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# EXPERIMENTAL STUDY OF THE EFFECT OF MECHANICAL AGITATION OF THE ELECTROLYTE SOLUTION ON THE FORMATION OF NANOSTRUCTURED COATINGS

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**ABSTRACT:** This study experimentally analyzes the influence of mechanical agitation of the electrolyte on the formation of nanostructured coatings obtained by electrodeposition of chromium nanoparticles onto the surface of AISI 1020 steel, considering that the hydrodynamic conditions of the electrolytic bath directly influence mass transport and the availability of the deposited material at the electrode–electrolyte interface. The study is based on the principles of mass transport and on the hydrodynamic characterization of the system using the Reynolds number, a parameter that allows the electrolyte flow regime to be related to the mechanisms of dispersion and transport of nanoparticles in suspension. For the experimental setup, an electrolyte containing chromium nanoparticles at a concentration of 100 ppm was prepared, and three mechanical agitation conditions corresponding to 30 rpm, 60 rpm, and 120 rpm were evaluated, while keeping the electroplating parameters constant. Initial observations of the hydrodynamic behavior showed that at 30 rpm, sedimentation of the feed material occurs due to insufficient dispersion of the nanoparticles in the electrolyte, while at 120 rpm, radial redistribution of the particles toward the vessel walls occurs due to increased hydrodynamic forces, reducing their availability in the deposition zone. In contrast, at 60 rpm, a homogeneous suspension of the feed material was obtained, favoring the transport of nanoparticles toward the electrode surface. The coatings obtained were characterized using scanning electron microscopy (SEM), showing that this condition produces homogeneous deposits of uniform thickness, while the conditions at 30 and 120 rpm generate uneven coatings. The results demonstrate that the hydrodynamics of the electrolyte

control mass transport and the availability of nanoparticles at the electrode–electrolyte interface, allowing the establishment of an optimal stirring condition for obtaining homogeneous nanostructured coatings and providing experimental evidence of the importance of controlling hydrodynamic conditions in nanoparticle electrodeposition processes.

**KEYWORDS:** electrodeposition, nanostructured coating, chromium nanoparticles, mass transport, electrolyte hydrodynamics.

## Introduction

At the industrial level, friction, wear, and corrosion are among the primary mechanisms that compromise the structural integrity of mechanical equipment and components, leading to premature failures and costly operational downtime (Toyo., 2023). In developed countries, it is estimated that corrosion alone accounts for economic losses of 3–5% of gross domestic product (GDP), according to (Shan et al., 2022), equivalent to billions of dollars annually; this does not include losses due to environmental impacts and risks to human life. This highlights the urgent need for technological alternatives aimed at extending the service life and protecting the surfaces of various metal substrates against the effects of corrosion, wear, and friction. Currently, to minimize the aforementioned negative effects, researchers and engineers are working on new technological alternatives, such as the development of new materials and improvements to existing ones, as well as new processing techniques and methods for using materials. In this context, nanotechnology has emerged as a viable and cost-effective option for the manufacture of

advanced surface coatings applicable in industrial, biomedical, chemical, and environmental sectors, promoting improvements in hardness, wear resistance, and corrosion resistance. Various nanomaterials have been tested for these purposes, with metal nanoparticles standing out as the most researched and widely used in this field.

However, as Zhang et al. (2020) note, the quality and performance of a surface coating depend, in equal measure, on both the feedstock material from which the coating is formed and the technique used for its deposition. Among the various available techniques, electrodeposition is a viable electrochemical process for the fabrication of nanoscale coatings, whose formation is governed by a set of operational parameters, such as current density, deposition time, pH, and the concentration of electroactive species in the electrolyte. Among these parameters, the hydrodynamic conditions of the electrolytic bath (BHD) are particularly relevant. BHD, commonly associated with the rate of mechanical agitation during the process, promotes (i) particle suspension and prevents sedimentation, (ii) forced convection and diffusive transport toward the electrode-electrolyte interface, and (iii) the thickness of the diffusive boundary layer, which controls the local availability of electroactive species and the kinetics of deposit nucleation and growth. Agitation can promote homogeneous interfacial conditions and uniform nucleation; however, it has been observed that increasing the agitation rate can lead to both an increase and a decrease in the content of incorporated particles, depending on the hydrodynamic regime (laminar or turbulent) and particle-particle and particle-surface interactions, which modify the electrocrystallization of

the coating due to the boundary layer effect and the supply of particles at the interface (Kerr et al., 2000).

