anju, et al. 2018).

Recently, natural polymers have been used in new PAni composites for different applications (Babaladimath, et al., 2017; Thambidurai & Pandiselvi, 2018; Sulaiman & AL-Farga, 2020). Similar to what happens with the use of inorganic, organic, and aromatic acid dopants, the solubility of PAni can be improved in the presence of ionic natural polymers, such as some plant gums (Sinha & Kaur, 2019; Quintanilha, et al., 2014). Gonçalves et al. (Gonçalves, et al., 2021) obtained a composite PAni/ gum Arabic with excellent electroactive, physicochemical, and biological properties. Oliveira, (2020) developed a composite of PAni and cashew gum with good electrochemical stability and invariable conductivity, showing high sensitivity to ammonia vapor due to the sudden color change.

Gum Arabic (GA) is a hydrocolloid biopolymer obtained from the gummy exudate of the Acacia tree (Cornelsen, et al., 2015). In aqueous media, anionic groups can lead to the formation of polyelectrolyte complexes, which favors the production of novel composites. The use of gum Arabic and polyaniline for the formation of composites by chemical oxidation is already found in the literature (Gonçalves, et al., 2021; Oliveira, 2020; Quintanilha, et al.,), but few studies are using electrochemical synthesis, which is considered a method easier, with few steps, specific, and more reproducible (Guimard, et al., 2007). Thus, in this study, a novel composite of gum Arabic and polyaniline was grown electrochemically using the cyclic voltammetry technique. The PAni/GA composite was tested in the presence of ammonia gas and its response was measured

using colorimeter-type equipment.

2. Methodology

2.1 Reagents

Aniline (99% purity grade), sulfuric acid (H_2SO_4 - 98%), and hydrochloric acid (HCl - 37%) were obtained from Sigma-Aldrich (St. Louis, USA). Potassium hydroxide (KOH), potassium ferricyanide ($K_3[Fe(CN)_6]$), and alumina (Al_2O_3) were acquired from Vetec. Milli-Q ultrapure water (18.2 OMΩ cm at 25 °C) (Millipore Corporation®) was used to prepare the solutions. Gum Arabica was acquired from JB Química Indústria e Comércio Ltda.

2.2 Aniline purification

Aniline was purified by fractional distillation at 60 °C and pressure of 50 mmHg. Then, 460 µL of purified aniline was added to 25 mL of 0.5 mol L^{-1} HCl and submitted to sonication in an ultrasonic bath for 10 min.

2.3 Preparation of gold electrodes

The conventional gold electrode was hand polished with alumina (0.3 µm particle size) for 10 min. Afterward, they were immersed in 96% ethanol for 5 min and then in Milli-Q water for 5 min with sonication in an ultrasonic bath at each step. Finally, the working electrode was submitted to electrochemical cleaning by cyclic voltammetry in the potential range of -0.8-2.0 V (vs. Ag/AgCl) during five scanning cycles, in the presence of 0.5 mol L^{-1} of H_2SO_4 and washed with Milli-Q water. The printed gold electrodes were subjected to differential pulse voltammetry (DPV) in the potential range of -1.3 to 0.1 V, with a pulse interval of 0.5 s, in the presence of 0.5 mol L^{-1} of KOH

2.4 Electrosynthesis of the PAni/GA composite

First, a 20% (w/v) GA solution was prepared and added with PAni to obtain a final 0.2 mol L^{-1}. Then, the solution was submitted to constant stirring for 5 min and was sonicated in an ultrasonic bath for 10 min. The PAni/GA electrosynthesis on the gold surfaces was performed using the potentiodynamic technique (cyclic voltammetry), applying a potential range of –0.2-1.4 V (up to 10 cycles). The stability of the system and optimization of the electrosynthesis process of the PAni/GA composite was performed through variations in pH (5.81 and 0.0) and scanning speed (10 and 50 mV s^{-1}). Electrochemical measurements were performed using an Autolab PGSTAT 12 potentiostat/galvanostat (Ecochemie, Netherlands) and Nova 2.1 software (Methroms®). A 25 mL electrochemical cell was assembled from: a) a conventional three-electrode system for the optimization and stability studies, with a crystalline gold disk electrode as working electrode ($\varnothing$ = 0.08 cm^2), an electrode helical platinum auxiliary and Ag/AgCl | KCl 3 mol L^{-1} as reference electrode, b) Printed gold electrodes from Dropsense - C220AT® (disposable electrodes), with an area of 0.5024 cm^2 were used for the colorimetry and SEM studies.

