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Journal of Research and Development

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In the first chapter we present, *Nitride layers formed on austenitic stainless steel: Scratch and VDI appraisal* by García-Chávez, Jorge Alan, Melo-Máximo, Lizbeth, Solis-Romero, José and Vega-Morón, Roberto Carlos, with adscription in the Instituto Tecnológico de Tlalnepantla, as the next article we present, *Early Harvest in Maize Lines: Correlation between physiological maturity and seed germination* by Sánchez-Nuño, José Alberto, Sánchez-Martínez, José, Avendaño-López, Adriana Natividad and Padilla-García, José Miguel, with adscription in the Universidad de Guadalajara, as the next article we present, *Causal model of the dynamics of free-roaming dogs in Huatusco, Veracruz: A mixed-methods analysis based on territorial and social evidence* by Hernández-Armenta, Abigail, Córdoba-Gómez, Luis Tomás, Solís-Jiménez, Miguel Ángel and Calderón-Palomares, Luis Antonio, with adscription in the Instituto Tecnológico Superior de Huatusco, as the next article we present, *Analytical platform for operational performance assessment using Random Forest and K-Means* by Vázquez-De los Santos, Laura Cristina, Burciaga-Alarcón, Ricardo, Rodríguez-Silva, Jesús Rolando and Rodríguez-Arzola, Adrián, with adscription in the Universidad Autónoma de Coahuila, as the next article we present, *Physicochemical characterization of goat whey from different cheese processes* by Mandujano-González, Virginia, Álvarez-Cervante, Jorge, Morales-Aguilar, Santiago and Alonso-Segura, Diana, with adscription in the Universidad Tecnológica de Corregidora and Universidad Politécnica de Pachuca, as the next article we present, *Mycosynthesis and Characterization of Silver Nanoparticles from Phytophthora infestans for Antimicrobial Evaluation Purposes* by Granados-Olvera, Jorge Alberto, Vergara-Reyes, Hugo Yuriel, Rangel-Ruíz, Karelia Liliana and Del Angel-Francisco, Lesly Alabel, with adscription in the Universidad Politécnica de Cuautitlán Izcalli, as the last article we present, *Obtaining and characterization of terephthalic acid via acid and basic hydrolysis of recycled poly (ethylene terephthalate)* by De la Cruz, Boris, Domínguez, Ricardo and Moreno, Juan, with adscription in the Universidad Salvadoreña Alberto Masferrer.

Content

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Article

Nitride layers formed on austenitic stainless steel: Scratch and VDI appraisal

Capas de nitruros formadas sobre un acero inoxidable austenítico: Evaluación por rasgado y VDI

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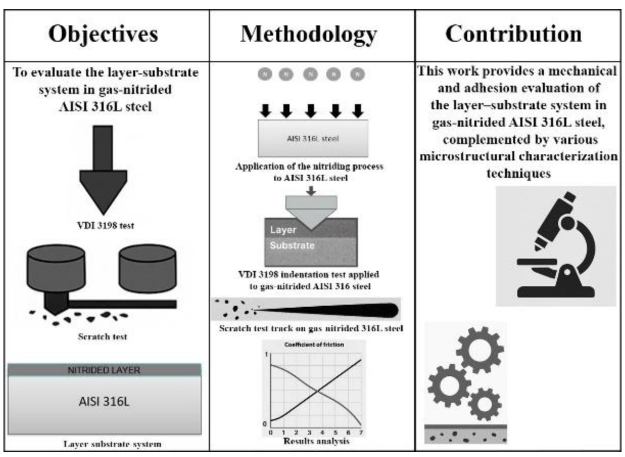
Abstract

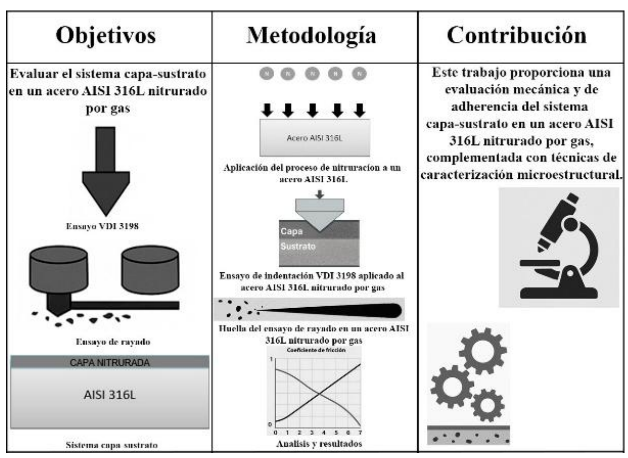
This study appraised the adhesion of the layers developed on AISI 316L austenitic stainless subjected to gas nitriding at 560 °C during 24 and 72 h of exposure time. The resulting nitrided layers formed at the surface of the AISI 316L steel presented a thickness of 60.5 µm for 24 h, and 79.5 µm for 72 h. Furthermore, by applying the VDI 3198 indentation tests along with scratch testing operating in progressive load mode, the nitride layers integrity was examined. Whether 24 or 72 hours of exposure time, the layers exhibited an adhesion classified as acceptable, according to the chart disclosed in the standard, presenting radial cracks without significant spallations in both layers. Finally, only cohesive damage was produced by scratch testing, the corresponding critical loads in 24 and 72 h were determined, associated with tensile cracks, lateral spallation and angular cracks.



Nitriding, scratch, adhesion

Resumen

Este estudio evaluó la adhesión de las capas desarrolladas en acero inoxidable austenítico AISI 316L sometido a nitruración gaseosa a 560 °C durante 24 y 72 h. Las capas formadas en la superficie del acero AISI 316L presentaron espesores de 60.5 µm para 24 h y 79.5 µm para 72 h. Además, mediante la aplicación de ensayos de indentación según la norma VDI 3198, junto con ensayos de rasgado en modo de carga progresiva, se examinó la integridad de las capas nitruradas. Tanto para 24 y 72 horas de exposición, las capas mostraron una adhesión clasificada como aceptable, presentando grietas radiales sin desprendimientos significativos en ambos casos. Finalmente, en los ensayos de rasgado únicamente se generaron fallas cohesivas y se determinaron las cargas críticas correspondientes para 24 y 72 h, asociadas a grietas de tensión, desprendimiento lateral y grietas angulares.



Nitrurado, rasgado, adhesión

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Introduction

AISI 316L steel belongs to the family of austenitic steels and is used in a wide variety of industries, such as aerospace, automotive, marine, and medical. Among its remarkable properties are its high corrosion resistance and biocompatibility with the human body, which make it a valuable material for engineering applications. However, despite these attractive properties, it presents an important limitation for long-term applications, since it possesses low mechanical and tribological properties (D'Andrea, 2023; Rodríguez-Castro et al., 2016)

There are different ways to address this problem through surface modifications; one of them is thermochemical treatments, which allow improvement of the tribological properties of steels (Mertgenç, 2025). One of these thermochemical treatments is boriding, carried out at high temperatures that enable the diffusion of boron atoms into the base material. When applied to steels, a layer of iron borides is produced with a hardness that can exceed 20 GPa, resulting in improved wear resistance (Garcia-Bustos et al., 2013). Another thermochemical treatment is nitriding, in which nitrogen atoms diffuse into the substrate. This can be performed through different methods, one of the most common being gas nitriding. This process is well known for surface hardening and can be applied to austenitic steels to increase surface hardness and improve wear resistance. Among its advantages are its low cost and the ability to be applied to parts of various sizes on a large scale (Shuaeib & Benyounis, 2016)

On the other hand, when a material is coated or subjected to a thermochemical treatment, it is essential that there is good adhesion between the coating or layer and the substrate, since this will determine whether the process is functional or not. Adhesion in a layer or coating is a key factor in ensuring coating quality; there are several methods to evaluate adhesion in engineering coatings, and the scratch test is one of the most common and effective methods, as it allows a semi-quantitative evaluation (Mittal, 2001). This method consists of producing controlled damage on the coating surface using a diamond counterpart, generally a Rockwell C indenter (with a tip radius of 200 µm), which travels across the surface applying a constant or progressive load.

In addition, another way to determine adhesion in a material is through a quick and reliable test called the Rockwell C indentation test, which allows a qualitative evaluation of the coating. This test is described in the VDI 3198 standard; it is a destructive test of coating quality, consisting of indenting the surface of the material with a conical diamond tip, causing plastic deformation of the material and fracture of the layer. The volume of the failure zone allows the adhesion of the layer to be evaluated through optical microscopy inspection, although scanning electron microscopy can also be used (Vidakis et al., 2003).

According to the above, there are some studies in the literature on adhesion and stainless steels subjected to thermochemical treatments. For example, Rodríguez et al., (2016) studied the borided layer of an AISI 304 steel using Rockwell C indentation and scratch tests. The authors concluded that despite having critical failures in the FeB phase, the borided layer reduces scratch resistance. They also highlighted that the exposure time during the boriding process is an important factor, since at 6 hours better results were obtained than at 2 hours.

On the other hand, Manfrinato et al., (2022) performed scratch tests on nitrided and nitrocarburized AISI 321 steel, both treatments carried out for 6 hours at temperatures of 400 and 500 °C. The results showed that at 500 °C a higher residual depth and a more irregular coefficient of friction (COF) were obtained, while the best results were obtained in the nitrocarburized sample at 400 °C, which exhibited better integrity after the test.

Likewise, regarding AISI 316L steel, Yildiz & Alsaran, (2010) performed multipass scratch tests on AISI 316L steel and Ti6Al4V alloy to study the coefficient of friction and compare the results between both samples after being subjected to plasma nitriding. The temperatures applied were 400 and 450 °C for the steel and 700 and 750 °C for the Ti6Al4V alloy. According to the results, the nitrided layer of AISI 316L steel proved to be unstable due to the significant presence of CrN and Fe_4N, causing fractures in the contact area. In contrast, the Ti6Al4V alloy showed greater stability in the sample treated at 750 °C, achieving a lower and more stable coefficient of friction.

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Therefore, although the literature reports work related to surface modifications through thermochemical treatments applied to austenitic stainless steels, there is limited information regarding the qualitative and semi-quantitative adhesion of AISI 316L steel subjected to gas nitriding treatment. Hence, the objective of this work is the evaluation of adhesion, through scratch and VDI 3198 tests, of the layer obtained by gas nitriding under two treatment conditions.

Methodology

For this study, the specimens were prepared from an AISI 316L stainless steel bar with a diameter of 25.4 mm, which was sectioned into 7-mm-thick disks. The samples were progressively ground to achieve a mirror-like surface finish, suitable for the subsequent thermochemical nitriding process. Prior to nitriding, the steel surfaces were subjected to a chemical pickling treatment.

After, the gas nitriding process was performed at a temperature of 560 °C and two exposure times: 24 h, corresponding to a single cycle (named 1C hereinafter), and 72 h, corresponding to three cycles (identified 3C hereinafter).

Next, a conventional metallographic process of the nitrided AISI 316L samples was conducted. The samples were cross-sectioned, mounted and ground sequentially with emery papers, from 120 to 1000 grit. The mirror finish was achieved by polishing with 0.25 µm diamond paste. The chemical etching was performed with Marble's reagent (HCl, $CuSO_4$, H_2O). The nitrided layer formed on each specimen was observed through optical microscopy, and its thickness was measured using ImagePro Plus software. Next, X-ray diffraction tests (XRD) were conducted (D8 Advance, Bruker), using Cu-Kα (λ = 0.154 nm) radiation, and a 2θ from 30 to 80°.

Later, the first adhesion assessment method, VDI indentation test, was conducted using a DISITEC DIS-DUR hardness tester, applying a load of 150 kgf with a Rockwell C diamond indenter, oriented perpendicular to the nitrided surfaces. The indentation imprints were examined by scanning electron microscopy and compared with the chart disclosed in the VDI 3198 procedure.

Additionally, the cross-sectional residual depth profile of each indentation was acquired by optical profilometry (GT-K Contour 3D, Bruker). Finally, progressive-load scratch tests (Revetest Xpress+, CSM Instruments) were conducted to investigate the practical adhesion resistance of the nitride layers. The tests employed an initial load of 5 N and a final load of 150 N, with a scratch length of 7 mm at a speed of 1.21 mm·min^{-1}, using a Rockwell C diamond indenter with a 200 µm tip radius. Three repetitions were performed for each sample. The resulting scratch tracks were analyzed using optical microscopy, scanning electron microscopy (SEM), and optical profilometry.

Results

The nitrided layers formed on the surface of AISI 316L steel subjected to the gas nitriding treatment are showed in Figure 1. The thicknesses were obtained through optical microscope, obtaining 60.5 µm for the process of to 24 h (1C), and 79.5 µm in the 72 h (3C) treatment. After chemical etching, the layer presented a dark tonality, which revealed the precipitation of the CrN phase.

Box 1

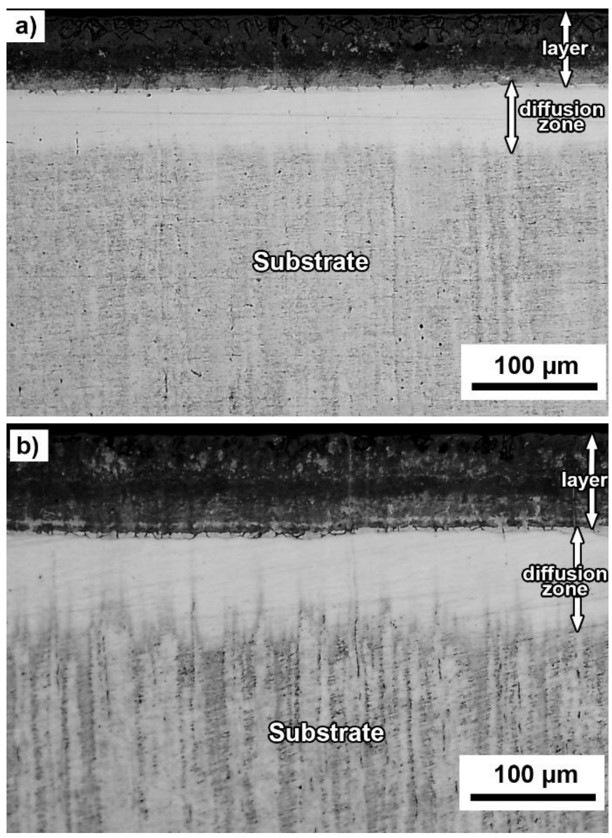

Figure 1
Cross-section micrographs of the AISI 316L subjected to nitriding: a) 24 h (1C); b) 72 h (3C).

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The results allow to establish that nitriding is a diffusion-controlled process; since longer exposure times resulted in higher thicknesses. However, it has been reported that when the CrN phase precipitates, other properties, such as corrosion resistance, might be compromised.

In Figure 2 is presented the XRD spectra obtained for 1C and 3C layers. It can be seen that iron nitrides Fe_3N and Fe_4N were identified, along with some oxides associated to the process (Fe_2O_3 and Cr_2O_3), but more noticeable, the precipitation of CrN phase occurred, as suggested by the dark tonality seen in the metallographic process. Although this phase presents a high hardness, it also can compromise the corrosion properties of the steel, since the available Cr used to produce and regenerate the passivation layer, is used to form this compound (Larisch et al., 1999).

Box 2

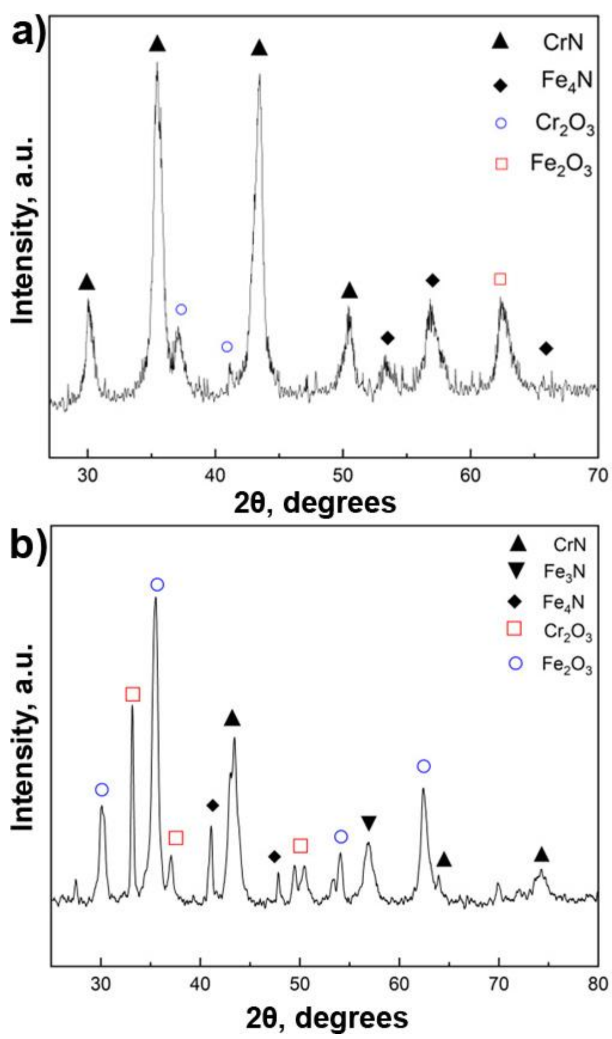

Figure 2
X-ray diffraction patterns: a) 24 h (1C); b) 72 h (3C).

In the Fig. 3 is presented the damage classification chart disclosed in the VDI 3198 adhesion test standard. The imprints obtained in the AISI 316L steel samples subjected to gas nitriding were compared with the chart, and after a visual inspection by optical microscopy, the damage was classified as acceptable or not acceptable (from HF1 to HF6), providing then a qualitative evaluation of the adhesion of the layers.

Box 3

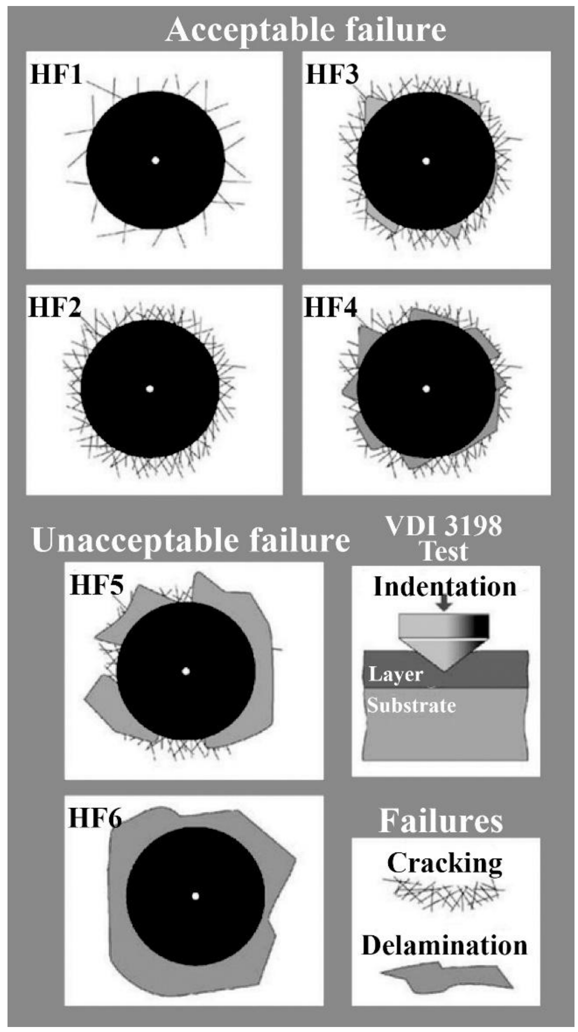

Figure 3
VDI 3198 failure chart.

A representative imprint obtained by SEM at the surface of the 1C sample is presented in Figure 4, along with details of the failures and their cross-sectional profile obtained by optical profilometry. As can be seen, only ring-shaped cracking occurred on the layer. Although a considerable number of cracks were registered, no partial or full delamination of the layer was detected.

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Therefore, for this 1C layer, an acceptable adhesion was determined. From the residual depth profile, it can be seen that the deformation exceeded the layer thickness, however this indicates remarkable adhesion properties, since only cohesive failures occurred; the release of the deformation energy occurred by cracking rather than detaching the layer.