Although various studies related to BHD have been conducted, there is still ambiguity regarding the effect of stirring speed on the incorporation of inert particles into the deposit. Some authors report that high stirring speeds generate hydrodynamic forces that hinder particle adhesion to the electrode surface, reducing their incorporation into the coating growth (Fransaer et al., 1992). However, other studies indicate that increasing the stirring speed promotes the dispersion and transport of particles toward the electrode surface (Low et al., 2006). Likewise, it has been reported that the effect of agitation on the electrode depends on the properties of the electrolyte, the particle size, and the conditions of the electrochemical process, and this influence can vary depending on the type of fluid or the application under consideration (Walsh, 2014). In this context, precise control of hydrodynamic conditions, such as mechanical agitation, is essential to achieve greater particle incorporation into the deposit and obtain homogeneous coatings with uniform dispersion

Therefore, this research focuses on the experimental study of the effect of mechanical agitation of the electrolytic solution as one of the fundamental hydrodynamic conditions or those with the greatest influence on the formation of nanostructured coatings; considering the effect of the distribution of the feedstock on the homogeneous formation of the deposit, to obtain useful agitation values for the electrodeposition process of nanostructured coatings based on chromium metal nanoparticles.

## Theoretical foundations

In electroplating processes, the formation of the metal coating depends not only on the electrochemical conditions of the system, but also on hydrodynamic phenomena present in the electrolyte. Fluid motion directly influences the transport of electroactive species, which determines the feed material during the growth of the metal deposit. In the experimental setup, motion is generated by magnetic stirring, which induces convection in the electrolyte, improving solution mixing and mass transport toward the electrode-electrolyte interface. For this reason, the analysis of mass transport and the hydrodynamic conditions of the electrolyte, characterized by the Reynolds number, is fundamental to understanding the formation of nanostructured coatings in electrodeposition processes.

### Mass transport

To determine the hydrodynamic conditions in aqueous fluids used in electrodeposition processes, the mass transport phenomenon that controls the availability of the deposited material at the electrolyte–electrode interface is analyzed. Coating growth depends on the continuous supply of electroactive species to the cathode surface, typically in the form of metal ions or inert nanoparticles in the electrolyte, which undergo electrochemical reduction to form the metal deposit (Bard et al., 2001). When the electrochemical reaction rate is high, the process may be limited by the mass transport of electroactive species from the electrolyte volume to the electrode surface. Under steady-state conditions, a limiting current is established, which represents the maximum current the system can sustain

when the concentration of the reactive species at the electrode surface tends to zero. In this regime, the deposition rate is related to the transport of  $M^{n+}$  ions toward the electrode (Texcucano et al., 2021).

The limiting current is described by the following equation:

$$I_L = \frac{nFAD}{\delta} C_{M^{n+}}^* \quad \text{Eq. 1.}$$

Where:

$I_L$  = limiting current (A).

$n$  = number of electrons transferred in the reaction.

$F$ : Faraday's constant, which is equal to 96.487 C

$A$ : electrode area.

$D$ : diffusion coefficient of species  $M^{N+}$ .

$\delta$ : thickness of the diffusion layer.

$C_{M^{n+}}^*$  : concentration of ions in the solution.

Equation (1) shows that the limiting current is directly related to the concentration of the feed material, the diffusion coefficient, and the thickness of the diffusion boundary layer ( $\delta$ ), representing the region near the electrode surface where species transport occurs primarily by diffusion (Landolt., 2007). However, the boundary layer is not a constant parameter but depends on the hydrodynamic conditions of the electrolyte; when the electrolyte is at rest, the diffusive boundary layer tends to be relatively thick, which impedes species transport toward the electrode surface. Conversely, when the electrolyte is subjected to mechanical agitation, convective fluid motion is generated, which reduces the thickness of the diffusive boundary layer; consequently, the flow of elec-

troactive species increases, favoring coating growth (Walsh et al., 1993). This behavior is expressed in simplified form as:

$$I_L \propto \frac{1}{\delta} \quad \text{Eq. 2.}$$

According to the above relationship, the decrease in the boundary current is inversely proportional to the thickness of the diffusive boundary layer and, therefore, leads to an increase in mass transport toward the electrode surface, favoring the growth of the electrodeposited coating.