2.5 Ammonia Vapor Detection

A closed system in a capped glass flask, containing a cotton swab and electrodes with the composites was prepared. In the glass flasks, 1 mL of NH_4OH was added at different concentrations (50; 25; 12.5; 6.25; 3.125; 1.56; 0.78; 0.39; 0.19, and 0.09 mmol L^{-1}) on the cotton (0.200 g), closing the flask immediately to avoid loss of the analyte (Oliveira, Romero, Alves, Biswas, Cheng, & Furtado, 2019), the response was analyzed after 7 days. The change in color of the PAni film was evaluated with a digital colorimeter (Konica MINOLTA, model CR-400).

2.6 Color analysis

The L*, a*, and b* color parameters of the PAni/GA composites and their isolated polymers (GA and PAni) were measured using a digital colorimeter (Konica MINOLTA, model CR-400), calibrated with a white calibration plate. Following the CIE Lab system (Ji, et al., 2010), the L* parameter represents the luminosity of the sample ranging from zero (black) to one hundred (white), and a* indicates the colors on a scale from red to green (positive values tend to red and negatives tend to green) and b* represents the color on a scale from yellow to blue (positive values tend to yellow and negative values to blue). Color measurements of composites and isolated polymers were performed in two situations: before exposure to ammonia vapor and after exposure, is calculated by Equation 1:

$$\Delta E: \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}}$$

where, ΔL* is the luminosity difference between the samples, while Δa* and Δb* are the differences in red and yellow colors, respectively, between the samples. Color analyzes were performed under low lighting and white surface. The result obtained was the average of the tests performed in quintuplicates.

2.7 Scanning electron microscopy

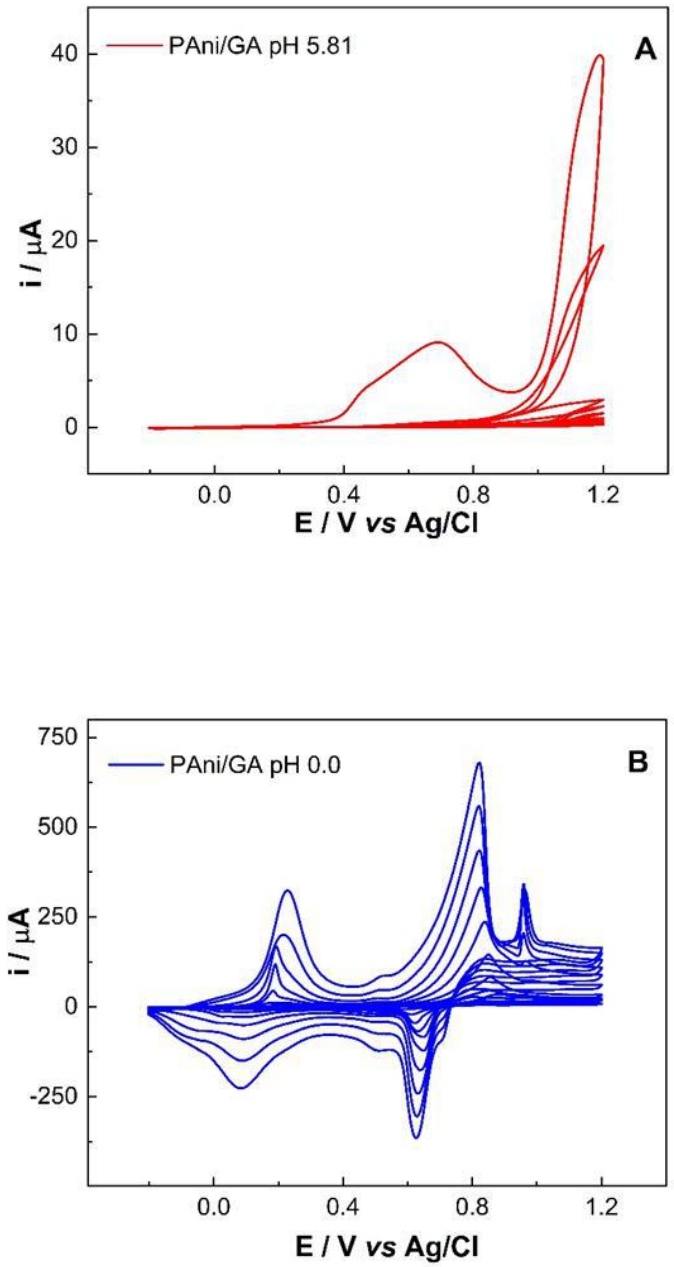
Micrographs were obtained using a scanning electron microscope (SEM; Quanta 450 FEG System: FEI Company, USA) with a scanning voltage of 15 kV. The samples were metalized with a thin gold layer by sputter (Emitech Model k550, from Quorum Technologies, UK).