Box 4

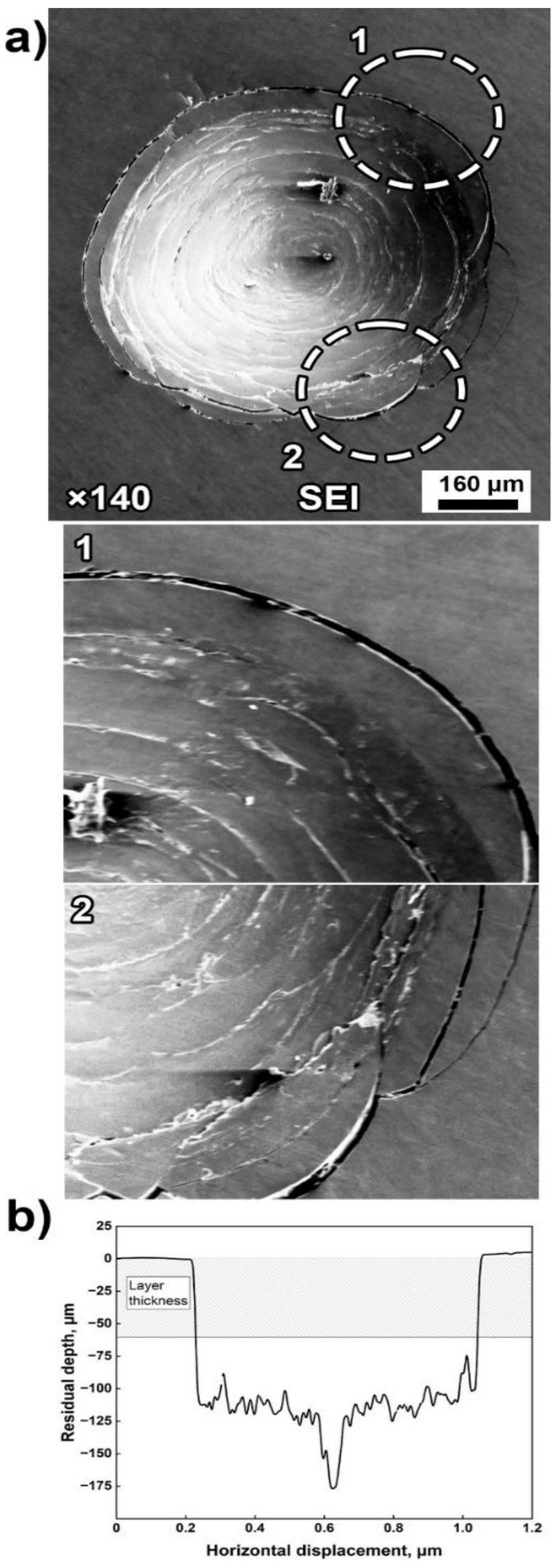

Figure 4
VDI results of 1C sample a) SEM micrographs; b) residual depth profile.

Moreover, in the Figure 5 is presented a representative indentation imprint of the 3C layer and its corresponding failures and cross-sectional profile. Ring-shaped cracking was again observed, although more severe than in 1C layer. However, these cracks did not produced delamination of the layer. Since no significant detachment was observed around the indentation edges, the adhesion was qualitatively considered as acceptable. Further, the indentation depth exceeded the layer thickness; however, severe delamination did not occur. Comparing both layers, the integrity of the 3C nitrided layer was more impaired than 1C, based on the number and size of cracks. Literature reports indicate that although the CrN phase can reach high hardness values, it is associated with more brittle behavior.

Box 5

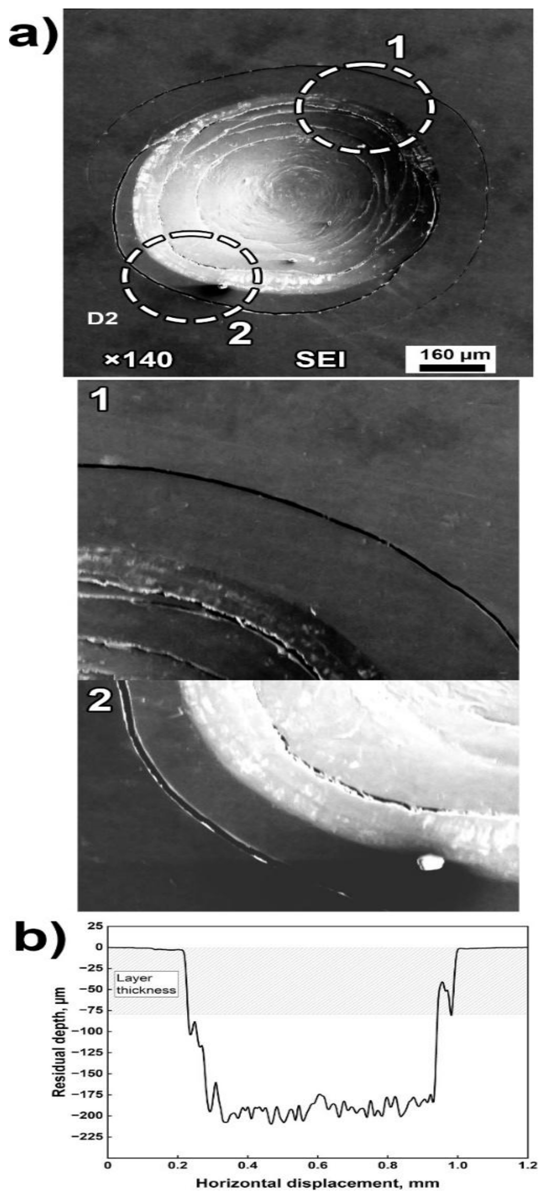

Figure 5
VDI results of 3C sample a) SEM micrographs; b) residual depth profile

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Representative scratch tracks obtained from the samples are presented in Figure 6a, along with the corresponding coefficient of friction and residual depth profiles (Figure 6b). For the 1C sample, an attenuated coefficient of friction (COF) behavior was observed, with no severe fluctuations. Some small variations appeared around 3.5 mm of displacement, but its amplitude is limited.

The COF fluctuations during a scratch test are associated with failures occurring within the layer. The final coefficient of friction value was around 0.33. From the residual depth profile can be concluded that under experimental conditions employed, the indenter finished within the layer thickness. Although the scratch test did not completely exceed the layer, it still allows to evaluate the practical adhesion and the types of failure that occur under applied loading conditions. On the other hand, for the 3C layer, a final coefficient of friction of 0.35 was recorded. In this layer, the attenuated behavior can be associated with the absence of severe spallations or detachments of the layer.

Box 6

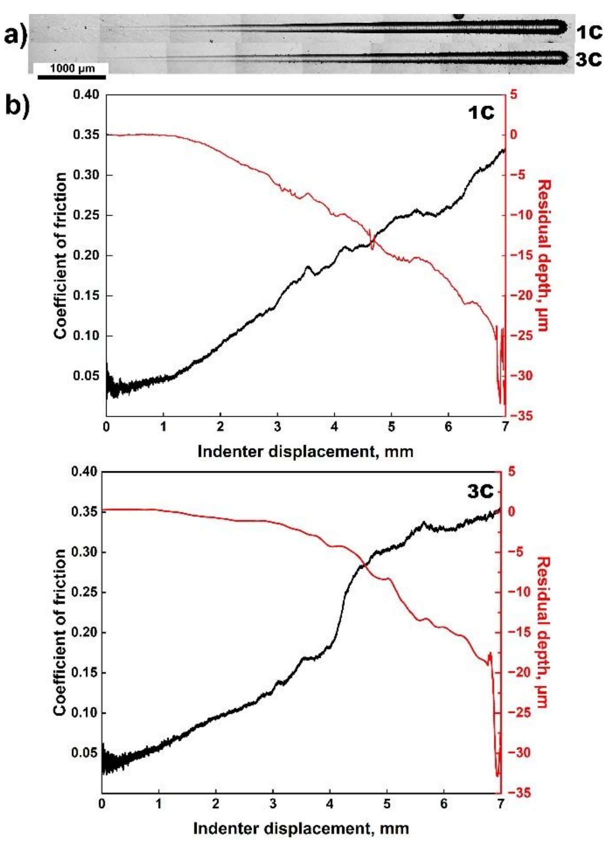

Figure 6

a) Representative scratch tracks; b) COF and residual depth behavior

Subsequently, the critical loads were calculated, according to the expression disclosed in the ASTM C1624 standard:

$$L_{c_n} = \left(\frac{l_n}{X_n} \cdot L_r\right) + L_i \quad (1)$$

where l_n is the critical distance (mm), or the distance where the particular failure appears; X_n is the linear speed ($mm \cdot min^{-1}$) of the indenter; L_r is the load rate ($N \cdot min^{-1}$), and L_i is the initial load (N). The Table 1 presents the critical loads for both layers.

Box 7

Table 1

Critical loads

Failure mechanism	Nitride layer	
	1C	3C
Tensile cracks	70±4 N	83.4±0.4 N
Chipping	95±5 N	66±2 N
Angular cracks	102±5 N	--------

It can be observed that although the tensile cracking increased its critical load with treatment time, the lateral spalling occurred earlier for 3C. This may be associated with the fact that 3C could better absorb deformation imposed by the indenter and also having a greater thickness. Figure 7 shows details of the scratch tracks obtained by SEM. The tensile cracks are formed behind the indenter during displacement movement, whereas the angular cracks occur due to high stress concentration behind the indenter and at the edge of the scratch track. Finally, chipping is the response of the layer–substrate system to release stored energy caused by compressive stresses ahead of the indenter (Bull, 1997).

Box 8

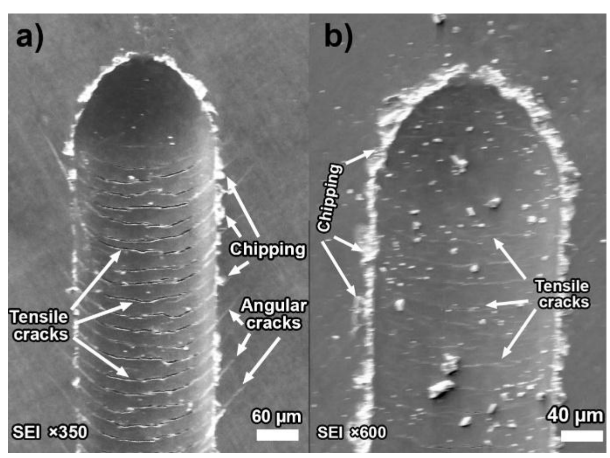

Figure 7

SEM micrograph of the scratch track a) 1C layer; b) 3C layer

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Conclusions

This work presented the adhesion appraisal of the nitride layers formed on the surface of AISI 316L austenitic stainless-steel formed by gas nitriding. The treatments were performed at a temperature of 560 °C with two exposure times (24 and 72 h), obtaining thicknesses of 60.5 and 79.5 µm, respectively. Through VDI 3198 tests, a qualitative adhesion examination was conducted, obtaining failures that were classified as acceptable, since no full delamination of the layer occurred. Furthermore, the practical adhesion was studied by the scratch test, where only cohesive-type failures were observed, i.e. along the layer thickness. Although a good integrity was observed after both adhesion tests, the formation of CrN compromise other properties, therefore this treatment under the experimental setup employed should be carefully driven according to the application required.

Conflict of interest

The authors declare no interest conflict. They have no known competing financial interests or personal relationships that could have appeared to influence the article reported in this article.

Author Contribution

García Chávez Jorge Alan: Adhesion tests, data formatting and writing original draft

Melo Máximo Lizbeth: Microstructural characterization (SEM and XRD), formal analysis, data curation

Solis Romero José: Nitriding treatment supervision, formal analysis, methodology

Vega Morón Roberto Carlos: Project idea, resources, formal analysis, data curation and writing, review & editing of the manuscript.

Availability of data and materials

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Abbreviations

AISI	American Iron and Steel Institute
COF	Coefficient of Friction
SEM	Scanning Elctronic Microscope
VDI	Verein Deutscher Ingenieure
XRD	X-ray Diffraction

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Antecedents

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Article

Early Harvest in Maize Lines: Correlation between physiological maturity and seed germination

Cosecha temprana en variedades de maíz: Correlación entre la madurez fisiológica y la germinación de las semillas

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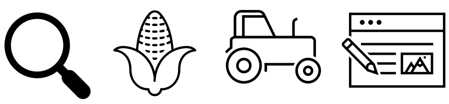
Abstract

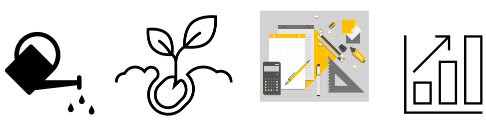
The objective of this study was to determine the relationship between physiological maturity and germination capacity of maize (Zea mays L.) lines in order to identify an early harvest stage that enables the production of viable seed for multiplication plots. The female parental line 14, progenitor of the single-cross hybrid JS-341, was evaluated across five distinct harvest dates, ranging from the R3 (milk stage) to the R6 (physiological maturity) phenological stages. Ears were air-dried until reaching 14% moisture, after which the standard germination test was applied. The experimental design was completely randomized, and mean comparisons were performed using Tukey's test ($p \leq 0.1$). Results showed significant differences among stages, with the highest germination percentages recorded at R5 and R6 (99.5–100%). However, early harvest at the R3 stage, when ears were kept with husks, maintained acceptable germination, representing a useful alternative for genetic rescue and the multiplication of parental lines.

Objectives



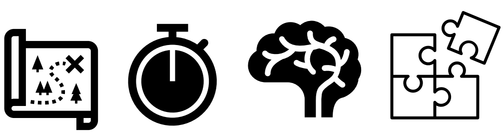
To determine the relationship between physiological maturity and germination in line 14.

Methodology



Harvest at stages R3–R6, natural drying to 14% moisture content, standard germination test, DCA, and Tukey's test.

Contribution



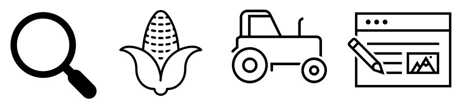
R5–R6 stages showed 99.5–100% germination; R3 kernels with husk preserved viability for genetic rescue and early multiplication.

***Zea mays L.*, physiological quality, phenology**

Resumen

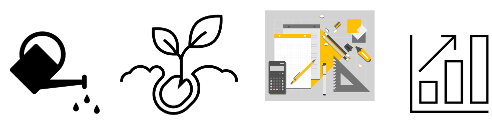
El objetivo de este estudio fue determinar la relación entre la madurez fisiológica y la capacidad de germinación de líneas de maíz (Zea mays L.) con el fin de identificar una etapa temprana de cosecha que permita la producción de semillas viables para parcelas de multiplicación. La línea parental femenina 14, progenitora del híbrido de cruce simple JS-341, se evaluó en cinco fechas de cosecha distintas, que abarcaban desde la fase fenológica R3 (fase lechosa) hasta la fase R6 (madurez fisiológica). Las mazorcas se secaron al aire hasta alcanzar un 14 % de humedad, tras lo cual se aplicó la prueba de germinación estándar. El diseño experimental fue completamente aleatorio y las comparaciones de medias se realizaron mediante la prueba de Tukey ($p \leq 0,1$). Los resultados mostraron diferencias significativas entre las etapas, con los porcentajes de germinación más altos registrados en R5 y R6 (99,5-100 %). Sin embargo, la cosecha temprana en la etapa R3, cuando las mazorcas se mantuvieron con las hojas, mantuvo una germinación aceptable, lo que representa una alternativa útil para el rescate genético y la multiplicación de líneas parentales.

Objetivos



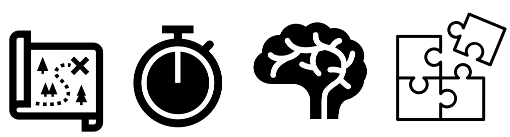
Determinar la relación entre madurez fisiológica y germinación en línea 14.

Metodología



Cosecha R3 – R6, secado natural 14%H. germinación estándar, DCA y Tukey.

Contribución



R5-R6 con 99.5 – 100% germinación: R3 con totomoxtle conserva viabilidad para rescate genético y multiplicación temprana.

***Zea mays* L., calidad fisiológica, fenología**

Area: Strengthening the scientific community

Citation: Sánchez-Nuño, José Alberto, Sánchez-Martínez, José, Avendaño-López, Adriana Natividad and Padilla-García, José Miguel. [2025]. Early Harvest in Maize Lines: Correlation between physiological maturity and seed germination. Journal of Research and Development. 11[26] 1-7: e21126107.

Introduction

Maize (*Zea mays* L.) is one of the most important crops worldwide due to its nutritional, industrial, and genetic value. The production of high-quality seed is a critical component of seed production programs, as it determines the uniform establishment of fields and the yield potential of the hybrid. Physiological maturity represents the stage at which the seed reaches its maximum dry weight and highest viability. However, in agricultural practice, adverse climatic conditions, pest pressure, or limited infrastructure often force producers to harvest earlier, before seeds reach full maturity. Moreover, harvesting prior to full harvest maturity may provide advantages for seed quality, as seeds are exposed for a shorter period to abrupt temperature fluctuations and to potential damage from pests and diseases during the maturation-to-harvest interval.

In this context, it is essential to understand how the developmental stage influences the physiological quality of seeds, particularly in parental lines used for hybrid production. Understanding this relationship allows for the establishment of early-harvest criteria that ensure viable seed while reducing field time and minimizing losses caused by environmental factors.

The purpose of the present study is to determine the relationship between physiological maturity and germination in maize lines, evaluating different phenological stages (R3 to R6).

Methodology

The experiment was conducted in the experimental fields of the Centro Universitario de Ciencias Biológicas y Agropecuarias (CUCBA) at the University of Guadalajara, during the agricultural cycle from June to November 2024.

Plant Material

Maize line 14 (*Zea mays* L.), the female parent of the single-cross hybrid JS-341 and part of the CUCBA maize breeding program, was used in this study.

Experimental Design

The objective was to evaluate the relationship between physiological maturity and germination through five harvest dates representing different grain developmental stages. Harvests were conducted on the following dates and days after sowing (DAS) (Table 1). The experimental design was completely randomized, and mean comparisons were performed using Tukey's test ($p \leq 0.1$).

Box 1

Table 1

Ear Harvest Dates

Harvest	Date	Days After Sowing (DAS)	Phenological Stage	Ear Condition
1	October 10	110 DAS	R3 (milk stage)	10 ears husked (without bracts)
2	October 10	110 DAS	R3 (milk stage)	10 ears with husk (with bracts)
3	October 19	119 DAS	R4 (dough stage)	10 ears husked
4	October 31	131 DAS	R5 (dent stage)	10 ears husked
5	November 10	141 DAS	R5–R6 (transition to physiological maturity)	10 ears husked
6	November 23	154 DAS	R6 (physiological maturity)	10 ears husked

DDS = Days After Drying

Drying and Germination Evaluation

All samples were naturally dried in the Seed Laboratory at CUCBA until reaching a moisture content of 14%. Subsequently, the standard germination test was performed following the procedures of the International Seed Testing Association (ISTA).

Statistical Analysis

A completely randomized design (CRD) was used, with treatments corresponding to the five harvest dates. Results were analyzed using analysis of variance (ANOVA), and mean comparisons were conducted using Tukey's test ($p \leq 0.1$), employing specialized statistical software.

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Arcsine Transformation

In this experiment, the germination percentages (%G) obtained across treatments were transformed using the arcsine square-root function. This transformation, recommended by Steel *et al.* (1997), facilitates the normalization of proportional data and stabilizes variances prior to conducting analysis of variance (ANOVA) or Tukey's test.

Results

Box 2

Table 2

Analysis of Variance for Germination

T	%G	RI	T1	RII	T2	RIII	T3	RIV	T4
1	51	46	42.7	60	50.8	60	50.8	38	38.1
2	83.5	78	62	84	66.4	82	64.9	90	71.6
3	90.5	86	68	96	78.5	90	71.6	90	71.6
4	97	98	81.9	92	73.6	100	90	98	81.9
5	100	100	90	100	90	100	90	100	90
6	99.5	98	81.9	100	90	100	90	100	90

T = Treatments, %G = Germination Percentage, R1 = Replication 1, T1 = Arcsine Transformation 1.

The results show a progressive increase in germination percentage (%G) as the physiological maturity of maize seeds advances. Treatment 1 (50.99%), corresponding to an early harvest (≈110 days, R3–R4 stage, milk grain), exhibited the lowest germination, reflecting physiologically immature seeds with low vigor and viability. As seeds reached more advanced developmental stages (Treatments 2 to 4), germination increased from 83.5% to 97%, indicating strengthening of embryonic tissues and greater accumulation of reserves.