These conditions directly influence mass transport. When the electrolyte is at rest, species transport occurs by diffusion, which generates concentration gradients and leads to a decrease in the feed material in the area where electrodeposition occurs. Therefore, mechanical agitation introduced as forced convection promotes mixing of the electrolyte and reduces the thickness of the diffusion boundary layer.

According to Eq. 1, a decrease in the thickness of the diffusion boundary layer increases mass transport toward the electrode surface and promotes coating growth.

In colloidal systems, the hydrodynamics of the electrolyte influence the dispersion and stability of the particles. Insufficient agitation promotes sedimentation or agglomeration of nanoparticles, reducing their availability in the deposition zone. By contrast, adequate agitation allows for the maintenance of a homogeneous distribution of the feed material within the solution volume (Guan and Zhang., 2024).

In conventional electrodeposition processes, mass transport and the hydrodynamic conditions of the electrolyte determine the supply of electroactive species to the

electrode surface, which controls the deposition rate and positively or negatively affects coating formation; low rates result in little deposition, while high rates lead to radial redistribution. In these agitation systems, at the macroscopic level, fluid convection is enhanced, the thickness of the diffusive boundary layer is reduced, and the transport of species toward the electrode is improved. However, when the process is aimed at forming nanostructured coatings, controlling these phenomena becomes more important, since small variations in mass transport and the hydrodynamic regime can alter the local availability of electroactive species and affect the nucleation and growth kinetics of the deposit. For this reason, the analysis of the electrolyte flow regime using hydrodynamic parameters such as the Reynolds number is fundamental for the formation of nanoscale coatings. However, when the electrolyte contains nanoparticles dispersed at low concentrations, the system can be treated as a colloidal suspension. For this type of electrolyte, when the particle concentration is relatively low, the associated mass is small compared to the total mass of the base fluid, so the hydrodynamic behavior of the system remains dominated by the properties of the electrolyte. Under these conditions, the suspension can be treated as a continuous fluid with effective properties, where parameters such as density and viscosity represent the overall behavior of the system (Hunter, 2001). Various studies have shown that the viscosity of a suspension increases slightly with the volume fraction of particles, allowing the system to be described using effective properties when the concentration is low (Einstein, A. 1906). Consequently, hydrodynamic models developed for fluids at the macroscopic scale can be used to analyze mass transport and the flow regime

in nanoparticle electrodeposition systems, considering that nanoparticles are transported by convection, diffusion, and dispersion mechanisms that govern the movement of the electrolyte.

### Characterization of the hydrodynamic regime.

To characterize the hydrodynamic behavior of the electrolyte, the Reynolds number is used, a dimensionless parameter that describes the ratio of inertial forces to viscous forces present in the flow of an (aqueous) fluid; it is defined as shown in Equation 3 (Mott., 2015).

$$Re = \frac{\rho N D^2}{\mu} \quad \text{Eq. 3.}$$

Where:

Re = Reynolds number.

$\rho$  = fluid density.

N = rotational speed.

D = diameter of the agitator.

$\mu$  = dynamic viscosity of the fluid (20°C–25°C).

This parameter allows the flow regime present in the system to be identified; see Table 1.

Reynolds number	Regime
Re < 2000	Laminar
2000 < Re < 4000	Transitional
Re > 4000	Turbulent

Table 1. Classification of flow regimes.

In electroplating processes, the flow regime directly affects mass transport toward the electrode. As the Reynolds number increases, the intensity of electrolyte mixing

increases, which reduces the thickness of the diffusive boundary layer and improves the transport of electroactive species toward the surface.

In this study, the Reynolds number analysis was used to estimate the hydrodynamic behavior at different stirring speeds employed in the electrodeposition process. The stirring speeds used in the experiments (30, 60, and 120 rpm) and the estimated Reynolds numbers for each value, with the aim of identifying the associated flow regime, are shown in Table 2.

Stirring speed (rpm)	Reynolds number	Stirring
30	722	Low transitional
60	1444	Transitional
120	2888	High transitional

Table 2. Estimation of the Reynolds number for different stirring speeds.

As can be seen, an increase in stirring speed leads to an increase in the Reynolds number, altering mass transport and the hydrodynamic regime of the electrolyte toward the electrode surface.