3. Results and discussion

3.1 Optimization of film growth

First, an evaluation of the influence of two pH conditions on the process of composite synthesis using a conventional gold electrode was performed in this study (Figure 1).

Figure 1. Cyclic voltammogram of the electrochemical profile of the Pani/GA composite: (a) without pH adjustment (5.81) and (b) with pH adjustment (pH 0), 10th cycle at a potential window of -0.2 at 1.2 V, 10 mV s^{-1}.



Source: Authors.

Studies by Oliveira, (2020), Eiras, et al., (2007), and Hussain and Kumar, (2003) reported better conditions for the growth and polymerization of aniline in an acidic medium. A study was conducted with the addition of HCl (PA) in the PAni/GA solution to obtain a solution with pH 0 and pH 5.81 (without pH adjustment). In an acidic medium, the PAni/GA solution showed a decrease in viscosity, which facilitated the use of the 20% gum Arabic solution. Voltamograms showed that in the more acidic medium, a distinct voltammetric profile was observed, with an increase in current density, indicating a better electrochemical activity. This may have happened due to the possible increase in charge transport, with an increase in the molecular mass of the composite formed, as already reported in the work by Eiras et al., (2007) and Oliveira, (2020).

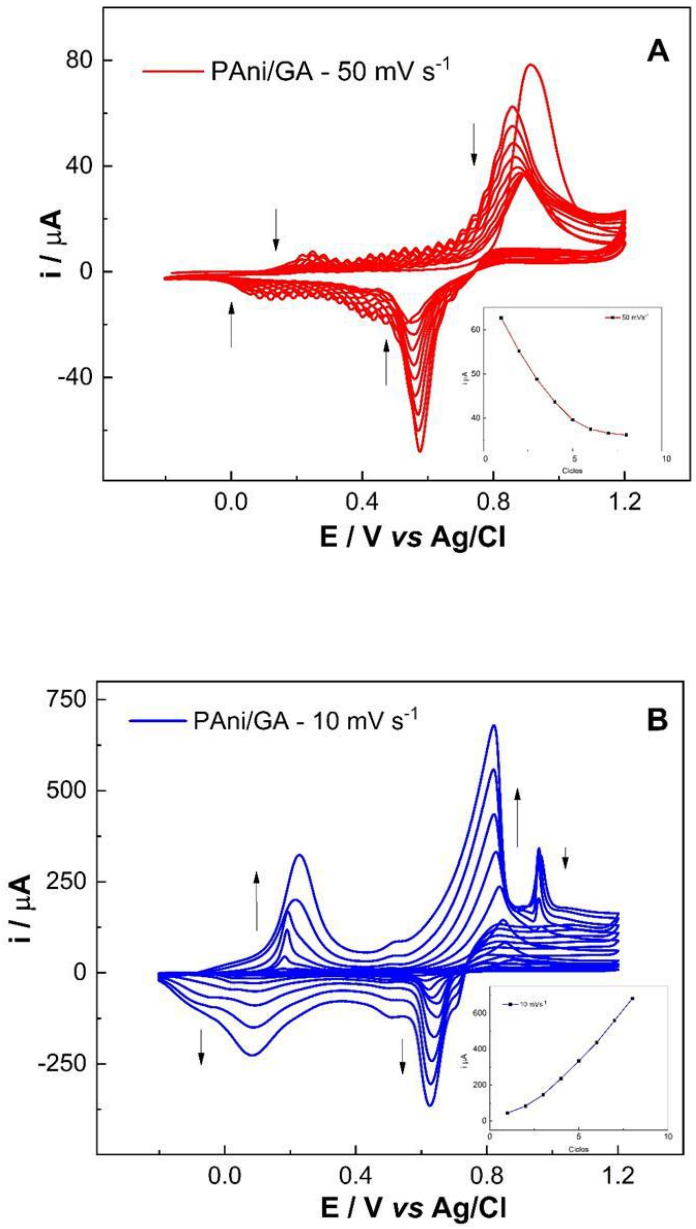
The electrosynthesis reaction of PAni/GA in a medium at pH 5.81 did not provide the composite formation. In Figure 1a, it can be seen that the formation of peaks characteristic of aniline oxidation and reduction did not occur, unlike in Figure 1b, consequently, there is no oxidation of the conductive polymer on the electrode surface, only a discrete deposition, possibly of GA. Also, the film (Figure 1a) was passive right after the first cycle. Studies such as the one by Stejskal and Gilbert, (2002) and Oliveira, (2020) demonstrated that the pH variation directly influences the oxidation of the conductive polymer, in a strongly acidic medium, mainly at pH < 2.5, where the polymer conductivity becomes above 10^{-3} S cm^{-1}. Experimentally, the increase in pH can promote a decrease in the oxidation and/or reduction of peak potential, and in the peak current, which is not desired (Mattoso, 1996).