In Treatments 5 and 6, representing the latest harvests (R6, 141–154 days), germination reached its highest values (100% and 99.5%), indicating full physiological maturity. Germination and physiological maturity show a directly proportional relationship. Early harvest (R3–R4) results in seed with low germinative capacity due to embryo immaturity and high moisture content. In contrast, the R5–R6 stages present the most favorable physiological conditions for obtaining viable and vigorous seed, with stable performance even after statistical transformation. Seed harvested 141 days after sowing (R6) can be considered optimal for storage or for use in multiplication plantings (Table 2).

According to the technical-operational manuals of CIMMYT, MacRobert *et al.* (2014) established standardized protocols for the production, certification, and harvest of hybrid maize seed, indicating that harvest is recommended when most kernels exhibit a visible black layer and moisture content of 30–35%, as this ensures ≥90% germination, genetic purity, and vigor. The reference stages for physiological maturity correspond to R5–R6.

The techniques developed in this study complement and validated the guidelines proposed in the CIMMYT manual and can be applied within the regulatory seed industry for hybrid production, in experimental research for the production of parental lines and hybrids, and as replicable material for academic purposes.

Box 3

Table 3

ANOVA of the Experiment

SV	DF	SS	MS	FC	FT (0.05)
Treat.	5	5, 532.96	1, 106.59	48.82**	2.77
Exp. Error	18	407.97	22.66	-	-
Total	23	5, 940.93	-	-	-
CV	6.43	-	-	-	-

FV= Fuentes de variación, GL= Grados de libertad, SC= Suma de cuadrados, CM= Cuadrados medios, FC= F de cuadros, FT= F de tablas, Trat= Tratamientos, ** Diferencias altamente significativas, CV= Coeficiente de variación.

The variation attributable to treatments (5,532.96) represents most of the total variability (5,940.93). This indicates that the stage of physiological maturity at harvest significantly affects the germination capacity of the seeds. The mean square (MS) for treatments (1,106.59) is substantially higher than the MS for error (22.66), which already suggests marked differences among treatments and confirms that the different harvest dates produce seeds with distinct physiological potential.

Because the calculated F-value (FC) is greater than the tabulated F-value (FT), the result is highly significant ($p < 0.05$). The ANOVA supports the hypothesis that harvest stage has a significant influence on germination. This confirms the presence of real statistical differences among treatments with respect to germination (Table 4).

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Box 4

Table 4
Tukey Test

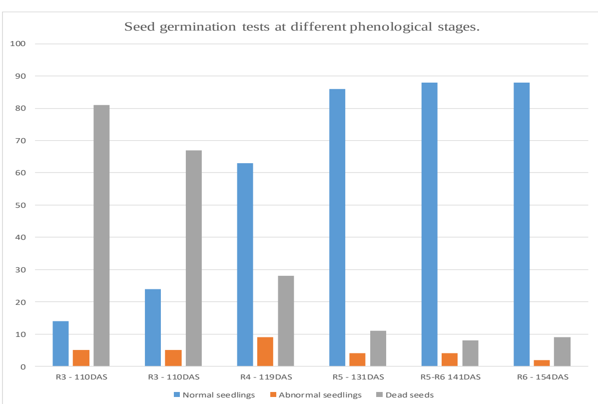
Treat	DAS	% G	Tukey
5	141	100	A
6	154	99.5	AB
4	131	97	C
3	119	90.5	D
2	110	83.5	E
1	110	50.99	F

Treat = Treatments, DAS = Days After Sowing, %G = Germination Percentage.

Treatment 5 (141 days) exhibited the highest germination value (100%) and was classified with the letter A, indicating that it was significantly superior to the other treatments. Treatment 6 (154 days) showed a germination rate of 99.5% and belonged to the AB group, meaning it did not differ statistically from Treatment 5, confirming that full physiological maturity is reached from 141 days onward. Treatments 4 (131 days) and 3 (119 days) obtained germination values of 97% and 90.5%, respectively, classified as C and D, demonstrating a progressive reduction in vigor as maturity decreases. Treatments 2 and 1 (110 days) recorded the lowest germination values (83.5% and 50.99%), corresponding to groups E and F, and showed significant differences compared to the more advanced maturity stages (Table 4).

Box 5

Figure 1
Evolution of Seed Germination Behavior



DAS = Days After Sowing

In the earliest stage (R3–110 DAS), the proportion of normal seedlings is minimal (≈15–20%), while dead seeds exceed 80%. This indicates that the embryo has not yet completed physiological maturation and therefore lacks the capacity to germinate or withstand drying.

The high percentage of mortality reflects embryonic tissue immaturity and elevated moisture content. In the intermediate R4 stage (119 DAS), the proportion of normal seedlings increases (≈60–65%), while dead seeds decrease to less than 30%. By R5 (131 DAS), normal seedlings exceed 85%, indicating that the seed has reached an advanced stage of physiological maturity, with adequate vigor and energy reserves. The number of abnormal seedlings remains low (≈5–10%), demonstrating uniformity in germination.

In the R5–R6 (141 DAS) and R6 (154 DAS) stages, the highest germination percentages are obtained (≈90%), with minimal mortality (<10%) and a very low presence of abnormal seedlings. This confirms that seeds reach full physiological maturity from 141 days after sowing, expressing maximum vigor and viability. No notable differences were observed between 141 and 154 DAS, which is consistent with the stabilization observed in Tukey's test (Figure 1).

The ANOVA (Table 3) supports the conclusion that the maize seed maturation process has a direct and measurable impact on germination. The differences found justify establishing optimal harvest criteria (R5–R6) for obtaining high physiological quality seed.

According to Tukey's test, at 141 days after sowing, the seed reaches physiological maturity and expresses its maximum germination potential (100%). Harvesting between 141 and 154 days does not produce significant differences; therefore, either moment can be considered optimal for seed collection. Harvests conducted before 130 days correspond to reproductive stages R4–R5 (dough or dent stage), where seeds still present physiological immaturity, higher moisture content, and deficient germination (Table 3).

Figure 1 confirms that physiological maturity in maize occurs between 141 and 154 days after sowing. During this interval: Normal seedlings predominate (≈90%). Dead seeds and abnormal seedlings are reduced to a minimum. Therefore, harvesting at the R6 stage ensures seed of high physiological quality, whereas early harvests (R3–R4) produce material with low vigor and high mortality.

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Discussion and Conclusions

The maize maturation process directly influences the physiological quality of the seed. Physiological maturity is reached between 141 and 154 days after sowing (R6 stage), a period during which germination is maximal and stable. Early harvests (R4–R5) exhibit immaturity, high moisture content, and low germination. The stability observed at the R6 stage is associated with intact cellular membranes and high metabolic potential, determined genetically.

The relationship between phenology, moisture content, and germination is validated, providing a model applicable to the seed industry for both genetic rescue and parental seed production under rainfed conditions.

A practical recommendation is to avoid premature harvest and forced drying; slow natural drying with husks is preferable, as it optimizes seed quality. Estrada, *et al*., (2023) reported that as seed moisture decreases to 25–28%, germination rises to 95–98%. In the present study, it was demonstrated that at 14% moisture (natural drying), germination reached 99.5–100%. Estrada *et al.*, (2023) also noted that the black layer marks the cessation of photoassimilate flow; however, if seed moisture remains high, vigor can be reduced.

In this evaluation, it is argued that complete drying at the R6 stage represents the optimal point for maximum germination. Jacob, *et al.* (2014) stated that the milk line is a useful early indicator for anticipating the optimal harvest time, with high germination (≈98–100%) reached upon black layer formation.

In this study, it was confirmed that seeds at the R3–R4 stages still exhibit physiological immaturity and low germination Vieira, *et al.*, (1995) analyzed maize seed physiology and found that grain moisture between 30–35% has a negative relationship with vigor; in contrast, in the present work, natural drying to 14% moisture resulted in maximum germination and stable vigor. Gu, *et al.*, (2017) also identified an ideal harvest point approximately five days before physiological maturity (PM), noting that germination reaches its maximum at PM in both their experiments. Early harvests showed declines in germination, consistent with the findings of Han, *et al.*, (2022).

Feng, *et al*., (2024) compared the physiological behavior of inbred lines similar to those evaluated in this study. Their work argues that vigor and germination do not depend solely on time or moisture content, but rather on the embryo's internal physiological maturity. They conclude that germination percentage depends on the optimal harvest point, which biologically explains the germination stability observed at the R6 stage: mature seeds maintain intact membranes and high metabolic potential. In other words, the genotype regulates the optimal harvest time.

Comparisons with the work of Estrada, *et al*., (2023) confirm that the gradual decrease in grain moisture is a determining factor for reaching physiological maturity and improving germination. Nonetheless, harvesting maize ears at the R3 milk stage and drying them with husks intact can still result in acceptable germination—this technique is used exclusively for genetic seed rescue.

Méndez (2025**),** reported that physiology and crop management determine productivity, noting that planting density directly affects yield. In the present study, we also suggest maintaining an intermediate density recommended for each parental line or hybrid, as well as ensuring adequate irrigation throughout each phenological stage, together with timely harvest at the R5–R6 stages to guarantee vigorous and physiologically stable seed.

Llamocca, (2025) recommended fertilization with 8–10 tons/ha of poultry manure compost and the adoption of an adequate plant density (55,000–60,000 plants/ha) as important factors influencing yield and seed germination. Such practices contribute to producing plants derived from high-quality seed with strong physiological potential.

Fuentes, (2025) also recommended conservation of tillage combined with legume cover crops to improve soil infiltration, moisture, and fertility, thereby enhancing maize seed germination and overall yield. His findings demonstrate improved maize productivity under these conditions. By promoting better soil structure, crop growth and yield are enhanced, particularly when harvest is carried out at the R6 stage and natural ear drying is maintained. This approach protects the physiological integrity of the embryo–seed unit while simultaneously supporting the soil–ecosystem continuum.

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In the work of Cuevas, (2025) spectral indices derived from segmentation and spatial analysis were evaluated. These approaches could also be applied in the present experiment to identify spectral responses in the maize canopy that correspond to the R5–R6 phenological stages. Specific spectral indices may detect decreases in leaf chlorophyll and changes in water content through variations in reflectance patterns. Based on this work, it is proposed that the method is functional for obtaining viable parental seed under anticipated harvest conditions, allowing seed to be collected from stages R3 through R6.

Conflict of Interest

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

Author Contributions

Sánchez Nuño, José Alberto: Manuscript writing, literature review, discussion, and conclusion.

Sánchez Martínez, José: Experiment execution, materials and methods, manuscript writing.

Avendaño López, Adriana Natividad: Laboratory assays, manuscript writing, materials and methods.

Padilla García, José Miguel: Manuscript writing, materials and methods.

Data and Materials Availability

The data generated in this study are available upon request.

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Abbreviations

ISTA = International Seed Testing Association
DAS = Days after sowing
CRD = Completely randomized design
ANOVA = Analysis of variance
FV = Sources of variation
DF = Degrees of freedom
SS = Sum of squares
MS = Mean squares
FC = Calculated F-value
FT = Tabulated F-value
Treat = Treatments
** = Highly significant differences
CV = Coefficient of variation
%G = Germination percentage

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Causal model of the dynamics of free-roaming dogs in Huatusco, Veracruz: A mixed-methods analysis based on territorial and social evidence

Modelo causal de la dinámica de perros en situación de calle en Huatusco, Veracruz: Un análisis mixto basado en evidencia territorial y social

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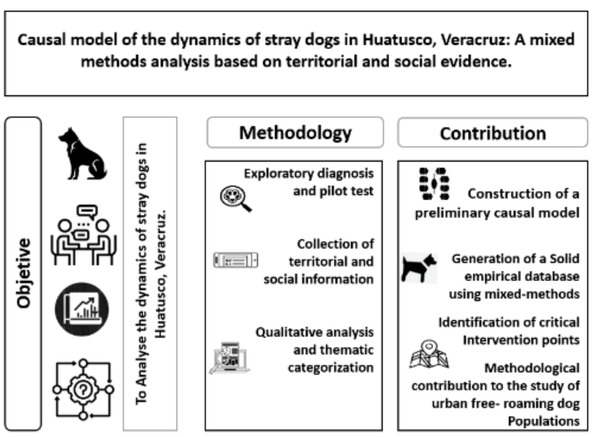
Abstract

The study analyzes the dynamics of the free-roaming dog population in Huatusco, Veracruz, using a mixed-methods approach that integrates household surveys, georeferenced territorial transects, and interviews with key stakeholders. A total of 217 dogs were recorded in the surveyed colonies, and 240 questionnaires were administered, revealing perceptions of sanitary risk, irresponsible pet ownership, and low participation in control actions. Interviews with municipal authorities, the health sector, veterinarians, and animal welfare organizations helped identify structural drivers such as uncontrolled reproduction, abandonment, and food availability, among others. Based on this information, a causal model was developed to synthesize the relationships among the biological, social, and environmental factors that shape the system. The model highlights system components that require greater attention—such as sterilization, waste management, community education, and institutional coordination—and provides the conceptual basis for future stages of dynamic simulation.



Free-roaming dogs, Population Dynamics, causal model

Resumen

El estudio analiza la dinámica de la población de perros en situación de calle en Huatusco, Veracruz, mediante una metodología mixta que integra encuestas ciudadanas, muestreos territoriales georreferenciados y entrevistas a actores clave. Se registraron 217 perros en las colonias muestreadas y se aplicaron 240 encuestas que evidenciaron percepciones de riesgo sanitario, prácticas de tenencia irresponsable y baja participación en acciones de control. Las entrevistas con autoridades, sector salud, veterinarios y asociaciones civiles permitieron identificar causas estructurales como la reproducción no controlada, el abandono, la disponibilidad de alimento entre otras. Con esta información se construyó un modelo causal que sintetiza las relaciones entre los factores biológicos, sociales y ambientales que configuran el sistema. El modelo permite identificar los elementos del sistema que requieren mayor atención como la esterilización, la gestión de residuos, la educación comunitaria y la coordinación institucional, además constituye la base para futuras etapas de simulación dinámica.

Perros callejeros, dinámica poblacional, modelo causal

Area: Advocacy and attention to national problems

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Introduction

The proliferation of stray dogs (Canis lupus familiaris) represents a complex and multi-phase problem that affects the vast majority of cities worldwide, characterized by the interaction of social, environmental and health factors (Kaczorkiewicz, A. J. 2008). The main objective of this study is to develop a dynamic model to analyze the complex interaction of factors that contribute to the presence of unsupervised dogs in public spaces in Huatusco, Veracruz, and its repercussions on public health and animal welfare. The problem is not only due to a lack of education and proper laws, but also poses a serious risk to the health of people, animals, and the environment.

From the perspective of systems thinking, complex urban problems such as the proliferation of stray dogs cannot be understood through simple linear relationships, but from the interactions between multiple social, environmental and biological factors. This approach coincides with the approaches of system dynamics, where the internal structure of a system determines its behavior over time (Forrester, 1961; Aracil & Gordillo, 1997). Under this logic, the phenomenon analyzed in Huatusco can be considered a system characterized by feedback loops that favor its persistence and that need to be represented by causal models to understand its functioning.

The population dynamics of stray dogs are influenced by a variety of interrelated factors (ICAM, 2019) The availability of food resources, such as garbage, plays a crucial role in sustaining homeless dog populations (Control of Stray Dog Populations; (De la Reta, et al., n.d.). The lack of adequate shelters, interaction between animals with and without owners, and irresponsible ownership practices also contribute to overpopulation, socioeconomic and cultural factors influence responsible pet ownership and the community's attitude towards stray dogs (Contreras Torres, E. G. et al., 2017).

The accumulation of waste, the presence of micro-dumps and vacant lots are associated with a greater presence of dogs, while areas with better waste management tend to have fewer stray dogs; (De la Reta, et al., n.d.).

From a health perspective, coexistence between humans and stray dogs increases the risk of transmission of zoonotic diseases, including rabies, leptospirosis, toxocariasis and hookworm. In addition, the presence of these animals in public spaces can generate hygiene problems and environmental pollution (Contreras, et al., 2017) The lack of control of canine populations also hinders efforts to eradicate rabies (ICAM, 2019) Finally, the uncontrolled growth of the canine population has a negative impact on animal welfare, exposing dogs to adverse living conditions, abuse, accidents, and diseases (Contreras Torres, E. G. et al., 2017) (ICAM, 2019). It is essential to understand the dynamics of this problem in order to implement effective and ethical population control strategies (ICAM, 2019) Mass sterilization is an essential tool for controlling overpopulation, as long as it is carried out with a humanitarian approach (Contreras Torres, E. G. et al., 2017), (Escareño, et al., 2023).

However, it must be part of a comprehensive program that also includes adoption, education, and legislation programs (ICAM, 2019). A dynamic model will make it possible to analyze these complex interrelationships and propose solutions based on data and scientific evidence (ICAM, 2019). The use of mathematical models and advanced statistical analysis is a promising approach for understanding, monitoring, and decision-making in dog population management, as long as periodic assessments are carried out (ICAM, 2019)

Methodology

The study was developed under an applied research approach, with the purpose of understanding the problem of stray dogs in Huatusco, Veracruz, and representing their interrelationships through a causal model. The methodology was structured in four successive stages, aimed at integrating quantitative and qualitative information until the formulation of the conceptual model. The use of a mixed approach, combining quantitative and qualitative analysis, is suitable for complex social phenomena such as the present. This type of design has been widely described in the methodological literature applied to social studies, where the integration of multiple sources of information allows for more complete and contextualized interpretations (Alaminos-Fernández, 2025).

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Stage 1. Exploratory diagnosis and pilot test

In a first phase, an exploratory diagnosis was carried out in the Reserva Territorial colony, where pilot sampling was applied in order to evaluate the relevance of the collection instruments.

Box 1

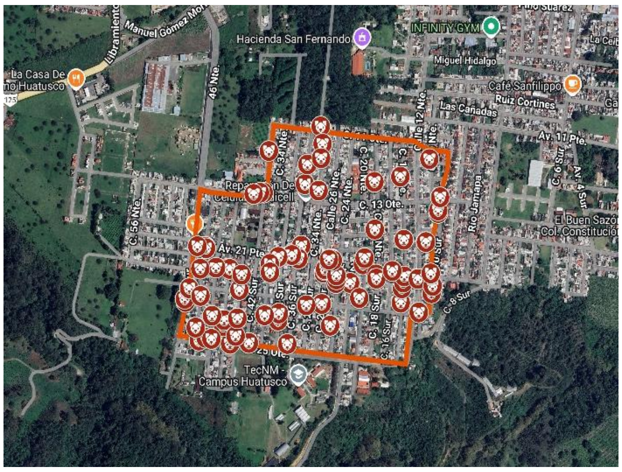

Figure 1
Colonia Reserva Territorial, Sampled stray dogs
Source: own elaboration

This stage allowed the identification of conditions of the urban environment, factors associated with the presence of stray dogs and initial perceptions of the population, Figure 1, the result of the transect sampling that was carried out in the Territorial Reserve can be seen. As a result, the questionnaire was validated and its usefulness in capturing information on citizen knowledge, attitudes and practices was determined.

Once validated, the instrument was prepared for application to a larger sample in various neighborhoods of the municipality. In addition, the methodology for the detection of stray dogs was refined, highlighting the need to carry out the route at the same time through several elements with the intention of avoiding duplication of counting.

Stage 2. Survey of territorial and social information

In the second stage, the fieldwork was extended to the entire urban area of the municipality through three main activities:

Survey Application

240 surveys were applied to inhabitants of different neighborhoods, with the purpose of obtaining a broader characterization of the citizen perception regarding the handling of stray dogs. It had the support of several students from the Higher Technological Institute of Huatusco of the Industrial Engineering career, who participated in the standardization and survey of the surveys.

Territorial sampling and direct observation

Transect tours were carried out that covered approximately 70% of the urban area, this can be seen in Figure 2.

Box 2

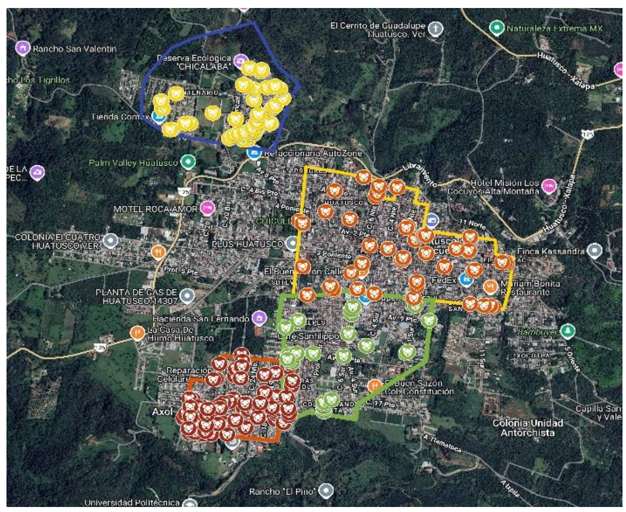

Figure 2
Map with the sampled colonies
Source: own elaboration

During these tours, georeferenced photographs of the dogs observed were recorded, with the aim of spatially locating the distribution of the phenomenon.