This behavior is of utmost importance in electrodeposition systems incorporating metallic nanoparticles, since hydrodynamics directly influences the dispersion, suspension, and availability of the feedstock in the deposition zone. In this context, analysis of the Reynolds number allows us to estimate the hydrodynamic behavior of the system and establish the agitation conditions that favor mass transport and the incorporation of nanoparticles during the formation of nanostructured coatings.

## Methodology

### Specimen Preparation

Specimen preparation was carried out using mechanical roughing and polishing operations to obtain a flat surface with a mirror-like finish and free of grease. For this purpose, specimens were fabricated from commercial AISI 1020 steel, measuring 1 cm in base width, 2 cm in height, and 0.3 cm in thickness. Once the specimens were prepared, the surface to be treated was polished; for this, 100-, 200-, and 600-grit metal sandpaper was used, applied to the surface in the specified order to achieve the required surface finish. The specimens were then polished at high speed with a cotton cloth and washed with acetone to remove any grease from the surface. Finally, the water curtain test was performed, and the specimens were left to air dry. The resulting specimens are shown in Figure 1.



Figure 1. Test specimens treated and stored for electrode

The test specimens were carefully stored in a Ziploc bag and set aside for experimental testing.

### Preparation of the electrolytic bath

To prepare the electrolytic solution, 1000 mL of deionized water was placed in a beaker of equal volume, Figure 2(a). The beaker was then tared, and 0.1 g of fine

chromium nanoparticle powder was added, Figure 2(b). Subsequently, the mixture was agitated in an ultrasonic bath for 5 minutes (Figure 2(c)) to break up the agglomerates of particles and achieve a homogeneous solution. The complete procedure is illustrated in Figure 2.

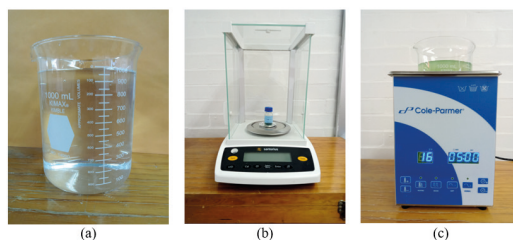


Figure 2. Sequence for preparing the electrolytic bath.

The result was an electrolytic solution, of an intense green color, at a concentration of 100 ppm.

### Preparation of the experimental setup for electrodeposition testing

A vibrating table with adjustable agitation speed and temperature was used as the base for the experimental setup (Figure 3(a)). The beaker containing the electrolytic solution was placed on top of it (Figure 3(b)). Subsequently, the working and sacrificial electrodes were mounted using a universal chemistry lab stand (Figure 3(c)). The physical arrangement of the electrodes placed the interacting surfaces parallel to each other, facing one another, as shown in Figure 3. The electrodes were connected to a regulated DC voltage source, Figure 3(d). The anode (sacrificial electrode) was connected to the positive terminal, and the cathode (working electrode) was connected to the negative terminal. The electrode assembly was immersed in the electrolytic solution such that the surface to be coated

was below the meniscus of the solution. The vibrating table was turned on, the agitation speed was adjusted, the electrolytic solution was allowed to homogenize due to agitation, and finally, the power supply was turned on to perform the electrodeposition.

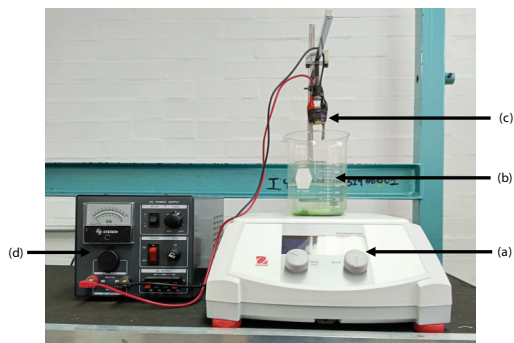


Figure 3. Experimental setup and physical arrangement of components.

## Experimental Procedure

Once the test specimens, the electrolytic bath, and the experimental setup were ready, the electrodeposition tests were conducted. For this purpose, the electrodeposition parameters shown in Table 3 were used.

Parameter	Magnitude	Unit
Voltage	12.5 – 17.5	V <sub>DC</sub>
Current	0.3	A
Concentration	100	ppm
Residence time	>15	min

Table 3. Main electrodeposition parameters used in the experimental tests.