The polymerization/growth process of the film is also influenced by the acid used. The chemical nature of the dopant not only affects electroactivity as well as the structural properties of the surface and weight. The choice of dopant becomes essential for synthesis, as it can modulate the electrical conductivity and structural properties of the surface (Guimard, et al, 2007; Veras, 2020). Guimard, et al, (2007) described those dopants could have different natures, examples of which would be salt ions, polymers including polysaccharides, proteins, and peptides. Chloride anions are most commonly used because of their good compatibility, thus reinforcing their application for composite development (Oliveira, 2020).

The speed of the electrosynthesis process significantly influences the development and final structure of the film. In Figure 2, two scan speeds (50 and 10 mV s^{-1}) were evaluated for the electropolymerization process. It was observed that at the speed of 10 mV s^{-1} (Oliveira, et al., 2009) there was the formation of a greenish film, totally differing when the scan speed was increased to 50 mV s^{-1} which did not show film formation (Oliveira, 2020).

When using the speed of 50 mV s^{-1} (Figure 2a) it was noticed the formation of a more resistive and non-conductive film. As much as there is an increase in the number of cycles, it is not possible to view the film, in addition to suffering a decrease in current density at each new cycle.

Figure 2. Cyclic voltammograms of the influence of velocity at (a) 50 mV s^{-1} and (b) 10 mV s^{-1} on the formation of the composite PAni 0.2 mol L^{-1} / GA 20% in 10 cycles. Insert refers to 0.85V potential.



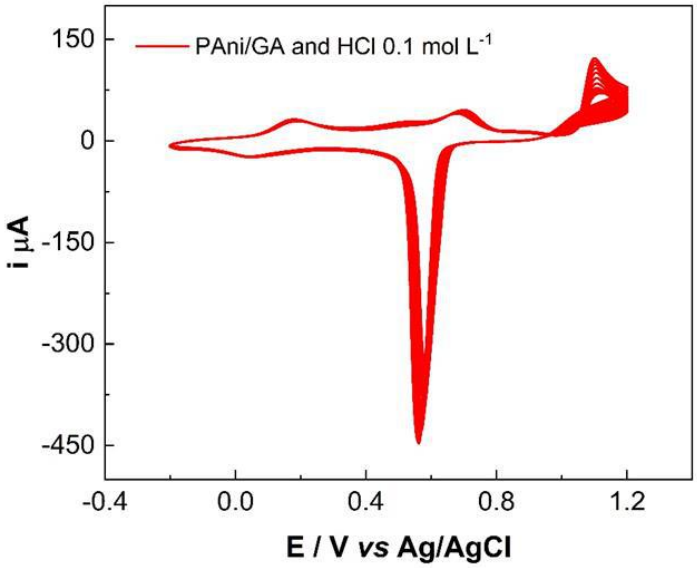
Source: Authors.