Semi-structured interviews

The thematic axes that guided the semi-structured interviews were defined based on the literature on urban wildlife management, animal welfare and public health, but also taking into account what was observed in the pilot phase and in the initial sampling of the project. The theoretical review made it possible to identify the central dimensions of the problem, such as:

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a. Structural causes
b. Health and environmental impacts
c. Current strategies
d. Institutional capacities and constraints
e. Citizen participation and perception

while fieldwork helped to adjust and prioritize these issues according to the local reality. This combination of theory and evidence made it possible to construct interviews focused on the key aspects of the phenomenon and with useful information for the development of the causal model.

The interviews were carried out with representatives of the Directorate of Environment and Ecology of the municipality of Huatusco, the Sanitary Jurisdiction, veterinarians and civil associations, in order to incorporate institutional and social perspectives into the analysis of the system.

Stage 3. Qualitative analysis and thematic categorization

The information obtained from the interviews was transcribed and organized using qualitative matrices. Recurrent thematic categories were identified and a comparative analysis was carried out between actors, which allowed the recognition of patterns, coincidences and contrasts.

This process served to integrate and triangulate the findings from interviews, surveys, and territorial sampling, generating the necessary conceptual basis for the causal model.

Stage 4. Construction of the causal model

From the systematized information, a preliminary causal model was constructed that represents the fundamental interrelationships of the system. In this stage, the connections between the identified elements were defined and the hypotheses that explain the observed behavior were established.

The result consisted of a causal diagram that integrates the main reinforcing and balancing feedback loops, constituting the conceptual structure of the system and serving as a foundation for future stages of dynamic analysis.

Results

Stage 1: Exploratory diagnosis and pilot test

The first stage of the project focused on carrying out an exploratory diagnosis that would allow understanding the initial conditions of the problem and evaluating the relevance of the selected methodological instruments. This phase was carried out in the Reserva Territorial neighborhood, an urban area characterized by a mixture of formal and informal housing areas, the presence of vacant lots and the constant circulation of stray dogs. The choice of this colony as a starting point responded both to preliminary empirical evidence and to citizen reports that indicated a high presence of animals without owners.

During this pilot phase, a first set of surveys was applied with the aim of evaluating their clarity, internal coherence and ability to capture information on knowledge, attitudes and practices of the population regarding the management of stray dogs. The responses obtained showed that residents had diverse perceptions of the magnitude of the problem, while revealing that practices such as informal feeding, abandonment and lack of sterilization were mentioned recurrently. This first survey made it possible to verify that the items of the instrument were understandable and useful to characterize citizen perception, which is why the questionnaire was validated for application to a larger sample.

In addition, an exploratory territorial sampling was carried out in the same colony.

This exercise consisted of systematic tours of streets and spaces for public use, during which stray dogs were counted and environmental conditions were documented that favored their presence, such as accumulation of garbage, little traveled areas and uninhabited homes. Direct observation confirmed that there was a sustained presence of dogs (Figure 3), some with signs of active reproduction, which evidenced the relevance of including variables related to birth, abandonment, and food availability in the later stages of the analysis.

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The pilot phase also made it possible to establish the logistical feasibility of the project. The participation of Industrial Engineering students was key to evaluate the operability of the routes, the times needed to complete transects and the ability to georeference observations with mobile devices. This initial exercise demonstrated that it was feasible to scale the sampling to other colonies and that the field methodology was adequate to generate a reliable record of the stray dog population.

Finally, the triangulation between surveys, direct observation and field notes made it possible to refine the instruments and define more clearly the variables that should be studied at the municipal level. Among them, responsible ownership, abandonment, uncontrolled reproduction, the perception of health risk, informal feeding and citizen participation stood out.

Box 3

Figure 3
Stray dogs

Source: own elaboration

Based on these findings, the methodological route was established that gave way to the second phase of the project, in which the application of surveys was extended to 240 people and territorial sampling was extended to approximately 70% of the urban area of Huatusco.

Stage 2: Survey of territorial and social information

The second stage of the study allowed obtaining a broad and detailed characterization of both the territorial behavior of stray dogs and the perceptions, practices and experiences of the population regarding this problem. The integrated analysis of 240 surveys and 217 georeferenced records of stray dogs allowed to delimit clear patterns in the urban distribution, as well as in the social factors associated with the phenomenon.

2.1 Results of surveys of the urban population

The analysis of the data showed that social perception recognizes the presence of stray dogs as a problem of a mainly health and social nature. More than 60% of the people surveyed considered the problem as serious or very serious, coinciding with the presence observed in the sampled neighborhoods.

The direct experience with stray dogs was significant. Between 25% and 30% of the population reported having experienced risk situations such as bites, chases or attempted attacks, which reinforces the public health dimension identified in institutional interviews. The intersection between educational level and perception of the problem showed that, regardless of the level of schooling, people tend to perceive the presence of stray dogs as a health risk, although people with high school and higher education showed greater willingness to participate in adoption, sterilization or awareness campaigns.

Regarding responsible ownership practices, the data indicate that a considerable percentage of the population keeps dogs unsterilized, which coincides with the explanation given by veterinarians and activists around uncontrolled reproduction. In addition, people who have correct information about sterilization showed more favorable attitudes towards population control and lower tolerance to abandonment.

The analysis also showed that households with a higher number of pets are more likely to allow pets to go out into public space, indirectly contributing to the growth of the street population. This finding highlights the relationship between domestic practices and the presence of animals on public roads.

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Finally, the crossovers between perception of the problem and willingness to collaborate reveal that people who consider the problem as very serious are also those who show more openness to participate in community solutions, which constitutes an important potential for citizen involvement policies.

2.2 Results of territorial sampling and direct observation

The territorial sampling allowed the identification of 217 stray dogs distributed in four strategic neighborhoods:

– Territorial Reserve: 88
– Center: 62
– Centenary: 42
– Emiliano Zapata: 25

The Territorial Reserve concentrated the largest number of dogs observed, which is associated with the presence of uninhabited homes, large spaces, vacant lots and areas with little urban control. This type of environment creates favorable conditions for animal mobility, reproduction, and survival.

The Centro neighborhood, with 62 registered dogs, presents different dynamics: the high commercial activity, the constant transit of people, the presence of food establishments and the accumulation of waste generate conditions that facilitate access to food. This confirms the hypothesis that informal feeding, both by available garbage and by human interaction, is a key factor in the structure of the system.

The Centenario and Emiliano Zapata neighborhoods, although with smaller numbers, maintain a significant presence. The observed characteristics (particularly areas of low surveillance and limited food availability) reinforce the role of the urban environment in the configuration of the system.

Box 4

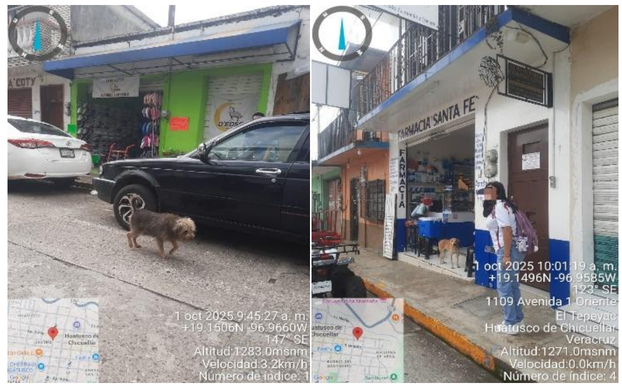

Figure 4
Stray dogs in the city center
Source: own elaboration

Photographic georeferencing made it possible to spatially locate the points where the presence of dogs is concentrated, identifying specific quadrants that function as corridors or micro-territories. These spatial patterns are consistent with the public perception that certain sectors present greater risk or greater interaction between people and animals.

2.3 Integration of social and territorial outcomes

The cross-results between citizen perception and territorial distribution show great coherence. The neighborhoods where the greatest presence of stray dogs was recorded coincide with the areas where the population expresses more health concern and greater frequency of conflicts with animals. Likewise, the data confirm that the problem is not distributed homogeneously, but is concentrated in environments with specific structural conditions: availability of food, low urban surveillance, housing density and the presence of abandoned spaces. The findings of this stage allow us to clearly identify the critical urban nodes, associated social behaviors, and interaction patterns that feed the dynamics of the system. This integration between territorial and social data constitutes the fundamental basis for the construction of the causal model in later stages.

Stage 3: Qualitative analysis and thematic categorization

The qualitative analysis allowed the integration of the information obtained through semi-structured interviews with municipal authorities, civil associations, veterinarians and health personnel.

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This stage was fundamental to identify the social, institutional and environmental elements that make up the dynamics of the system, as well as the relationships that support the causal structure developed later.

The transcriptions were organized into thematic matrices that allowed the discourses to be grouped into central categories, each associated with dimensions of the system that emerged recurrently among the different actors. The categories identified provided clarity on the structural factors, feedback processes and conditioning factors that influence the persistence of stray dogs within the municipality.

3.1 Structural causes of the problem

All the interviews agreed that the growth of the stray dog population is determined by structural factors that are repeated in different areas of the city. Among them are:

– Lack of preventive sterilization, pointed out by both activists and veterinarians, who report low demand for the service and economic and cultural barriers.
– Irresponsible tenure and abandonment, a problem described by all actors as a widespread practice reinforced by the lack of effective sanctions.
– Availability of food, either through access to garbage or informal food provided by citizens, which favors the survival and stability of canine groups.

These factors are configured as core components of the system, and will later give rise to variables such as fertile dogs, birth rate, abandonment, informal feeding and survival in the causal model.

3.2 Health, environmental and social impacts

The qualitative analysis revealed a shared concern about the effects of the presence of stray dogs. Among the main impacts mentioned are:

– Public health risks, especially bites, parasitosis, scabies and potential zoonoses.
– Pollution on public roads, associated with broken garbage bags, feces in the streets and bad odors.
– Negative perception and neighborhood conflicts, particularly in areas with a higher concentration of animals.

These findings made it possible to identify variables related to incidents, health impact, environmental impact, and social pressure, all of which are fundamental to explaining how the effects of the system generate citizen and government responses.

3.3 Current strategies and their scope

The analysis showed that the existing actions to control the problem are reactive, sporadic and with limited resources. Among the main activities observed are:

– Rabies vaccination campaigns organized by the health sector.
– Intermittent sterilization campaigns promoted by civil associations.
– Specific actions of the City Council for capture or removal for specific incidents.

The qualitative information made it possible to differentiate between strategies with potential impact (such as sterilization) and actions with limited effect (such as temporary capture). These observations are subsequently translated into causal variables such as sterilization campaigns, institutional support, capture/removal, and mortality.

3.4 Institutional capacities and constraints

The interviewees agreed that institutions face important restrictions:

– Shortage of budget and specialized personnel in the municipal area.
– Absence of a permanent animal control program.
– Dependence on operational volunteers for rescue, dissemination and sterilization activities.

These limitations are elements that affect the strength of the system's balancing loops; they are then represented by variables such as institutional capacity or frequency of campaigns.

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3.5 Citizen participation and social perception

The interviews showed that citizens have a heterogeneous participation: while some sectors support with food, adoptions or donations, others express discomfort, indifference or rejection towards dogs or associations.

The relevance of these social perceptions coincides with studies on community-wildlife interaction, where local knowledge and citizen attitudes significantly influence management and conservation strategies (Lemus Ramírez, 2025).

Likewise, an incipient process of sensitization was identified in schools and among some families.

From this category, relevant social variables emerge such as citizen attitudes, willingness to collaborate and citizen reports, which subsequently interact with the generation of social pressure on the authorities.

3.6 Conceptual integration towards the causal model

The triangulation between the categories derived from interviews, the results of the surveys and the territorial distribution made it possible to clearly identify the endogenous components that structure the dynamics of the system.

From this stage emerge the conceptual foundations that will sustain the causal model, particularly around:

– Mechanisms of reproduction and survival,
– Population flows associated with adoptions and mortality,
– Health and environmental impacts,
– Citizen and institutional responses,
– Reinforcing and balancing feedback.

These relationships are formalized in the next stage through the construction of causal diagrams that synthesize the internal structure of the system and explain the persistence of the problem in the municipality.

4: Construction of the causal model

The fourth stage of the study consisted of the conceptual integration and formalization of the elements identified in the previous stages within a causal model, elaborated under the logic of system dynamics.

This model synthesizes the internal relationships of the system, organizes the factors detected through fieldwork and establishes the endogenous hypotheses that explain the persistence and evolution of the phenomenon of stray dogs in Huatusco.

The use of feedback-based causal models is based on the principles of system dynamics, where the internal structures of the system determine its behavior over time (Aracil & Gordillo, 1997).

The use of causal models and approaches based on system dynamics has been recommended to analyze urban and socio-environmental problems characterized by high levels of interaction and feedback, as shown by the dynamic modeling work developed for other complex urban systems (Linares-Fleites et al., 2016).

4.1 Identification of the central variables of the system

From the triangulation between the data from surveys, territorial sampling, and interviews, a set of variables was established that operate as essential components of the system. Among the most relevant are:

– Population of stray dogs
– Birth rate / fertile dogs
– Abandonment and irresponsible tenure
– Food availability (garbage, informal feeding)
– Survival
– Incidents and bites
– Citizen Reports
– Capture the retreat
– Mortality
– Saturation of shelters
– Adoptions
– Health and environmental impact
– Social pressure
– Institutional support
– Sterilization campaigns

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These variables were selected for their recurrence in the actors' discourse, their consistency with quantitative data, and their role within the social, biological, and urban processes observed.

It is important to note that the variables identified in this stage correspond only to the endogenous elements of the system. These variables reflect the internal structure of the phenomenon and allow us to understand how the dynamics of the dog population in Huatusco are generated and sustained.

On the other hand, the definition of exogenous variables will be carried out in later phases, once the dynamic model has been built and the simulation scenarios have been developed. These variables will correspond to external factors that influence the system without directly depending on its internal behavior.

Among the possible exogenous elements that could be incorporated are: the municipal budget allocated to animal control, changes in state or federal regulations, extreme weather events, urban growth processes or population migration, economic variations that affect responsible ownership and the intervention of external organizations through massive campaigns or extraordinary support, among others. Their inclusion will depend on the relevance and relevance they show during the experimentation phase of the model.

4.2 Internal relationships and causal hypotheses

The causal relationships identified between the variables constitute the endogenous hypotheses of the system, formulated from the evidence collected in surveys, interviews and territorial sampling. This approach coincides with the approaches of Forrester (1961), who established that social and urban systems present patterns of behavior derived from internal feedback structures.

These hypotheses describe the internal mechanisms that explain the persistence of the stray dog population in the municipality and allow structuring the causal model. Each of the identified hypotheses is described below:

a) First, it was established that the birth rate depends directly on the number of fertile dogs, so a greater number of unsterilized animals increases the probability of reproduction and, consequently, the stray population.
b) In a complementary way, the availability of food, whether through accessible garbage or informal food provided by people, favors the survival of dogs and contributes to the sustained growth of the system.
c) At the social and health level, it was observed that an increase in the street population increases the frequency of incidents, such as bites, neighborhood conflicts and risk situations, which in turn increases citizen reports. This relationship constitutes a hypothesis of reactive response, in which the authorities carry out capture or removal actions motivated by specific complaints, although with temporary effects and limited in scope.
d) The analysis also identified that the health and environmental impacts derived from the presence of dogs such as scattered garbage, feces on public roads and risk of zoonoses, intensify social pressure on municipal authorities. This pressure influences the probability of receiving institutional support, which conditions the frequency of sterilization campaigns. These campaigns reduce the number of fertile dogs and, with it, the birth rate, forming a control mechanism with significant potential, although dependent on limited resources.
e) Interviews with civil associations also showed that the saturation of shelters increases as the street population and rescues grow. This saturation restricts the ability to manage adoptions, which weakens the effectiveness of this strategy as a mechanism for population reduction.
f) Finally, mortality operates as a natural starting factor, but its effect is insufficient in the face of the mechanisms that favor reproduction and survival.

All the above-mentioned relationships identified, as well as internal relationships and endogenous hypotheses of the system are shown in Figure 5.

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4.3 Causal hypotheses

The causal model allowed us to identify several feedback loops that explain the dynamics of growth and regulation of the dog population.

As Forrester (1969) demonstrated in studies of urban dynamics, reinforcing and balancing loops explain why certain phenomena tend to persist or amplify even when there are partial interventions. This conceptual framework allows for a proper interpretation of the loops identified in the stray dog system.

a) Uncontrolled playback reinforcer loop.

The presence of fertile dogs increases the birth rate, which increases the stray population and perpetuates the number of unsterilized animals. This cycle favors a continuous growth of the system.

b) Facilitated survival reinforcer loop.

The availability of food increases the survival of dogs and increases the stray population, which in turn perpetuates practices and conditions that maintain the supply of food.

c) Sterilization balancer loop.

The social pressure derived from the health and environmental impacts affects the institutional response, promoting sterilization campaigns that reduce the number of fertile dogs. This mechanism has regulatory potential, although it depends on limited resources.

d) Reactive action balancer loop.

The increase in dogs increases incidences and citizen reports, which activates capture or removal actions. However, its effect is temporary and does not act on the structural factors of the system.

e) Adoption Balancer Loop (weak).

The saturation of shelters limits the ability to promote adoptions, reducing the influence of this mechanism on the street population. Its regulatory effect is marginal.

Box 5

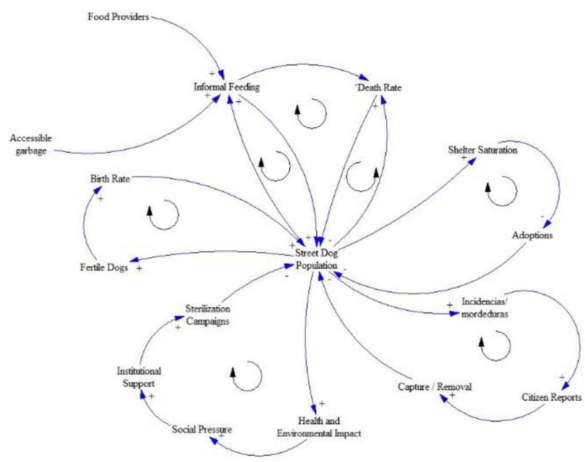

Figure 5

Causal Model of Stray Dog Population Dynamics

Source: own elaboration

Conclusions

The present study allowed a broad understanding of the problem of stray dogs in Huatusco, Veracruz, evidencing that it is a complex system where biological, health, social, and institutional factors interact, as indicated by the international literature on urban wildlife management (Contreras Torres, E. G. et al., 2017); (OIE, 2011); (ICAM, 2019). The mixed approach employed that included surveys, georeferenced territorial observation, and semi-structured interviews provided a solid empirical basis to characterize the local dynamics and support the construction of the causal model.

The evidence points to the possibility that the stray dog population in Huatusco is maintained by the joint action of internal reinforcing mechanisms, especially uncontrolled reproduction, abandonment and the wide availability of food through garbage or informal feeding practices. This behavior coincides with previous studies that highlight the importance of food supply and irresponsible ownership as key drivers of the growth of unsupervised dog populations (De la Reta, et al., n.d.); Benítez-García & Miranda-Cabrera, 2018). It is also presumed that regulatory mechanisms such as sterilization, adoptions, reactive capture or mortality have a limited scope. The intermittency of campaigns, the saturation of shelters, budget insufficiency, and low social accountability can contribute to the fact that balancing loops are significantly weaker than reinforcers, which coincides with what has been reported in the literature on the failure of fragmented or non-continuous strategies.

Hernández-Armenta, Abigail, Córdoba-Gómez, Luis Tomás, Solís-Jiménez, Miguel Ángel and Calderón-Palomares, Luis Antonio. [2025]. Causal model of the dynamics of free-roaming dogs in Huatusco, Veracruz: A mixed-methods analysis based on territorial and social evidence. Journal of Research and Development. 11[26]1-12: e31126112
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The causal model constructed synthesizes these interrelationships and shows the possibility of why the dog population tends to remain high and expand in areas with favorable conditions, such as accumulation of waste, presence of vacant lots or lack of territorial control. The analysis of loops reveals that the structure of the system favors population permanence and growth, while existing interventions fail to modify the structural causes of the problem. This reinforces the need, already pointed out by international organizations (OIE, 2011); (ICAM, 2019), to implement comprehensive, sustained, and multi-stakeholder strategies.