The data in Table 3 correspond to the main electrodeposition parameters, as mentioned by Bedolla et al. (2022); as can be seen, solution agitation is not considered within these parameters; however, authors such as Tey et al. (2021) note that the formation of a nanoscale surface coating can be positively influenced by solution agita-

tion. Therefore, this parameter was included in the design of these experimental tests; so that the practical effect of mechanical agitation of the solution on the achievement of a homogeneous, nanostructured surface coating of constant thickness can be evaluated. The mechanical agitation values used in this research were taken from the models and simulations presented by Bedolla et al. (2025). Values of 30, 60, and 120 rpm were considered, and the behavior of the solution and the dispersion of the nanoparticles were visually verified. The findings are shown in Figure 4(a), (b), and (c), respectively.

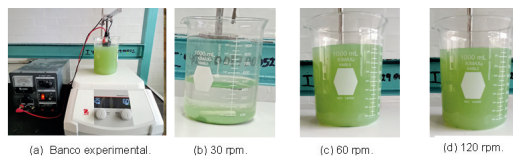


Figure 4. (a) Experimental setup and stirring of the electrolytic solution; (b) 30, (c) 60, and (d) 120 rpm.

As can be seen, at a speed of 30 rpm, after approximately 10 minutes, a sediment begins to form at the bottom of the beaker; this indicates agglomeration occurring in the feedstock, which had been broken up by the ultrasonic bath during the preparation of the electrolytic solution.

This demonstrates that agitation at 30 rpm is not sufficient to maintain the homogeneity of the solution concentration throughout the entire volume; consequently, in the area where electrodeposition is taking place, there is less feed material available, and the nanoparticles present are smaller than those that have settled. Therefore, it is concluded that a stirring speed of 30 rpm will not promote the formation of the nanostructured coating.

Meanwhile, for a stirring speed of the electrolytic solution of 60 rpm, a homogeneous color distribution of the solution and the absence of sediment at the bottom of the beaker can be observed; this allows us to infer that there is a homogeneous distribution of the filler material and a constant concentration of the electrolytic solution in the area where electrodeposition takes place, and it is expected that this value will promote the formation and homogeneous growth of the coating.

Finally, the solution was stirred at a speed of 120 rpm. At this speed, no sediment formation was observed at the bottom of the beaker either; however, the feed material—the chromium nanoparticles—moved toward the walls of the beaker. This was observed through the color change that occurred in the solution, with an intense green color near the walls and a lighter color toward the center. This is attributed to the turbulence formed in the center of the beaker due to the arrangement of the stirring element used, which causes this displacement of the feed material away from the zone where the electrodeposition takes place—the center of the beaker. This allowed us to determine that a stirring speed of 120 rpm is also not recommended for this electrodeposition process and the formation of the coating.

Based on the above, it was determined that 60 rpm would be the recommended speed for conducting the experimental electroplating tests, and this value was added as the fifth electroplating parameter.

## Results and Analysis

To verify the effect of mechanical agitation of the solution on coating formation, a series of three tests was conducted using an equal number of test tubes. The experimental electrodeposition tests were performed using the values indicated in Table 1. The electrodes were placed at mid-height in the electrolytic solution to position them in the area with the greatest homogeneity when the solution was agitated. Subsequently, the obtained samples were characterized using scanning electron microscopy (SEM).

Figure 5 shows the surface SEM micrographs obtained for the different stirring speeds evaluated.

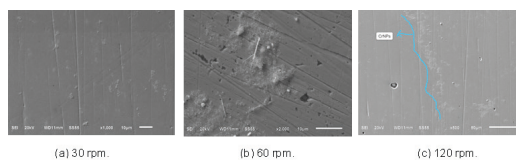


Figure 5. Surface SEM micrographs of the coating obtained at different stirring speeds: a) 30 rpm, b) 60 rpm, and c) 120 rpm. Scale bars: 10  $\mu\text{m}$  for 30 rpm and 60 rpm; 50  $\mu\text{m}$  for 120 rpm.