The difference in current peaks at the two scan speeds in this study is notorious. It is observed that in the composite formed with a slower speed, the formation of oxidation/reduction pairs at the potential of 0.2 and 0.06 V, respectively, is evident. These pairs are characteristic of the interconversion between the oxidation states of leukoesmeraldine (yellow) and emeraldine (green), and peak displacement may occur due to the solution being in a very acidic medium (Oliveira, 2020; Veras, 2020). The gradual increase in the amplitude of the redox peaks and the small displacement of the oxidation potential indicated the formation of a film with good conductive characteristics, a profile found in the composite formed at a lower speed. At a low speed, the process depended on the formation of the polymer/polymer on the electrode surface and not on the

transfer of electrons from the monomer, which generated a better profile using the speed of 10 mV s^{-1}.

After the formation of the composite film, the film was submitted to an acidic medium (HCl 0.1 mol L^{-1}) to assess the stability of the film after several consecutive scans. The successive scans of potential at 10 mV s^{-1} in the region of -0.2 - 1.2 V, showed that the current intensity of the anode and cathode peaks did not change significantly after 25 cycles, having a coefficient of variation of 15% indicating good stability (Figure 3).

Figure 3. Electrochemical stability of the composite as a function of the number of scans in an acidic medium (0.1 mol L^{-1} HCl) window from -0.2 to 1.2 V at 10 mV s^{-1}.



Source: Authors.

3.2 Ammonia Vapor Detection

PAni is already used as an indicator for the detection of ammonia vapors, as the emeraldine salt has a region that allows an adequate interaction so that the ammonia vapors establish a protonated coordination bond in the chain, through the nitrogen of both the compounds (Kukla, et al., 1996; Nicolas-Debarnot & Poncin-Epaillard, 2003; Tanguy, M. et al., 2018). The conversion of the PAni/GA emeraldine salt (green tint) to the PAni/GA emeraldine base is visually verified by the bluish tint change. The intensity of the color change depends on the ammonium hydroxide concentration (Tanguy, M. et al., 2018). The process of changing the sensor color suggests that the nitrogen atoms in the polymer chains act as adsorption centers for ammonia molecules (Tanguy, M. et al., 2018), after exposure to the composite. Thus, the PAni/GA films maintained the characteristics of the protonation and deprotonation process in the presence of nitrogen atoms.

This study was carried out using PAni/GA films grown on disposable gold electrodes, with a view to a future commercial application of the technology. The total color difference (ΔE) measured with a colorimeter was evaluated to visualize the behavior of the composite, mainly after exposure to ammonia vapor, where ΔEa is the variation of the film before exposure to steam using PAni as a control, ΔEd is the film variation after exposure to steam with the initial composite being the control. The results obtained from ΔEa = 0.38 and ΔEd = 0.93 demonstrated that ΔE had a gradual increase with color change that human eyes could hardly distinguish. The color change can be observed with the naked eye (untrained and experienced) when the value of ΔE is greater than 3 (Macedo, et al., 2021). According to (Tassanawat, et al., 2007) and Wang

et al., (2018), values of ΔE greater than 5.0 can be easily detected by human eyes, and ΔE greater than 12.0 may imply different color spaces. In this way, the sample did not show a noticeable change to the naked eye.

The color variation of the PAni/GA composite in the presence of ammonia gas was tested at different concentrations of the analyte (NH_4OH: 50; 25; 12.5; 6.25; 3.125; 1.56; 0.78; 0.39; 0.19, and 0.09 mmol L^{-1}), with the composite as control before exposure to steam. The results obtained can be seen in Table 1. It was observed that the device showed responses at all concentrations tested, with the lowest concentration of ammonia vapor tested having a color variation of 0.26, showing that at low concentrations, even if in a non-noticeable way without equipment, it is possible to have colorimetric responses with a device. It is important to note that the ΔE of the last applied concentration of 0.09 presents a reduction of almost 50% compared to the concentration of 0.19 mmol L^{-1}, demonstrating that the concentrations below this value are not as sensitive to ammonia vapor.