Likewise, the model allows the identification of critical points of intervention, including continuous sterilization with a preventive approach, proper waste management, citizen education to promote responsible ownership and effective coordination between authorities, the health sector, veterinarians and civil associations. The systemic perspective used shows that these elements cannot be addressed in isolation, since their interaction is what determines the behavior of the system.

Although this research does not yet advance towards the dynamic simulation of levels and flows, the formulation of the causal model is a fundamental step. The developed scheme offers a robust conceptual structure to guide future computational modeling efforts, evaluate intervention scenarios, and support evidence-based decision-making. Consequently, the results of this study lay the foundations for moving towards a dynamic model that allows us to assess, in a quantitative way, the potential impact of different population control policies.

Conflict of interest

The authors declare no interest conflict. They have no known competing financial interests or personal relationships that could have appeared to influence the article reported in this article.

Author contribution

Hernández-Armenta, Abigail: I carry out the collection of information, coordination in the application of surveys and automation of information, as well as the development of interviews with the different actors identified, as well as in the writing of the article.

Córdoba-Gómez, Luis Tomás: Contributes to the collection of information on current public policies regarding the proliferation of stray dogs; also in the coordination of transects for the count of stray dogs in the city; also supports in the interviews with the different actors identified in the project.

Jiménez-Solís, Miguel Ángel: I carry out the design of the research and the analysis of the data, reviewed and participated in the writing of the article, as well as in the elaboration of the causal model.

Calderón-Palomares, Luis Antonio: I contribute to the elaboration of the causal model, I also contribute to the evaluation of its variables and hypotheses.

Availability of data and materials

The project's data were obtained through three complementary strategies: surveys applied to inhabitants of different neighborhoods of Huatusco, territorial sampling with georeferenced photographic record of dogs observed on public roads, and semi-structured interviews conducted with key actors such as municipal authorities, the health sector, veterinarians, and civil associations

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A thank you is also extended to the Municipality of Huatusco for the facilities granted to carry out the territorial sampling and the necessary institutional coordination. Finally, sincere recognition is expressed to the actors interviewed: municipal authorities, the health sector, veterinarians and civil associations, for their willingness and valuable contribution to the analysis of the problem.

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Article

Analytical platform for operational performance assessment using Random Forest and K-Means

Plataforma analítica para la evaluación del desempeño operativo utilizando Random Forest y K-Means

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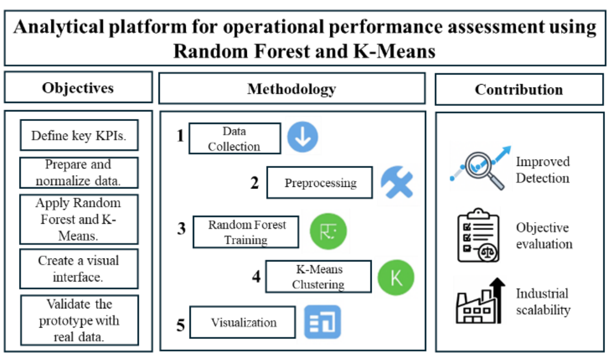
Abstract

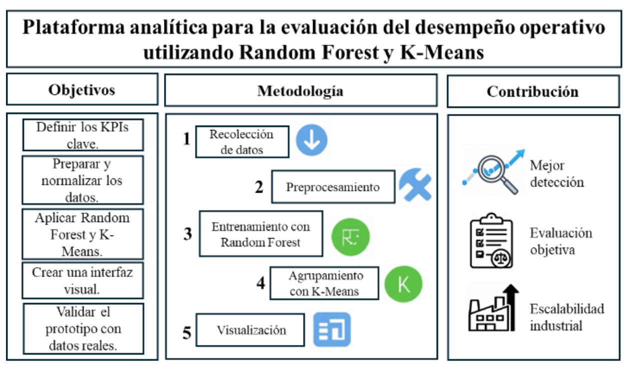
This study presents an analytical platform for assessing operational performance in industrial environments through the use of Random Forest classification and K-Means clustering. Five key performance indicators—scrap rate, cycle time, quality, attendance, and supervisor-based performance rating—were selected to evaluate a dataset of 293 operators from different production areas. After data cleaning and normalization, the Random Forest model achieved an accuracy of 89%, correctly classifying operators into High, Regular, and Critical categories. In parallel, K-Means generated three performance clusters that highlight distinct behavioral patterns among operators. The system was developed with a modular architecture using FastAPI, PostgreSQL, and React.js, and includes interactive dashboards created in Power BI to support decision-making. Practical validation showed benefits such as increased objectivity, transparency, and improved identification of both high and low performers. The results demonstrate that data-driven methods can enhance operational evaluation and provide a foundation for scalable industrial applications.



Random Forest, K-Means Clustering, Operational Performance

Resumen

Este estudio presenta una plataforma analítica para evaluar el desempeño operativo en entornos industriales mediante Random Forest y K-Means. Se seleccionaron cinco indicadores clave: tasa de scrap, tiempo de ciclo, calidad, asistencia y evaluación del supervisor, aplicados a un conjunto de 293 operadores de distintas áreas de producción. Tras la limpieza y normalización de datos, el modelo Random Forest alcanzó una precisión del 89%, clasificando correctamente a los operadores en categorías Alto, Regular y Crítico. Paralelamente, K-Means generó tres grupos que revelan patrones de comportamiento diferenciados. El sistema se desarrolló con arquitectura modular usando FastAPI, PostgreSQL y React.js, e incluye paneles interactivos en Power BI para apoyar la toma de decisiones. La validación práctica mostró mayor objetividad, transparencia e identificación más clara de desempeños altos y bajos. Los resultados evidencian que el análisis basado en datos fortalece la evaluación operativa y permite aplicaciones industriales escalables.



Random Forest, Agrupamiento K-Means, Desempeño Operativo

Area: Development of strategic leading-edge technologies and open innovation for social transformation

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Introduction

In today's highly competitive industrial environment, organizations must sustain elevated levels of efficiency, quality, and operational consistency to ensure their long-term viability. Within this context, the performance of line operators becomes a strategic variable, as their daily actions directly affect productivity, scrap generation, process stability, and overall operational outcomes. Accurate performance assessment is therefore essential not only for measuring results but also for identifying improvement opportunities, developing workforce capabilities, and strengthening organizational culture.

However, despite its importance, many manufacturing plants continue to rely on manual, subjective, and low-frequency evaluation methods that offer limited feedback, hinder timely decision-making, and may generate distrust among personnel.

The emergence of Industry 4.0 has transformed this landscape by introducing digital tools that enable the automation of data capture, processing, and interpretation. Artificial intelligence and machine learning allow organizations to analyze large datasets, recognize behavioral and operational patterns, and generate precise, real-time predictions that support more objective and consistent evaluations. These technologies facilitate the transition from intuition-based assessments toward evidence-based decision systems aligned with continuous improvement principles.

This article presents the development of a functional prototype of an intelligent digital system designed to assess operator performance in industrial settings. The proposed solution integrates key performance indicators (KPIs) with classification algorithms (Random Forest) and clustering techniques (K-Means), enabling both predictive modeling and the segmentation of operational profiles. Additionally, a user-friendly visual interface provides supervisors with accessible, timely insights that enhance fairness, transparency, and strategic decision-making. The results demonstrate the potential of combining data analytics with modern computational techniques to strengthen operational management and promote more efficient, equitable, and data-driven industrial environments.

Conceptual Foundations

Performance Evaluation in the Manufacturing Industry

In the maquiladora environment, performance evaluation is essential for identifying operational strengths and weaknesses, directly impacting overall efficiency. Indicators such as the number of units produced, adherence to standard cycle times, errors per shift, product quality, and defect rates are widely recognized as critical metrics for this purpose [Vega et al., 2022]. These indicators enable continuous monitoring of performance and support decision-making processes focused on operational improvement.

Beyond technical aspects, human factors such as job attachment, motivation, and recognition also exert a significant influence on performance. Their omission may lead to an underestimation of human talent and to a limited interpretation of operational results [Soto et al., 2024]. Likewise, the presence of an effective internal control system contributes to greater productivity and operational discipline, while its absence has been associated with recurrent failures and lower individual performance [Valdez & Salcido, 2021].

Furthermore, in sectors such as the textile industry, logistical conditions, production infrastructure, and supply chain management have been shown to directly affect the quality of operator performance. Factors such as material availability, efficiency of production flows, and workspace organization influence daily consistency and stability in operator output [International Labour Office, 1998; Algawatta & Jayasekara, 2025; Rahman et al., 2024]. These elements highlight the need to consider not only technical variables but also human and contextual factors to achieve more comprehensive and accurate performance assessments in industrial environments.

Lean Manufacturing, Technical Processes, and Digitalization

The Lean approach has contributed significantly to improving efficiency in various maquiladora environments; however, its full potential is often constrained when implementation is only partial, particularly when Lean principles are not fully integrated into technical and production processes [Chávez, 2022].

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Partial adoption typically results in fragmented workflows, limited standardization, and inconsistencies that prevent organizations from achieving the continuous improvement and process stability that Lean aims to promote.

In this context, digitalization has emerged as a critical complement to Lean initiatives. Digital tools such as interactive dashboards and real-time visualization systems provide immediate access to operational data, enabling supervisors and managers to continuously monitor performance, detect anomalies, and respond promptly to deviations that may compromise productivity or quality [Vega et al., 2022; Guzmán-Anaya, 2019]. These technologies support more agile and data-driven decision-making, strengthening the alignment between operational practices and Lean objectives.

In the textile sector specifically, researchers have emphasized the pressing need to modernize information systems to comply with international standards and to strengthen traceability across production and supply-chain operations. Improvements in digital infrastructure are essential for enhancing responsiveness, reducing uncertainty, and ensuring that production flows meet the growing demands of global markets [Díaz et al., n.d.; Silva-Castro et al., 2025]. These findings collectively underscore the importance of integrating Lean methodologies with robust digital systems to achieve greater efficiency, transparency, and operational consistency in modern manufacturing environments.

Application of Machine Learning

The application of machine learning has proven highly effective for processing large datasets and enhancing analytical decision-making in industrial environments. Algorithms such as Random Forest have demonstrated strong performance in multivariable classification tasks, particularly in scenarios where numerous operational indicators must be evaluated simultaneously. Their robustness to noise, ability to model nonlinear relationships, and capacity to handle heterogeneous feature sets make them well-suited for complex production systems [Kang et al., 2020; Tobar-Díaz et al., 2023].

These characteristics are especially valuable in maquiladora settings, where operator performance can be influenced by dynamic interactions between time, quality, workload, and equipment conditions.

Similarly, unsupervised learning techniques such as K-Means provide powerful tools for exploring underlying behavioral patterns in operational data. By grouping operators into clusters based on similar measures, K-Means enables the identification of natural performance profiles—ranging from highly consistent operators to those exhibiting irregular or risk-prone patterns—even when no prior labels or categories are available [Sandoval, 2018]. This capacity to discover latent structures is critical in environments seeking to implement preventive and improvement-oriented strategies.

When these analytical models are integrated into interactive graphical interfaces, traditional evaluation practices evolve into automated, transparent, and feedback-driven systems. Interfaces designed to visualize classifications, cluster assignments, and deviations allow supervisors to interpret results more easily, accelerating corrective action and promoting continuous improvement [Tobar-Díaz et al., 2023]. Such systems reduce subjective bias, standardize evaluation criteria, and strengthen the alignment between human resource management and operational performance.

Moreover, advances in artificial intelligence—supported by emerging fields such as quantum computing and nanotechnology—are expanding the predictive and computational capabilities available to industry, pointing toward increasingly sophisticated and autonomous evaluation mechanisms [Díaz-Ramírez, 2021]. These developments reinforce the need for organizations to adopt intelligent, data-driven tools capable of adapting to growing operational complexity. In highly competitive industrial contexts, the integration of supervised and unsupervised learning models becomes a strategic asset, enabling companies to anticipate issues, optimize performance, and maintain resilience amid rapidly evolving global demands.

Recent studies have also explored hybrid machine-learning approaches that combine supervised and unsupervised models to enhance analytical accuracy.

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For example, in the domain of connected healthcare systems, an enhanced Random Forest classifier integrated with K-Means clustering was used to detect and categorize anomalous behaviors in IoMT networks, demonstrating superior performance compared with standalone algorithms [Al-Abadi et al., 2023].

Although applied in a different domain, this work reinforces the effectiveness of combining classification and clustering techniques for improving decision-making in complex operational environments.

Additionally, work in textile manufacturing illustrates how AI-based systems are transforming traditionally manual and error-prone inspection processes. A recent study implemented an enhanced deep convolutional neural network (DCNN) integrated with robotic motion and sensor-based alerts, achieving automatic classification of 13 types of fabric defects with a mean Average Precision of 97.49%.

These results highlight the effectiveness of AI-driven inspection systems and reinforce the broader industrial shift toward objective, data-driven evaluation tools—an approach aligned with the intelligent operator-assessment framework proposed in this study [Hassan et al., 2024].

Integration of an Intelligent Evaluation System

The proposed technological architecture is built upon modern tools that enable efficient data management, analytical processing, and user-friendly visualization. FastAPI (Python) serves as the backend framework, providing a high-performance environment for handling requests, executing analytical models, and managing system logic. PostgreSQL is used as the main database engine, offering reliability, scalability, and robust support for structured operational records.

On the client side, React.js allows the development of dynamic and responsive interfaces capable of displaying performance indicators clearly and interactively. During the initial design stages, visualization modules were prototyped using Power BI to generate rapid dashboards and intuitive data exploration views [Guzmán-Anaya, 2019; Tobar-Díaz et al., 2023].

This technological structure supports the continuous ingestion, processing, and interpretation of operator-performance data, enabling the platform to provide timely analytical insights and to integrate machine-learning components into the evaluation workflow. Its modular and extensible design also makes it adaptable to a wide range of production contexts. In sectors such as apparel and textile manufacturing, previous studies have demonstrated the relevance of monitoring variables such as operation times, stitching quality, and process-control elements to reduce production failures, improve order fulfillment, and strengthen the consistency of manufacturing flows [Silva-Castro et al., 2025]. These findings reinforce the importance of intelligent evaluation systems capable of aligning digital infrastructure with the specific requirements of each production environment.

Methodology

This study is framed within an applied technological research approach, focused on the development and experimental validation of a functional prototype for evaluating operator performance. The methodology follows a quantitative orientation and a non-probabilistic experimental design. Its main objective was to develop and validate an intelligent evaluation system grounded in objective indicators and machine-learning algorithms, structured into five core phases.

A. Data Collection and Structuring

Five priority KPIs were identified for assessing operational performance: average scrap (number of defective units per operator), cycle time (average duration required to produce a single unit), quality (ratio of accepted to total produced units), attendance (percentage of days worked), and a global operator evaluation (categorized as Critical, Regular, or High based on supervisor criteria). These indicators were automatically captured from control sheets and operational databases, ensuring consistency and minimizing manual intervention. The dataset incorporated a total of 293 operators from different production areas, each anonymized using identification codes (e.g., OP297, OP613) to guarantee confidentiality while enabling full traceability of performance patterns across the evaluation period.

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This structured dataset provided a reliable foundation for the subsequent analytical phases and for the implementation of the machine-learning models integrated into the intelligent evaluation platform.

B. Data Preprocessing

The collected information was subjected to a comprehensive preprocessing stage that included data cleaning, statistical normalization, and manual verification to ensure internal consistency and reliability. The following techniques were applied during this phase:

– Data normalization, used to standardize the scale of numerical indicators and prevent disproportionate weighting in subsequent analytical models.

– One-hot encoding, implemented to convert categorical variables into binary vectors, allowing supervised algorithms to interpret them correctly.

– Training–testing split, performed to generate independent datasets for model training and performance evaluation, ensuring objective measurement of generalization capacity.

All preprocessing procedures were carried out using Python, primarily through the Pandas library for data manipulation and Scikit-learn for machine-learning transformations. This step ensured that the dataset was clean, structured, and computationally compatible with the supervised and unsupervised models used in later phases, forming a robust foundation for accurate analytical modeling.

C. Modeling with Machine Learning Algorithms

For the system's modeling stage, a complete workflow was implemented in Python integrating the selected algorithms through the Scikit-learn library. The Random Forest model was trained using historical operator records that had been previously labeled by supervisors. The normalized KPIs served as input features, ensuring that all variables contributed proportionally to the model's decision structure.

The dataset was partitioned into training and testing subsets, and cross-validation techniques were applied to guarantee consistency, reduce overfitting, and assess the model's generalization capability.

In parallel, the K-Means algorithm was applied using the same standardized indicators, which were normalized through z-score transformation to ensure comparable feature scales. The value of $k = 3$ was defined to generate three operator clusters, corresponding to distinct performance profiles. The k-means++ initialization method was used to improve centroid selection and promote stable convergence, reducing the likelihood of suboptimal clustering results.

Both models were encapsulated into modular Python scripts and integrated into the system's backend, allowing them to be executed automatically whenever new operator data are received. This modular architecture ensures scalability, maintainability, and seamless communication between the analytical components and the visualization interface, enabling real-time performance analysis within the intelligent evaluation platform.

D. Visual Interface Design

An interactive dashboard was developed as a functional prototype using Power BI (Fig. 2), with the purpose of facilitating the visual interpretation of results for supervisors and talent-management personnel. The interface was designed to provide a clear, accessible, and intuitive overview of operator performance, allowing users to explore the analytical outputs generated by the system.

Its main features include the visualization of key performance indicators for each operator, graphical distributions of evaluation outcomes, historical trends showing the evolution of performance over time, and the segmentation of operators based on cluster assignments. The dashboard also incorporates dynamic tables, filters, and color-coded indicators (similar to traffic-light systems) to highlight critical conditions and facilitate rapid decision-making. These elements collectively enhance usability and support a more transparent, data-driven evaluation process.

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Box 1

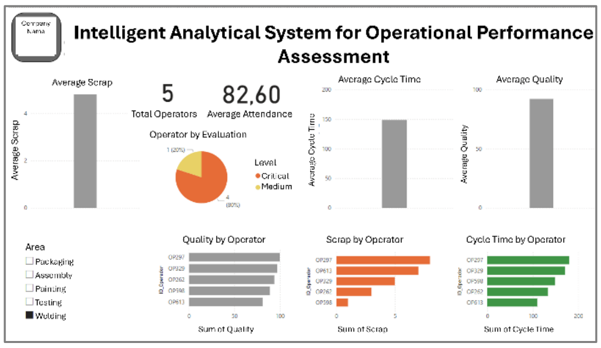

Figure 1
Dashboard of the intelligent system for operator performance evaluation

Source: Own Elaboration

The system was validated in a controlled testing environment using real operational data, replicating the complete evaluation workflow under conditions similar to those of a real production setting. Throughout this process, supervisory personnel highlighted several practical advantages. Among the most frequently mentioned were the increased objectivity and transparency of the evaluations, the reduction in the time required to generate performance reports, and the system's ability to reveal both high-performing operators and previously unnoticed low-performance cases. Supervisors also reported an improvement in the perceived fairness of the evaluation process, noting that the analytical criteria provided clearer and more consistent justification for classification outcomes. Although this validation was experimental and limited to a testing environment, the results demonstrate strong potential for future deployment in real industrial contexts. The system's capacity to integrate diverse performance indicators, automate analytical workflows, and generate interpretable visual outputs positions it as a valuable tool for organizations seeking to standardize evaluations and support data-driven decision-making.

System Architecture

The system was designed following a modular and scalable architectural approach based on modern web technologies, as illustrated in figure 2. This structure allows each component—backend services, database management, machine-learning models, and visualization interfaces—to operate independently while maintaining seamless communication across the platform.

Such modularity greatly simplifies maintenance, facilitates the integration of new analytical models or data sources, and enhances the system's adaptability to different operational areas within an organization.

This architectural flexibility ensures that the platform can evolve in response to new evaluation requirements, additional KPIs, or changes in production dynamics, making it a robust and future-oriented solution for intelligent performance evaluation.