The results show that at a stirring speed of 30 rpm (Figure 5a), coating formation is sparse and exhibits low surface homogeneity. This behavior is associated with the precipitation of the deposit material at the bottom of the vessel, which reduces its availability in the zone where electrodeposition occurs. In contrast, for the 60 rpm condition (Figure 5b), a homogeneous coating of relatively uniform thickness is observed on the surface. This behavior is attributed to adequate dispersion and availability of the chromium nanoparticles in the electrolytic solution, which facilitates their transport to the electrode surface and maintains a more constant concentration of the deposit ma-

material in the deposition zone. On the other hand, when the speed was increased to 120 rpm (see Figure c), low coating uniformity was again observed.

From a hydrodynamic perspective, analysis of the estimated Reynolds number for the experimental conditions (Table 2) indicates that the system operated within a transitional flow regime, which progressively increases the mixing of the electrolyte with the stirring speed, directly influencing mass transport and the dispersion of the inert nanoparticles in the solution. At a speed of 30 rpm, the flow is insufficient to maintain a nanoparticle suspension, favoring sedimentation and agglomeration. At a speed of 60 rpm, the system reaches a hydrodynamic condition that balances particle dispersion and their stability in suspension, favoring their transport toward the surface of the electrode, and allowing for uniform deposit formation. In contrast, at a stirring speed of 120 rpm, the increase in hydrodynamic forces induces the radial redistribution of chromium nanoparticles toward the vessel walls, reducing their availability in the active deposition zone. This behavior indicates that the hydrodynamic conditions of the electrolyte control mass transport toward the electrode surface, which determines the availability of the deposit material at the electrode-electrolyte interface and, consequently, directly influences the morphology and uniformity of the resulting nanostructured coating.

Figure 6 shows the cross-sectional SEM micrograph of the coating obtained under optimal stirring conditions

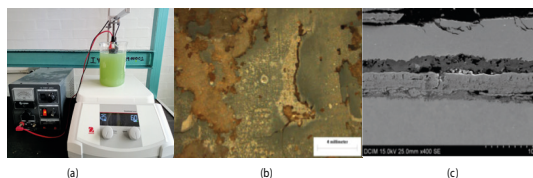


Figure 6. Cross-sectional SEM micrograph of the coating obtained at 60 rpm.

The results obtained are consistent with those reported by Low et al. (2006), who indicate that there is an opportunity for optimal hydrodynamic agitation in the electrodeposition process using suspended particles. Agitation improves mass transport and promotes the incorporation of particles into the coating; however, excessive agitation speeds can reduce incorporation efficiency due to increased hydrodynamic forces that remove particles from the electrode-electrolyte interface. The present study indicates that the optimal value corresponds to mechanical stirring at 60 rpm, a condition under which adequate suspension of nanoparticles and efficient transport of chromium nanoparticles ( $\text{Cr}^{3+}$ ) to the electrode surface are achieved, favoring the formation of homogeneous nanostructured coatings.

## Conclusions

Although the electrodeposition of composite and nanostructured coatings has been extensively studied, research experimentally analyzing the influence of the electrolyte's hydrodynamic conditions—particularly mechanical agitation—on the incorporation of nanoparticles into the deposit remains limited. In this context, the present work provides experimental evidence that contributes to the understanding of the effect of mechanical agitation of the electrolyte on nanostructured coatings. Agitation promotes fluid convection, redu-

ces the thickness of the diffusive boundary layer, and improves the transport of electroactive species toward the electrode surface, influencing the nucleation and growth kinetics of the deposit. The results obtained demonstrate that mechanical agitation of the electrolyte constitutes a determining hydrodynamic parameter in the electrodeposition process due to its influence on mass transport and the dispersion of nanoparticles in suspension. It was observed that low stirring speeds, such as 30 rpm, cause sedimentation of the feedstock, limiting the availability of nanoparticles at the electrode–electrolyte interface, while high speeds, such as 120 rpm, induce radial redistribution of particles toward the vessel walls, reducing their effective incorporation into the deposit. In contrast, an intermediate stirring speed of 60 rpm produces an adequate balance between particle dispersion, suspension stability, and mass transport toward the electrode surface, favoring the formation of homogeneous coatings of uniform thickness. The Reynolds number-based hydrodynamic analysis allowed the electrolyte flow regime to be correlated with the availability of the feed material, demonstrating the existence of an optimal hydrodynamic operating window for the formation of homogeneous nanostructured coatings. Consequently, this study contributes to the understanding of hydrodynamic conditions in nanoparticle electrodeposition processes and provides experimental criteria for the optimization of mechanical agitation in the fabrication of nanostructured coatings.

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