Table 1. Color variation of the ΔE of the PAni/GA composite grown at potential from -0.2 to 1.2 V, for 5 cycles at a speed of 10 mV s^{-1} in the presence of ammonium vapor at different concentrations

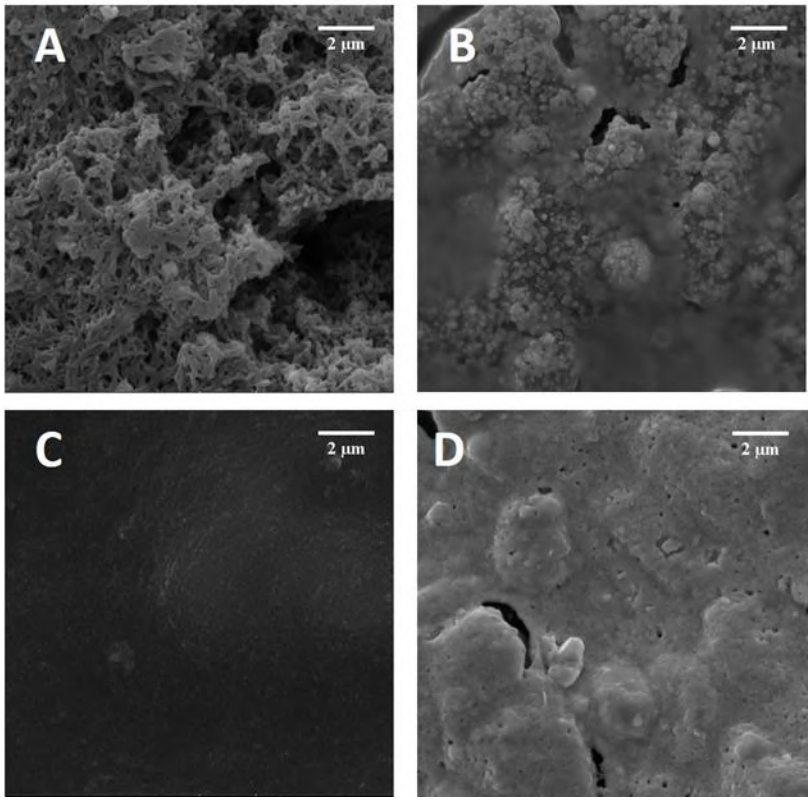
Concentrations of NH_4OH mmol L^{-1}	**ΔE**
50.00	0.93
25.00	0.68
12.50	0.53
6.25	0.58
3.12	0.52
1.56	0.42
0.78	0.53
0.39	0.50
0.19	0.45
0.09	0.26

Source: Authors.

3.3 Scanning Electron Microscopy (SEM)

In Figure 4 are the microscopic images of the PAni, PAni/GA, and GA film is grown by cyclic voltammetry, and the surface of the electrode is printed with clean gold (Au).

Figure 4. SEM images for films of (a) PAni, (b) GA, (c) PAni/GA, grown by cyclic voltammetry, and (d) gold printed electrode surface (Au).



Source: Authors.

The film formed only by PAni (Figure 4a) has large particles and an irregular surface, characteristics similar to those already described in the literature (Xu, et al.,2015). It is known that the electropolymerization conditions influence the morphology of the films obtained, interfering with the way the chains are arranged on the substrate (Mattos, 2016). The film formed by GA (Figure 4b) had a rough morphology, granules in its extension and greater uniformity than the film formed only by PAni.

The PAni/GA composite (Figure 4c) grown by cyclic voltammetry exhibited a compact morphology with small globules on the surface. This characteristic morphology of the composite can facilitate the entry of gases into these cavities (Oliveira, 2020), which can result in better trapping (adsorption) and the detection of vapors. The combination of PAni/GA through electrostatic bonds formed by the cyclic voltammetry process favored the synthesis of the composite in the electrode.

The interaction between polysaccharides and conducting polymers can enable better stabilization and good electrochemical performance, expanding the potential for applications (Zhou, et al., 2018; Petrov, et al., 2010; Shi, et al., 2013). For this reason, works like this are necessary, as they enable the interaction between PAni and other plant-derived polymers to form a conductive film with more compact areas.

4. Conclusion

Gum Arabic proved to be an efficient polyelectrolyte for the aniline electropolymerization process of aniline with good sensitivity for use as a colorimetric sensor. The optimization carried out in this study, regarding pH and film growth rate were essential for the synthesis of the composite with halochromic characteristics. Although the color change was not

noticeable to the naked eye, the use of a colorimeter was useful as a transducer for transforming the colorimetric signal into a measurable response. Thus, the work is promising for the application and commercial development of a colorimetric sensor for ammonia, in addition to being the first work described in the literature that produced a composite of gum Arabica and polyaniline by electrochemical means.

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Credit author statement

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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