Box 2

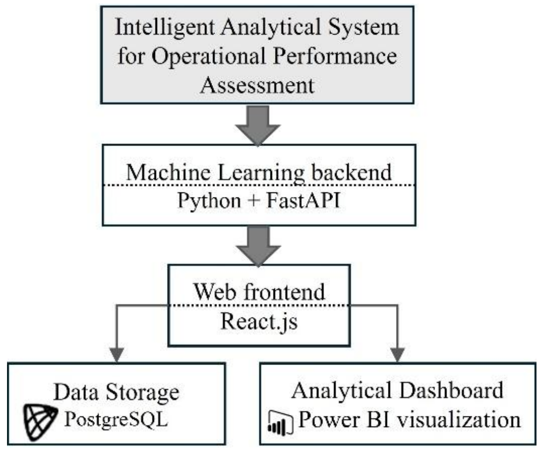

Figure 2
General system architecture with AI components and visualization

Source: Own Elaboration

Results and Discussion

As part of the development of the functional prototype, the machine-learning models (Random Forest and K-Means) were integrated and executed using a real operational dataset. These models constituted the core analytical components of the system, and their performance was evaluated alongside the data-processing workflow and the visualization interface. This joint evaluation made it possible to observe not only the predictive accuracy of each model but also their practical contribution to the decision-support capabilities of the intelligent evaluation platform.

A. Results of the Random Forest Model

The supervised classification model (Random Forest) achieved an accuracy of 89%, correctly assigning operators to three performance categories: High, Regular, and Critical, as shown in Table I.

This level of accuracy indicates that the model was able to capture relevant relationships among the key performance indicators (KPIs) and reproduce the supervisor-assigned labels with notable consistency.

The model's output also contributed to a more objective interpretation of operator performance by reducing subjective bias and providing transparent criteria for each classification. In the context of an industrial environment—where evaluation processes are often manual or partially standardized—this represents a significant improvement. The integration of Random Forest within the system's architecture allowed the classifications to be visualized in real time through the dashboard, enabling supervisors to identify patterns, detect emerging risks, and make more informed decisions regarding personnel management and process improvement.

Box 3

Table 1

Prediction Results

Operator ID	Scrap (%)	Time (s)	Quality (%)	Attendance (%)	RF Prediction
OP297	1.2	12.3	98.7	100	High
OP302	4.5	15.0	91.2	92	Medium
OP613	8.7	18.5	84.3	85	Critical
OP417	2.8	141	95.5	96	Medium
OP509	0.9	11.8	99.1	100	High

Source: Own Elaboration

Figure 3 presents the confusion matrix generated by the Random Forest model when classifying operator performance. Since all instances fall along the main diagonal, the results indicate complete agreement between the model's predictions and the actual evaluations conducted by supervisors. This perfect alignment demonstrates that, under the testing conditions, the model was able to accurately capture the patterns encoded in the training data and reproduce the supervisory criteria without misclassifications.

Such behavior reflects a notably high level of predictive reliability, suggesting that the selected KPIs carry sufficient discriminative power to differentiate between the performance categories.

Moreover, this outcome reinforces the model's potential as a robust decision-support tool, capable of contributing to more objective, consistent, and transparent evaluation processes in operational environments. The results also highlight the feasibility of integrating supervised learning techniques into daily management practices, particularly in contexts where rapid and data-driven assessments are essential.

Box 4

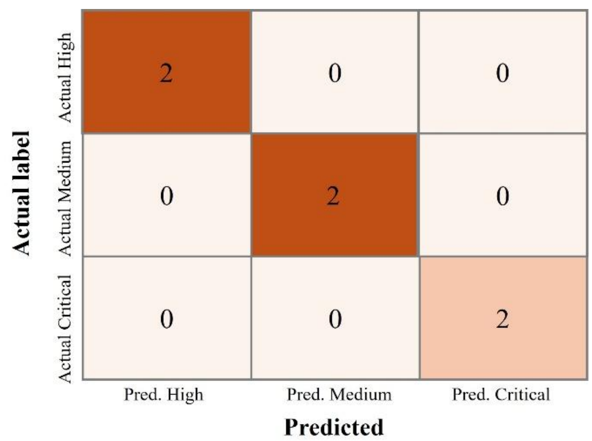

Figure 3

Confusion Matrix of the Random Forest Model

Source: Own Elaboration

B. Results of the K-Means Model

Figure 4 presents the comparison of average KPI performance values across the three clusters generated by the K-Means algorithm. Cluster 0 corresponds to the group of high-performing operators, characterized by consistently superior values in all indicators, including lower scrap, shorter cycle times, higher quality, and stronger attendance records. Cluster 1 represents an intermediate performance group, showing acceptable but not optimal results, which suggests the presence of operators with stable yet improvable behaviors. In contrast, Cluster 2 groups those operators with the highest scrap levels, reduced quality, and lower attendance, clearly identifying them as the critical segment requiring closer supervision and targeted corrective actions.

This segmentation provides a structured view of performance variability within the workforce and facilitates the identification of operational patterns that may not be evident through traditional evaluation methods. The detailed values associated with each cluster are presented in Table 2.

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Box 5

Table 2
Cluster Summary

Cluster	Scrap (%)	Time (s)	Quality (%)	Attendance (%)
0	7.28	12.20	90.00	91.36
1	2.65	14.03	94.91	89.57
2	6.88	17.55	90.40	90.62

Source: own elaboration

Box 6

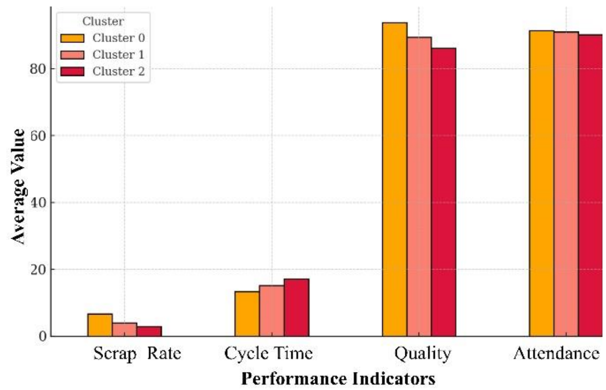

Figure 4
Comparison of average KPIs by cluster (K-Means)
Source: Own Elaboration

Consistent with the findings reported by [Kang et al., 2020], the implementation of machine-learning algorithms in production lines not only enables the automation of complex processes but also facilitates the discovery of meaningful patterns within operational data.

The three clusters identified by the K-Means algorithm—representing high, medium, and low performance profiles—allow organizations to adopt differentiated strategies for training, supervision, and personnel recognition. This aligns with the observations of [Tobar-Díaz et al., 2023], who highlight the usefulness of machine-learning models in multivariate environments. Likewise, the digitalization strategy presented in this study responds to the recommendations of [Díaz-Ramírez, 2021], who emphasizes that the convergence of machine learning, visualization, and automation will serve as a foundational pillar of future industrial systems. This technological integration also promotes meritocracy and transparency in personnel management, aligning with the continuous-improvement principles of Lean Manufacturing described by [Chávez, 2022].

The results demonstrate the value of applying artificial intelligence to performance evaluation in industrial environments. The strong agreement between the Random Forest model and historical supervisory assessments, combined with the clustering insights provided by K-Means, shows that traditional and subjective evaluation methods can be replaced with more objective, consistent, and continuous digital tools.

However, previous comparative studies show that machine-learning performance in industrial settings can vary considerably. [Kharitonov et al., 2022] evaluated ten algorithms for anomaly detection in manufacturing and reported strong differences in accuracy depending on data characteristics and noise levels. While models such as AutoEncoder and Feature Bagging showed limited applicability, KNN unexpectedly performed best. In contrast, the Random Forest and K-Means combination in this study delivered stable and interpretable results, suggesting that the proposed framework is well-suited to heterogeneous operator-performance data.

Conclusions

The system developed represents an initial step toward the intelligent automation of operational performance evaluation in industrial environments. Although this phase was validated only as a functional prototype, the results demonstrate its technical feasibility and its potential to replace traditional methods based on subjective assessments. The proposed architecture enables the solution to scale toward real-world implementation, with real-time integration of artificial intelligence models, continuous data processing, and automatic updating of key performance indicators.

Furthermore, the combination of supervised and unsupervised models proved effective for classifying and segmenting operators based on objective evidence, opening the possibility of applying differentiated strategies for training, task assignment, and continuous improvement. The integration of interactive dashboards strengthens the transparency of the process and facilitates the interpretation of results by supervisors and talent-management areas.

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This work lays the foundation for future research aimed at incorporating new predictive models, expanding the number of indicators analyzed, integrating IoT sensors or MES systems, and evaluating the system's impact on real productivity once deployed in an operational environment. The findings confirm that the adoption of intelligent tools is not only possible but necessary to enhance industrial competitiveness within the framework of digital transformation.

Declarations

Conflict of interest

The authors declare no interest conflict. They have no known competing financial interests or personal relationships that could have appeared to influence the article reported in this article.

Author contribution

Vázquez de los Santos, Laura Cristina: Supervised the methodological design and scientific rigor of the study, validated the theoretical framework, and conducted the critical review.

Burciaga Alarcón, Ricardo: Led the technical development of the system, including the conceptualization, architectural design, implementation of machine-learning models, and preparation of the initial manuscript draft.

Rodríguez Silva, Jesús Rolando: Contributed to data collection and preparation, KPI structuring, and preliminary performance analysis.

Rodríguez Arzola, Adrián: Supported backend and database integration, performed technical validation of the prototype, and assisted in assessing its operational feasibility.

Availability of data and materials

All data used for this research were derived from our own data analysis, no information from third parties was used.

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Abbreviations

ANN	Artificial Neural Network
KPI	Key Performance Indicator
RF	Random Forest
s	seconds
%	percentage

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Article

Physicochemical characterization of goat whey from different cheese processes

Caracterización Fisicoquímica del lactosuero caprino de diferentes procesos de elaboración de queso

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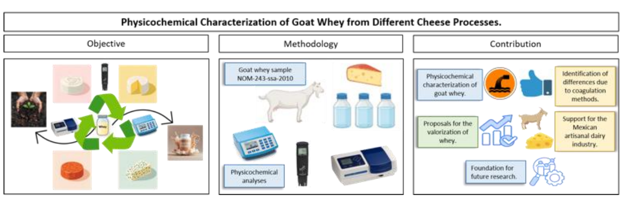
Abstract

This study characterizes the physicochemical properties of goat whey from four cheese processes (spreadable cream, Camembert, lactic-set, and feta) at an artisanal dairy in Queretaro, Mexico. Parameters including pH, electrical conductivity, total dissolved solids, soluble proteins, nitrates, phosphates, and potassium were analyzed using standardized methods (NOM-243-SSA1-2010). Results show variations: pH (3.20–4.20), electrical conductivity (7.30–9.43 mS/cm), total dissolved solids (3.50–4.70 ppt), and soluble proteins (1.50–2.07 mg/mL), influenced by coagulation methods (acidic, mixed, or enzymatic) and brine addition in feta. These differences highlight whey's potential for applications such as functional foods, animal feed, or fertilizers, promoting its valorization. This work supports sustainable practices in the mexican dairy industry, fostering a circular economy through byproduct reuse and waste reduction.



Goat whey, Cheese-making, Physicochemical properties

Resumen

Este estudio caracteriza propiedades fisicoquímicas del lactosuero caprino derivado de cuatro procesos de elaboración de queso (crema untable, camembert, coagulación láctica y feta) de una lechería artesanal en Querétaro, México. Se analizaron parámetros como pH, conductividad eléctrica, sólidos disueltos totales, proteínas solubles, nitratos, fosfatos y potasio, siguiendo métodos estandarizados (NOM-243-SSA1-2010). Los resultados revelaron variaciones: pH (3.20-4.20), conductividad eléctrica (7.30-9.43 mS/cm), sólidos disueltos (3.50-4.70 ppt) y proteínas solubles (1.50-2.07 mg/mL), influenciadas por los métodos de coagulación (ácida, mixta o enzimática) y la adición de salmuera en feta. Estas diferencias destacan el potencial del lactosuero para aplicaciones como alimentos funcionales, alimentación animal o fertilizantes, promoviendo su valorización. Este trabajo fomenta prácticas sostenibles en la industria láctea mexicana, apoyando la economía circular mediante la reutilización de subproductos y la reducción de residuos.

Lactosuero caprino, Elaboración de queso, Propiedades Fisicoquímicas

Area: Physicochemical characterization of goat whey

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Introduction

Whey, the liquid by-product of cheese production, represents both an environmental challenge and an untapped economic opportunity due to its high organic load, with a biochemical oxygen demand (BOD) often exceeding 40,000 mg/L, rendering it a significant pollutant if improperly managed [1]. In Mexico, goat cheese production has gained prominence, particularly within small- and medium- scale agro-industries, contributing to local economies and cultural heritage [2].

However, the whey generated during these processes is frequently discarded without further use, wasting valuable components such as lactose, soluble proteins (e.g., β-lactoglobulin, α-lactalbumin), and minerals like calcium, potassium, and phosphorus [3, 4]. This practice not only exacerbates environmental issues but also overlooks the potential for whey valorization in applications such as functional foods, animal feed, or biogas production [5, 6].

The physicochemical properties of whey vary significantly depending on the cheese-making process, which can involve enzymatic, acidic, or mixed coagulation methods. These variations influence the composition of whey, including pH, conductivity, protein content, and mineral profiles, which in turn affect its potential for reuse or treatment [7, 8].

While previous studies have characterized whey from cow's milk or single types of goat cheese, there is a lack of comparative data on whey derived from diverse goat cheese production processes, particularly in the Mexican context [3].

This study addresses this gap by conducting a comprehensive physicochemical characterization of goat whey obtained from four distinct cheese-making processes -spreadable cream cheese, Camembert, lactic-set cheese, and feta- produced at a small-scale dairy in Queretaro, Mexico. By analyzing parameters such as pH, electrical conductivity, total dissolved solids, soluble proteins, and key minerals (nitrates, phosphates, and potassium), this work provides novel insights into the compositional differences driven by coagulation and processing methods.

In the Mexican context, goat milk production has shown sustained growth, with Querétaro emerging as a key region due to its artisanal cheese-making tradition and proximity to urban markets [2]. However, the management of whey remains a critical challenge for small-scale producers, who often lack infrastructure for its treatment or valorization. According to Rodríguez-Afonso, A. [6], the implementation of good manufacturing practices (GMP) in dairy processing units is essential to ensure whey quality from the moment of separation, minimizing microbial contamination and preserving functional components such as proteinas and minerals. This is particularly relevant in artisanal settings like Granja La Serpentina, where manual processes dominate and hygienic control directly influences whey composition.

Furthermore, recent studies have demonstrated the technical and economic feasibility of transforming whey into value-added products. For instance, León-López et al. [9] developed fermented whey-based beverages supplemented with hydrolyzed collagen, achieving high protein content (up to 9.75 g/L), enhanced antioxidant activity (up to 48% ABTS radical inhibition), and in vitro bioavailability exceeding 70%, making them suitable for functional food applications. Similarly, Rodríguez-Afonso [6] designed a biogas plant for treating organic by-products, including whey, at the Benijos cheese factory (La Orotava), achieving CO_2 emission reductions and generating digestate as organic fertilizer, with a projected investment and energy return based on renewable biogas. These initiatives align with the principles of circular economy and sustainable development promoted by SECIHTI, emphasizing the reuse of agro-industrial by products to generate renewable energy and reduce environmental impact.

This study contributes to this framework by providing comparative data on whey from four cheese-making processes, enabling the identification of optimal valorization routes based on physicochemical profiles. The central hypothesis of this study is that differences in cheese-making techniques significantly alter the physicochemical properties of goat whey, enabling the identification of tailored strategies for its treatment or valorization.

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This research not only contributes to the scientific understanding of goat whey but also supports sustainable practices in the mexican dairy industry by proposing alternatives for whey management. By comparing whey from multiple cheese types, this study offers a unique perspective on its potential applications, aligning with global efforts to reduce food industry waste and promote circular economy principles [1, 5, 6].

Methodology

Samples collection. Whey samples were collected from the production of spreadable cream cheese, camembert, lactic-set cheese, and feta, crafted at Granja la Serpentina, located in El Marques, Queretaro, Mexico. The samples were obtained directly during the whey separation in the artisanal cheese-making process using goat´s milk, following the guidelines of NOM-243-SSA1-2010 [8], a Mexican standard for daily products safety and quality, to ensure representativeness and hygiene during collection. Each sample (n=3 per cheese type) had a volume of 500 mL and was collected in autoclaved glass jars. The samples were kept at 4 ºC during transport to the laboratory and analysed within 24 hours of collection to minimize microbial growth or chemical degradation.

Physicochemical Analyses

The following parameters were evaluated:

– **pH, Electrical conductivity and Total dissolved solids:** Calibrated with buffer solutions of pH 4.0 and 7.0 at 20 °C (Hanna Instruments, model HI-98129).

– **Soluble proteins:** Quantified by the Bradford method [10], using standard solutions of bovine serum albumin (BSA) prepared from a 2 mg/mL stock solution. A calibration curve was constructed with known BSA concentrations. Absorbance was measured at 595 nm using a UV-Vis spectrophotometer, and results were expressed in mg/mL.

– **Nitrate, phosphate, and potassium:** Quantified using a Multiparameter Photometer for Nutrient Analysis (Hanna Instruments, model HI-83200) with specific reagent kits for each analyte provided by the manufacturer. Results were expressed in mg/L [11].

– **Statistical Analysis.** To evaluate the differences in the physicochemical parameters of goat whey according to cheese type, a one-way analysis of variance (ANOVA) was performed at a 95% significance level ($p < 0.05$). When significant differences were identified, a post-hoc Tukey's mean comparison test was conducted to determine specific differences between cheese types. All statistical analyses were performed using Minitab Statistical Software (version 22). Results are presented as mean values ± standard deviation (n = 3 per cheese type) in the results section.

Results

The physicochemical composition of goat whey from four cheese-making processes (spreadable, camembert, lactic, and feta) was analyzed, revealing significant variations attributable to differences in coagulation and processing methods (Table 1, mean values ± standard deviation, n=3 per cheese type). The results for pH, electrical conductivity, total dissolved solids (TDS), soluble proteins, nitrates, phosphates, and potassium are presented below, with comparisons to literature values for sweet whey.

Box 1

Table 1

Results of the physicochemical analyses

Physicochemical Analyses	Spreadable cream	Camembert	Lactic-set	Feta
pH	4.20 ± 0.10^{a}	3.57 ± 0.11^{b}	3.20 ± 0.02^{c}	3.20 ± 0.02^{c}
Electrical conductivity (mS/cm)	7.3 ± 0.6^{b}	7.66 ± 0.10^{b}	7.37 ± 0.04^{b}	9.43 ± 0.09^{a}
Total dissolved solids (ppt)	3.50 ± 0.16^{c}	3.8 ± 0.04^{b}	3.76 ± 0.05^{bc}	4.70 ± 0.11^{a}
Soluble proteins (mg/mL)	2.07 ± 0.15^{a}	1.95 ± 0.02^{a}	1.50 ± 0.05^{b}	1.82 ± 0.09^{a}
Nitrate (mg/mL)	0.34 ± 0.11^{c}	3.20 ± 0.10^{b}	4.60 ± 0.01^{a}	1.40 ± 0.05^{c}
Phosphate (mg/mL)	$0.48\pm.00^{b}$	0.56 ± 0.03^{a}	0.15 ± 0.01^{d}	0.24 ± 0.01^{c}
Potassium (mg/mL)	1 ± 0^{c}	2.02 ± 0.50^{a}	1.81 ± 0.11^{b}	2.03 ± 0.50^{a}

a, b, c, d Means within a row not sharing a superscript letter differ significantly ($p<0.05$) according to Tukey´s HSD test.

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The pH ranged from 3.20 to 4.20, with spreadable cheese whey being the least acidic (4.20 ± 0.10), followed by camembert (3.57 ± 0.11), and lactic and feta wheys the most acidic (3.20 ± 0.02). These low pH values confirm the whey as acid whey, resulting from acidic (lactic, feta) or mixed (spreadable, camembert) coagulation processes, which enhance lactic acid production during fermentation or ripening. In contrast, sweet whey typically exhibits higher pH values (5.6–5.9) due to enzymatic coagulation with minimal acidification [5, 8].

Electrical conductivity varied from 7.30 to 9.43 mS/cm, with feta whey showing the highest value (9.43 ± 0.09 mS/cm), which differed significantly from the other types, while the remaining whyes showed similar values (7.3 – 7-66 mS/cm). The elevated conductivity in feta is attributed to brine addition, increasing sodium and chloride ions, while acidic or mixed coagulation in other processes reduces mineral solubility, particularly calcium and magnesium [9]. Sweet whey typically shows higher conductivity (8.0–11.0 mS/cm) due to greater mineral retention [5].

TDS ranged from 3.50 to 4.70 ppt, with feta whey having the highest (4.70 ± 0.11 ppt), differing significantly from the others, followed by camembert (3.80 ± 0.04 ppt), lactic (3.76 ± 0.05 ppt), and spreadable (3.50 ± 0.16 ppt). The high TDS in feta reflects increased mineral content from brine, while acidic coagulation reduces TDS in other wheys due to mineral precipitation. Sweet whey typically has higher TDS (60–92 ppt) [7].

Soluble proteins ranged from 1.50 to 2.07 mg/mL, with spreadable whey showing the highest content (2.07 ± 0.15 mg/mL), followed by camembert (1.95 ± 0.02 mg/mL), which did not differ significantly from feta (1.82 ± 0.09 mg/mL), but were higher than lactic (1.50 ± 0.05 mg/mL). Mixed coagulation in spreadable cheese retains more proteins (e.g., β-lactoglobulin, α-lactalbumin), while prolonged acidification in lactic whey increases protein precipitation into the curd. Sweet whey typically contains higher soluble proteins (6–10 mg/mL) due to minimal precipitation [9].

Mineral contents showed marked differences. Nitrates were highest in lactic whey (4.60 ± 0.01 mg/mL), differing significantly from the others, with the lowest in spreadable (0.34 ± 0.11 mg/mL). Phosphates were highest in camembert (0.56 ± 0.03 mg/mL) and lowest in lactic (0.15 ± 0.01 mg/mL), reflecting precipitation of calcium phosphates under acidic conditions. Potassium levels were similar in camembert (2.02 ± 0.50 mg/mL), lactic (1.81 ± 0.11 mg/mL), and feta (2.03 ± 0.50 mg/mL), but lower in spreadable (1.00 ± 0.01 mg/mL). Brine in feta enhances potassium retention, while acidic coagulation reduces mineral solubility [3].

These results highlight that acidic or mixed coagulation, along with brine addition in feta, significantly influences the physicochemical properties of goat whey, distinguishing it from sweet whey reported in the literature.

Conclusions

This study confirms that cheese-making techniques (acid, enzymatic, or mixed coagulation) significantly alter the physicochemical properties of goat whey, with variations in pH (3.20–4.20), electrical conductivity (7.30–9.43 mS/cm), dissolved solids (3.50–4.70 ppt), soluble proteins (1.50–2.07 mg/mL), and minerals such as nitrates, phosphates, and potassium.

These differences, influenced by acidification and brining (in feta), distinguish acidic whey from the sweet whey reported in literature, highlighting its lower protein and mineral content.

The results suggest valorisation opportunities: whey from spreadable cheese, rich in proteins, is ideal for functional foods, while feta whey, high in solids, could be used in animal feed or fertilizers.

For sustainable management, biological treatments are recommended. Future research on lactose profiles and the economic viability of these applications is suggested.

This work promotes sustainable practices in the mexican dairy industry, encouraging by product reuse and a circular economy.

Declarations

Funding

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Conflict of interest

The authors declare no conflict of interest. They have no known competing financial interests or personal relationships that could have appeared to influence the article reported in this article.

Author contribution

Mandujano-González, Virginia: Contributed to the idea of the project, manuscript elaboration, methodology, analysis of the results and conclusions.

Alvárez-Cervantes, Jorge and Alonso-Segura, Diana: Provided technical support.

Morales-Aguilar, Santiago: Contributed to the development and application of analytical methods.

Availability of data and materials

The data supporting the findings of this study, including raw measurements of physicochemical parameters (pH, electrical conductivity, total dissolved solids, soluble proteins, nitrates, phosphates, and potassium) are available from the corresponding author, Virginia Mandujano-González, upon reasonable request.

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Abbreviations

ANOVA: Analysis of Variance
BSA: Bovine Serum Albumin
HSD: Honestly Significant Difference
GMP: Good Manufacturing Practices

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Discussions

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Mandujano-González, Virginia, Álvarez-Cervante, Jorge, Morales-Aguilar, Santiago and Alonso-Segura, Diana. [2025]. Physicochemical characterization of goat whey from different cheese processes. 11[26] 1-6: e51126106.
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Journal of Research and Development 11[26]1-7: e61126107
Article

Mycosynthesis and Characterization of Silver Nanoparticles from *Phytophthora infestans* for Antimicrobial Evaluation Purposes

Micosíntesis y caracterización de nanopartículas de plata a partir de *Phytophthora infestans* con fines de evaluación antimicrobiana

Granados-Olvera, Jorge Alberto*[a], Vergara-Reyes, Hugo Yuriel[b], Rangel-Ruíz, Karelia Liliana[c] and Del Angel-Francisco, Lesly Alabel[d]

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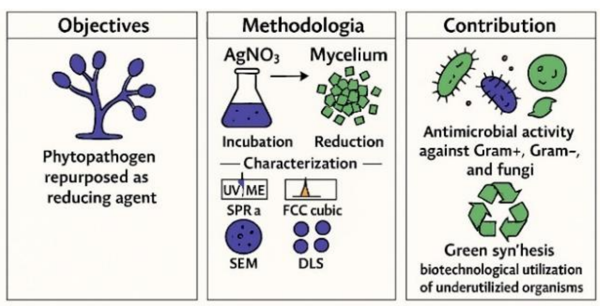
Abstract

The green synthesis of metal nanoparticles represents a sustainable alternative to conventional methods that employ toxic reagents and extreme conditions. In this study, Phytophthora infestans, a phytopathogenic oomycete, was used as a biological agent for the mycosynthesis of silver nanoparticles (AgNPs), taking advantage of its ability to secrete metabolites with reducing and stabilizing functions. The synthesis was carried out by incubating fungal mycelium in silver nitrate ($AgNO_3$) solutions at different concentrations, and the obtained AgNPs were characterized by UV-Vis spectroscopy, X-ray diffraction (XRD), scanning electron microscopy (SEM) and Dynamic Light Scattering (DLS).

Mycosynthesis and Characterization of Silver Nanoparticles from *Phytophthora infestans* for Antimicrobial Evaluation Purposes

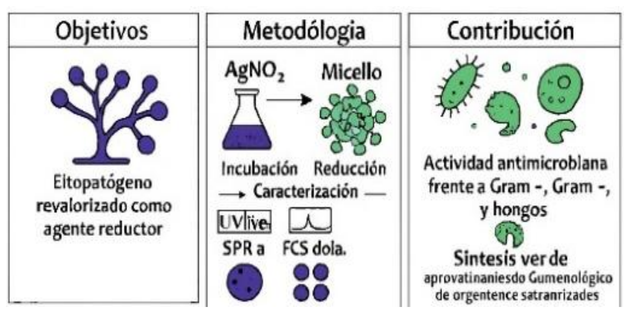


Mycosynthesis, Nanoparticles, Characterization

Resumen

La síntesis verde de nanopartículas metálicas representa una alternativa sustentable frente a los métodos convencionales que emplean reactivos tóxicos y condiciones extremas. En este estudio se empleó Phytophthora infestans, un oomiceto fitopatógeno, como agente biológico para la micosíntesis de nanopartículas de plata (AgNPs), aprovechando su capacidad para secretar metabolitos con funciones reductoras y estabilizantes. La síntesis se realizó mediante la incubación de micelio fúngico en soluciones de nitrato de plata ($AgNO_3$) a distintas concentraciones, y las AgNPs obtenidas fueron caracterizadas mediante espectroscopía UV-Vis, difracción de rayos X (DRX), microscopía electrónica de barrido (SEM) y Dispersión Dinámica de Luz (DLS).

Micosíntesis y caracterización de nanopartículas de plata a partir de *Phytophthora infestans* con fines de evaluación antimicrobiana



Micosíntesis, Nanopartículas, Caracterización

Area: Development of strategic leading-edge technologies and open innovation for social transformation

Citation: Granados-Olvera, Jorge Alberto, Vergara-Reyes, Hugo Yuriel, Rangel-Ruíz, Karelia Liliana and Del Angel-Francisco, Lesly Alabel. [2025]. Mycosynthesis and Characterization of Silver Nanoparticles from *Phytophthora infestans* for Antimicrobial Evaluation Purposes. Journal of Research and Development. 11[26] 1-7: e61126107.

Introduction

In recent decades, nanotechnology has emerged as a key discipline in the development of materials with unique properties at the nanoscale, opening new possibilities in fields such as medicine, electronics, agriculture and the environment (Khan et al., 2017). Within this area, metallic nanoparticles—especially silver nanoparticles (AgNPs)—have garnered significant attention due to their outstanding antimicrobial, optical and catalytic properties (Rai et al., 2009). However, conventional methods of AgNP synthesis often involve the use of toxic reducing agents, elevated temperatures and extreme pH conditions, raising environmental concerns and limiting their applicability in sustainable contexts (Iravani, 2011).

In light of this, green synthesis of nanoparticles has emerged as a promising alternative, based on the use of living organisms or natural extracts capable of reducing metal ions and stabilising the nanoparticles formed (Gómez, 2025). Among the various biological agents used, fungi and oomycetes stand out for their high efficiency in the production of nanomaterials, thanks to their ability to secrete enzymes, proteins and secondary metabolites with reducing and capping functions (Narayanan & Sakthivel, 2010). These organisms not only allow for a more environmentally friendly synthesis, but also offer advantages such as scalability, process simplicity and the possibility of controlling the size and morphology of the nanoparticles.

Phytophthora infestans, a phytopathogenic oomycete known for causing late blight disease in potato and tomato crops, has traditionally been studied for its agricultural impact (Moreno & Guerrero, 2025). However, its complex metabolism and its ability to produce bioactive compounds make it an interesting candidate for the biosynthesis of metallic nanoparticles (Guerrero, 2025). Although its use in nanotechnology is still in its early stages, recent research suggests that phytopathogenic organisms can be revalued as biotechnological tools, contributing to the circular economy and the utilisation of underutilised biological resources (Singh et al., 2016).

AgNPs synthesised by biological methods have demonstrated remarkable antimicrobial activity against Gram-positive and Gram-negative bacteria, as well as against pathogenic fungi.

This activity is attributed to multiple mechanisms, including the generation of reactive oxygen species (ROS), the alteration of cell membrane permeability, the interaction with proteins and nucleic acids, and the inhibition of essential metabolic processes (Lara et al., 2010). These properties make AgNPs especially useful in biomedical applications, such as antibacterial coatings, controlled drug delivery systems and medical devices.

The present work aims at the mycosynthesis of silver nanoparticles using extracts of Phytophthora infestans, their characterisation by spectroscopic and microscopic techniques—including UV-Vis Spectroscopy, X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), and Dynamic Light Scattering (DLS)—, as well as the evaluation of their antimicrobial activity against. This proposal seeks not only to contribute to the development of functional nanomaterials through sustainable processes, but also to explore the biotechnological potential of organisms traditionally considered harmful, promoting a more integrated and ecological vision of materials science.

Theoretical Framework

The synthesis of silver nanoparticles (AgNPs) has captured the attention of the scientific community for their antimicrobial, catalytic, and optical properties. Traditionally, these nanoparticles have been produced using physical-chemical methods that, while effective, involve the use of toxic reagents, high temperatures, and unsustainable conditions. In response to this problem, a new research trend has emerged focused on green synthesis, seeking more environmentally friendly alternatives (Nevarez, 2025).

Among the most explored strategies are the use of plant extracts, bacteria, fungi, and yeasts. Fungi, in particular, have demonstrated a remarkable ability to reduce metal ions and stabilize nanoparticles thanks to their secretion of enzymes and secondary metabolites.

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However, within this group, phytopathogens have been scarcely considered, despite their biotechnological potential.

Phytophthora infestans, primarily known for its devastating role in potato and tomato crops, represents a paradigmatic example of how an organism traditionally associated with damage can be repurposed as an ecological nanofabricator. Recent studies have begun to explore its ability to synthesize AgNPs, revealing an efficiency comparable to that of saprophytic fungi, with the added advantage of harnessing an organism that is already part of the agricultural ecosystem.

Furthermore, research such as that by Singh et al. (2016) has shown that AgNPs obtained by biological methods not only exhibit controlled morphology and good dispersion, but also superior antimicrobial activity against resistant strains. These findings open the door to applications in agriculture, medicine, and water treatment, consolidating the role of phytopathogens as agents of innovation in green nanotechnology (Contreras, 2025).

Methodology

The synthesis of silver nanoparticles (AgNPs) was carried out through a biogenic approach using the fungus *Phytophthora infestans* as a reducing agent of silver nitrate ($AgNO_3$).

Box

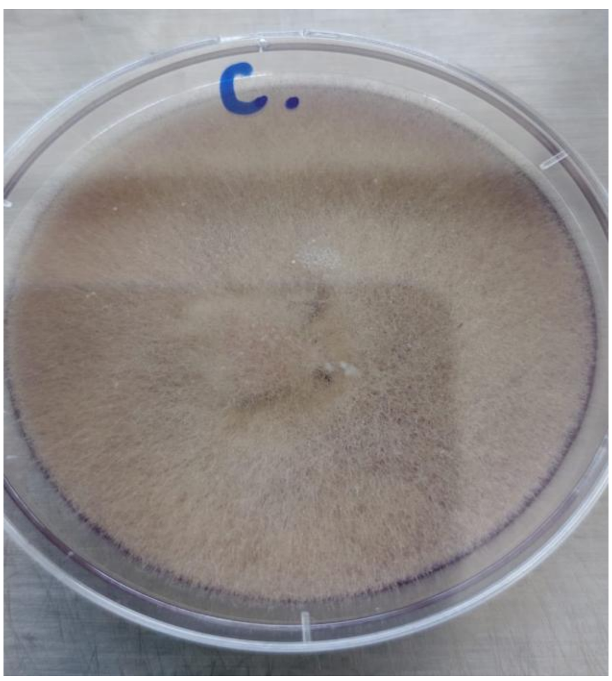

Figure 1
Fungus *Phytophthora infestans*
Own Source

The fungal strain was cultivated in PDA medium for 7 days, after which the mycelium was separated and subjected to a two-stage washing process: first with distilled water (24 h at 130 rpm) and subsequently with double-distilled water (24 h at 150 rpm). After washing, four $AgNO_3$ solutions were prepared at different concentrations (30 mM, 40 mM, 50 mM and 60 mM), into which the mycelium was incorporated and kept under constant stirring for 72 hours at 150 rpm to induce the reduction of the metal precursor.

Box

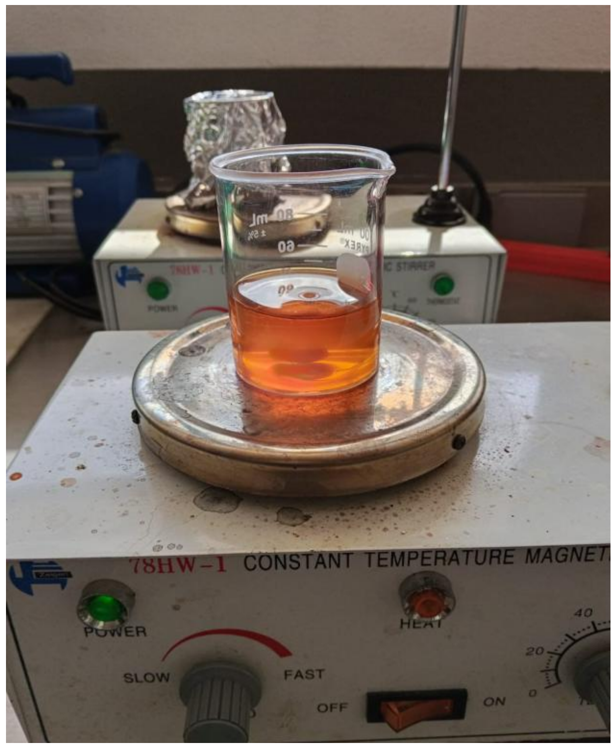

Figure 2
Silver nanoparticles
Own Source

Results

UV-Vis Spectroscopy of Silver Nanoparticles

Box

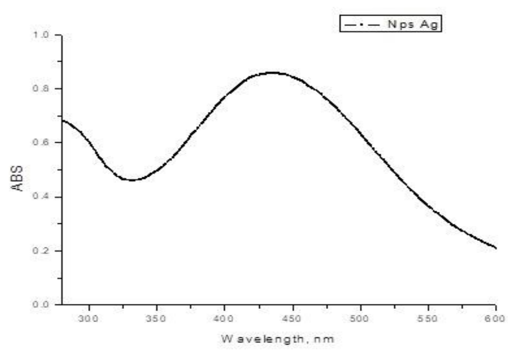

Figure 3
UV-Vis spectrum obtained from silver nanoparticles
Own Source

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The optical characterisation of the silver nanoparticles (AgNPs) obtained by mycosynthesis with Phytophthora infestans was performed by UV-Vis spectroscopy in the range of 300 to 600 nm. The spectrum obtained showed a well-defined absorbance peak around 450 nm, which is characteristic of the surface plasmon resonance (SPR) associated with AgNPs (Rai et al., 2009).

This peak indicates the presence of silver nanoparticles with a relatively uniform size distribution, as no shifts towards longer wavelengths or multiple peaks suggesting aggregation or polydispersity were observed. The intensity of the signal, with a maximum absorbance close to 1.0, suggests a high concentration of well-dispersed nanoparticles in the colloidal medium, which can be attributed to the stabilising action of the extracellular metabolites secreted by the oomycete during the synthesis process (Narayanan & Sakthivel, 2010).

The peak position at 450 nm also allows us to estimate that the average particle size is in the range of 20 to 60 nm, although this estimate will be corroborated by complementary techniques such as scanning electron microscopy (SEM) and X-ray diffraction analysis (XRD). These results confirm the effectiveness of the P. infestans extract as a reducing and stabilising agent in the green synthesis of AgNPs, and constitute a solid basis for its subsequent antimicrobial evaluation.

SEM Micrograph of Silver Nanoparticles

Box

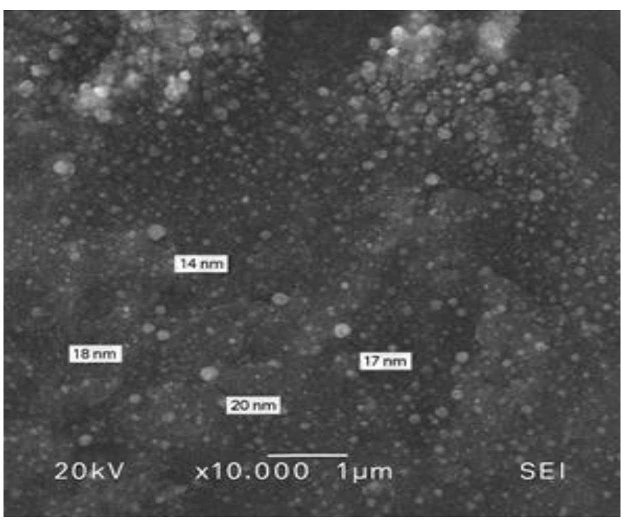

Figure 4
SEM micrograph obtained from silver nanoparticles
Own Source

The morphological characterisation of the silver nanoparticles (AgNPs) synthesised by mycosynthesis with *Phytophthora infestans* was performed by scanning electron microscopy (SEM), using an acceleration voltage of 20 kV and a magnification of x10,000. The image obtained, using the secondary electron mode (SEI), reveals a rough textured surface on which multiple particles of predominantly spherical morphology are distributed.

The nanoparticles are observed as bright and well-defined structures, with sizes estimated to be below 100 nm, dispersed heterogeneously on the substrate. In the upper left region of the micrograph, a higher density of particles is observed, while towards the lower right, the distribution becomes more dispersed. This variability suggests a possible influence of the oomycete's extracellular metabolites on the nucleation and growth of the particles.

The absence of significant aggregation and the uniformity in the spherical shape of the AgNPs indicate good colloidal stability, which is consistent with the results obtained by UV-Vis spectroscopy. The scale bar of 1 µm allows us to visually estimate that the particles are within the nanometric range, which will be corroborated by XRD analysis to determine the average crystalline size. These findings confirm the effectiveness of the P. infestans extract as a reducing and stabilising agent in the green synthesis of silver nanoparticles and support its potential application in subsequent studies of antimicrobial activity.

DRX of Silver Nanoparticles

Box

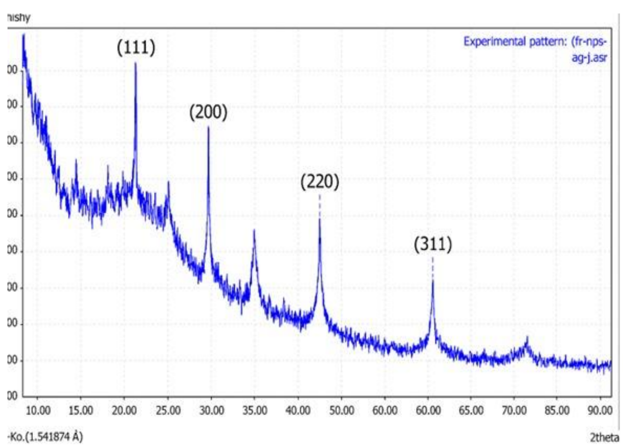

Figure 5
DRX obtained from silver nanoparticles
Own Source

X-ray diffraction analysis confirmed the formation of silver nanoparticles with a face-centered cubic (FCC) crystal structure. The diffraction pattern revealed four main peaks located approximately at 2θ = 38°, 44°, 64°, and 77°, corresponding to the (111), (200), (220), and (311) crystallographic planes, respectively. These values coincide with those reported in JCPDS card No. 04-0783 for Ag^0, indicating the presence of metallic silver without evident signs of oxidation.

The most intense peak, associated with the (111) plane, suggests a preferential orientation in this direction, a common phenomenon in green synthesis due to the specific interaction between biological reducing agents and crystalline surfaces. The absence of secondary peaks attributable to silver oxides (Ag_2O, AgO) reinforces the efficacy of the mycosynthesis process in obtaining pure nanoparticles. These results corroborate the fungal extract's ability to act as a reducing and stabilizing agent, promoting the formation of well-defined crystalline structures. The relative intensity of the peaks also suggests good crystallinity, which is favorable for antimicrobial and catalytic applications.

DLS of Silver Nanoparticles

Box

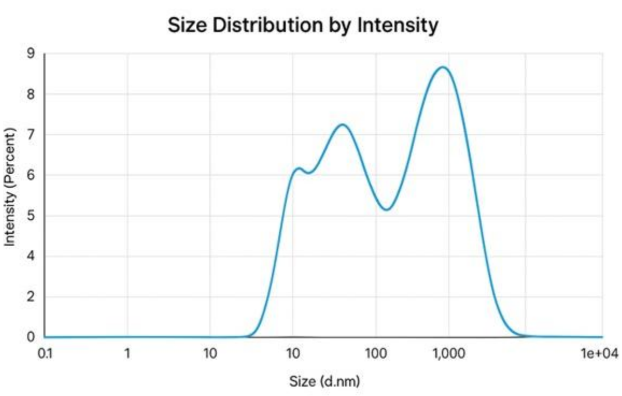

Figure 6
DLS obtained from silver nanoparticles
Own Source

Dynamic Light Scattering (DLS) analysis shows a multimodal distribution with three populations centered approximately at 10 nm, 100 nm, and 1000 nm, indicating significant heterogeneity in the hydrodynamic size of the synthesized nanoparticles.

– The peak at ~10 nm could correspond to individual nanoparticles well stabilized by fungal metabolites.
– The dominant peak at ~100 nm suggests the presence of partially aggregated nanoparticles or with coronas of organic compounds that increase their hydrodynamic size.
– The third peak at ~1000 nm likely represents larger aggregates or clusters, common in green syntheses where the stabilizing agents are natural and nonuniform.

Importantly, DLS measures hydrodynamic size, which includes not only the metallic core of the nanoparticle, but also the solvation shell and any molecules adsorbed on its surface. Therefore, the sizes reported by DLS are often larger than those observed using techniques such as SEM or TEM.

The observed dispersion can be attributed to variability in the composition of the fungal extract, temperature, and reaction time. These results reinforce the need to optimize synthesis conditions to obtain a more monodisperse distribution.

Conclusions

This research demonstrates that *Phytophthora infestans* can be effectively used as a reducing and stabilizing agent in the mycosynthesis of silver nanoparticles (AgNPs), offering a sustainable alternative to conventional chemical synthesis methods. Spectroscopic and structural characterization confirmed the formation of AgNPs with well-defined optical and crystalline properties, evidenced by the SPR peak at 450 nm and the face-centered cubic (FCC) structure observed in XRD analysis.

SEM micrographs revealed a predominantly spherical morphology and a homogeneous distribution of particles smaller than 100 nm, while DLS analysis indicated multimodal dispersion in hydrodynamic size, attributable to the complexity of the fungal extract and the synthesis conditions. These results suggest good colloidal stability, although they also point to the need to optimize experimental parameters to improve monodispersity.

Granados-Olvera, Jorge Alberto, Vergara-Reyes, Hugo Yuriel, Rangel-Ruíz, Karelia Liliana and Del Angel-Francisco, Lesly Alabel. [2025]. Mycosynthesis and Characterization of Silver Nanoparticles from *Phytophthora infestans* for Antimicrobial Evaluation Purposes. Journal of Research and Development. 11[26] 1-7: e61126107.
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Taken together, the findings support the antimicrobial potential of the obtained AgNPs, as well as their applicability in biomedical and agricultural contexts. Furthermore, this study proposes a biotechnological revaluation of phytopathogenic organisms such as P. infestans, traditionally considered harmful, promoting a more integrative and ecological approach to the development of functional nanomaterials.

Declarations

Conflict of interest

The authors declare no interest conflict. They have no known competing financial interests or personal relationships that could have appeared to influence the article reported in this article.

Author contribution

Granados-Olvera, Jorge Alberto: Contributed to the project idea, synthesis method and the samples characterization.

Vergara- Reyes, Hugo Yuriel: Contributed to the mycosynthesis, experimentation, and writing of the article.

Rangel- Ruíz, Karelia Liliana: Contributed to the development and evaluation in microbiological tests.

Del Angel-Francisco, Lesly Alabel: Contributed to the mycosynthesis, experimentation, and translation of the article.

Availability of data and materials

Indicate the availability of the data obtained in this research.

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Abbreviations

AgNP	Silver nanoparticles
DLS	Dynamic Light Scattering
ROS	Reactive Oxygen Species
SEM	Scanning Electron Microscopy
UV-VIS	Ultraviolet-Visible
XRD	X-ray diffraction

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Antecedents

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Supports

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Nevarez, J. A. V. (2025). Biosíntesis de nanopartículas de plata y óxido de cobre: caracterización y efecto antifúngico sobre Malassezia pachydermatis.

Differences

Contreras González, N. A. (2025). Verificación de la potencialización de la germinación de maíz chalqueño con el uso del aislado fúngico CMP4, obtenido en un medio con nanopartículas de plata “BioArgovit®”.

Discussions

Singh, P., Kim, Y. J., Zhang, D., & Yang, D. C. (2021). Biological synthesis of nanoparticles from plants and microorganisms. Trends in Biotechnology, 39(6), 629–640.

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Journal of Research and Development 11[26]1-9: e71126109
Article

Obtaining and characterization of terephthalic acid via acid and basic hydrolysis of recycled poly (ethylene terephthalate)

Obtención y caracterización de ácido tereftálico mediante hidrólisis ácida y básica de poli (etilen tereftalato) reciclado

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Subdiscipline: Organic Chemistry

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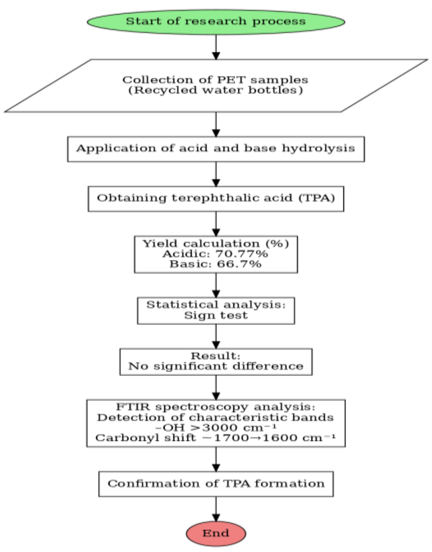
Abstract

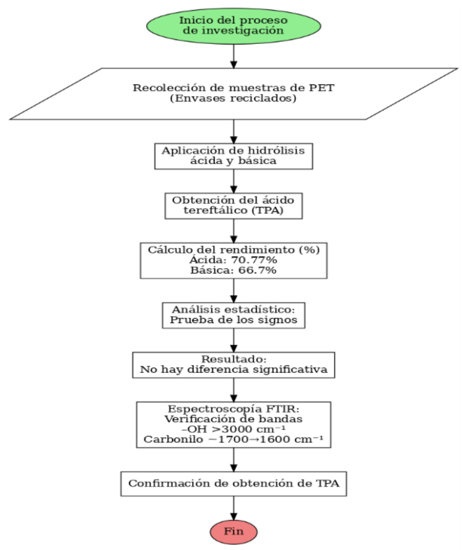
Acid-base hydrolysis is a methodology that can be used for the chemical treatment of plastic waste. This research aims to apply the mentioned methodology in poly (ethylene terephthalate) PET from reused plastic bottles to obtain terephthalic acid (TPA) under acid and basic hydrolysis conditions. Subsequently, the performance of both methodologies was compared. On average, performance percentages of 70.77% and 66.7% respectively were obtained. These data were statistically analyzed using the sign test, finding that there are no significant differences between the medians of the data sets studied. The analysis by FTIR spectroscopy of the products obtained, specifically through the appearance of the characteristic band for –OH corresponding to carboxylic acid above 3000 cm-1 and the shift of the carbonyl group band from approximately 1700 cm-1 (type ester) up to approximately 1600 cm-1 (acid) allowed us to verify the obtaining of TPA.



Hydrolysis, terephthalic acid, FTIR spectroscopy

Resumen

La hidrólisis ácido-base es un método de reciclaje utilizado para tratar residuos plásticos. Esta investigación se centró en aplicar dicha metodología en plásticos de poli (etilen tereftalato) (PET) recolectados de envases para el comercio de agua purificada, con el objetivo de obtener ácido tereftálico (TPA) bajo condiciones de hidrólisis ácida y básica. Posteriormente, se comparó el rendimiento de ambas metodologías. En promedio, se obtuvieron porcentajes de rendimiento de 70.77% y 66.7% respectivamente, estos datos se analizaron estadísticamente mediante la prueba de los signos, encontrándose que no hay diferencias significativas entre las medianas de los conjuntos de datos. El análisis por espectroscopía de FTIR, de los productos obtenidos, específicamente mediante la aparición de la banda característica para –OH correspondiente a ácido carboxílico sobre los 3000 cm^{-1} y el desplazamiento de la banda del grupo carbonilo desde aproximadamente 1700 cm^{-1} (tipo éster) hasta aproximadamente 1600 cm^{-1} (ácido) permitieron comprobar la obtención del TPA.



Hidrolisis, ácido tereftálico, espectroscopia FTIR

Area: Promotion of frontier research and basic science in all fields of knowledge

Citation: De la Cruz, Boris, Domínguez, Ricardo and Moreno, Juan. [2025]. Obtaining and characterization of terephthalic acid via acid and basic hydrolysis of recycled poly (ethylene terephthalate). Journal of Research and Development. 11[26]1-9: e71126109.

Introduction

Plastics have revolutionized our society, enabling the cost-effective manufacturing of a variety of materials with countless applications across diverse industries. The industrial-scale production of plastics significantly began to expand around 1950, when global plastic production reached 2 million tons per year (Green Chem., 2022, 24, 8899).

Currently, the accumulation of plastics in soil and aquatic bodies constitutes a global contamination problem, driven by the excessive amount of plastic waste resulting from various anthropogenic activities in these environments. This accumulation poses a serious risk to the environment and ecosystems, as plastics can enter the trophic chain, leading to detrimental effects such as ingestion and subsequent mortality in organisms. (Green Chem., 2022, 24, 8899; Castaño et al., 2018).

As of 2015, an astonishing 8.3 billion metric tons of plastics were estimated to have been manufactured. Furthermore, in 2017, 35.4 million tons of plastic were produced in the United States. To date, billions of tons of plastics have been produced, and the progressive accumulation of plastic waste over the past decades has contributed significantly to this issue (Lucas et al., 2021; Tessa et al., 2020; Kayee et al., 2021).

Poly(ethylene terephthalate) (PET), polyethylene (PE), polypropylene (PP), polystyrene (PS), polyvinyl chloride (PVC), and nylon (NYL) are the most common polymers present in plastic waste, primarily in both terrestrial environments and marine life (Diaz-Silvarrey et al., 2018; Dorin & Danuta, 2021). Their massive use has led to a significant increase in plastic waste, with an average annual growth rate of 8.7% (Mecozzi & Nisini, 2019). Given the depletion of oil resources and the increasing environmental impact, the recovery of monomers from plastic waste has become more necessary than ever. (Diaz-Silvarrey et al., 2018; Mecozzi & Nisini, 2019).

El Salvador, like many other nations globally, exhibits significant consumption of plastic material, the fragmented particles of which accumulate in various ecosystems, including the littoral zone.

Specifically, the occurrence of microplastics in Pacific Ocean surface water was confirmed within the Los Cóbanos Protected Natural Area in El Salvador. Furthermore, in 2017, a total of 21.7 g of plastic waste was reported within a 38,400 cm^2 area across different beaches along the country’s coast, based on measurements conducted during the 2016 wet and dry seasons. (Barraza, 2017).

"In the European Union (EU) and the United Kingdom (UK), plastic waste is estimated to account for up to 10–13% of Municipal Solid Waste (MSW), of which 7% (1.7 million tons) is Poly(ethylene terephthalate) (PET). Furthermore, of the plastic waste generated in the United States, 5 million tons was composed of PET (Tessa et al., 2020; Diaz-Silvarrey et al., 2018).

PET is a linear thermoplastic polymer formed by the reaction of terephthalic acid (TPA) and ethylene glycol (EG), or through the transesterification of dimethyl terephthalate and EG (Alnaqbi et al., 2015), PET is widely utilized in textiles, polyester fibers, food packaging, plastic films, electronic devices, mechanical equipment, and other fields, owing to its favorable properties such as chemical and thermal resistance, low CO_2 permeability, and lightness, among others (Yang et al., 2021; Barredo et al., 2023; Raheem et al., 2019; Čolnik et al., 2022; RSC Adv., 2018, 8, 8209). The chemical structure of the PET monomer unit is shown in Figure 1:

Box 1

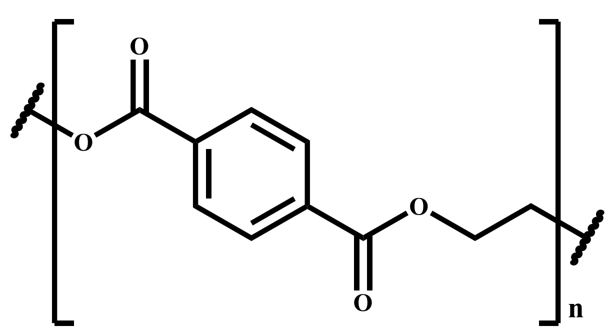


Figure 1
Chemical Structure of Polyethylene Terephthalate
Source: Authors

The majority of global PET manufacturing is allocated to the production of synthetic fibers (over 60%), while bottle production accounts for approximately 30% of the worldwide demand (Cosimbescu et al., 2021).

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The final disposal of PET is typically recycling, which is carried out in different plants that process the material for re-employment, such as in the packaging industry. However, recycled PET (rPET) cannot be used directly for food packaging due to hygiene concerns (Barraza, 2017; Ortiz-Pech et al., 2020; Das & Tiwari, 2019). In turn, various methodologies exist for the treatment of plastic waste such as PET, including mechanical, chemical, and enzymatic methods, among others. The chemical modification of PET represents an alternative for the final disposal of this material, since its monomers (TPA and EG) can serve as a starting point for obtaining materials with a diversity of applications (Dorin & Danuta, 2021; RSC Adv., 2018, 8, 8209).

One of the most promising chemical processes for the depolymerization of PET is hydrolysis. This process, whether conducted in the laboratory or in industrial plants, is typically performed at high temperatures and pressures under acid, basic, or neutral catalysis (Dorin & Danuta, 2021).

The basic hydrolysis of PET (Figure 2) is typically carried out using an aqueous solution of NaOH or KOH at a concentration of 4–20 wt% (Al-Sabagh et al., 2016). The reaction products are EG and the disodium or dipotassium terephthalate salt (TPA-Na2 and TPA-K2, respectively). Pure TPA can then be obtained by neutralizing the reaction mixture with a strong mineral acid (for example, H_2SO_4) (Karayannidis et al., 2002).

Box 2

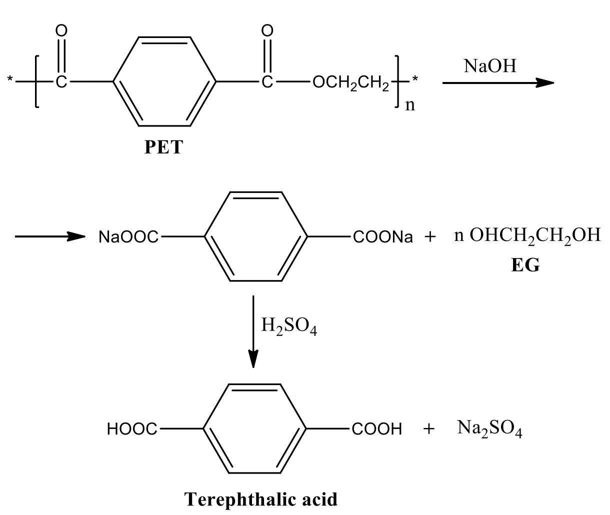

Figure 2

Basic Hydrolysis of PET using NaOH or KOH, adapted from [20]

Source: Authors

In the acid hydrolysis of PET waste, H_2SO_4, HNO_3, HCl, and H_3PO_4 are commonly used (Al-Sabagh et al., 2016; Islam et al., 2023). Acid hydrolysis is most frequently performed using concentrated sulfuric acid, as shown in Figure 3 (Al-Sabagh et al., 2016).

Box 3

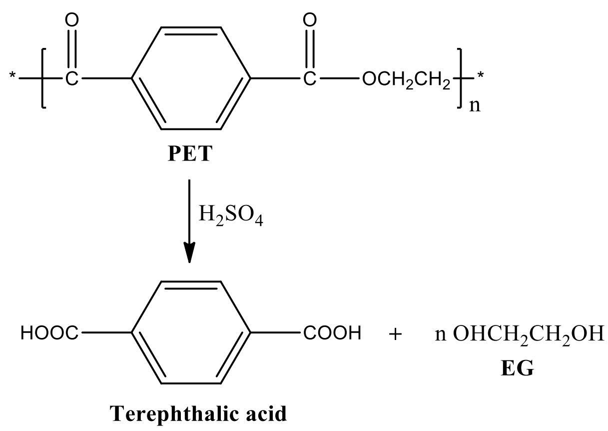

Figure 3

Basic Hydrolysis of PET using H_2SO_4 adapted from [20]

Source: Authors

The reaction products are EG and TPA. However, the process proves costly due to the requirement for recycling large quantities of concentrated H_2SO_4 and the subsequent purification of EG from the sulfuric acid (Al-Sabagh et al., 2016).

The purpose of this research is to perform the chemical recycling of PET bottles on the campus of the Universidad Salvadoreña Alberto Masferrer, by comparing straightforward basic and acid hydrolysis methodologies and employing a statistical test to determine which hydrolysis type yields the highest conversion percentage of PET to TPA. The ultimate goal is to present an alternative option for the recycling and treatment of PET-based plastic waste in El Salvador.

Methodology

Materiales

During the experimental development of this research, the following reagents were used: Sodium hydroxide pellets (P.A., Merck, Lot 106498, CAS 1310-73-2), sulfuric acid 95–98 wt% (P.A., Merck, Lot 100731, CAS 7664-93-9), butan-1-ol (P.A., Merck, Lot 101990, CAS 71-76-3), and potassium hydroxide flakes (P.A., Merck, Lot 105029, CAS 1310-58-3).

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PET Samples

PET derived from fragmented plastic bottles collected at the Universidad Salvadoreña Alberto Masferrer was used. The inclusion criterion for the plastic bottles required them to be colorless and composed exclusively of PET. These bottles were subsequently washed with soap and water and cut into fragments of approximately 0.25 cm^2.

Basic Hydrolysis of PET

Based on the methodology described by (Ramírez et al., 2010), the basic hydrolysis of PET was carried out by mixing approximately 1 g of PET with 40 mL of the 3.34 M KOH/1-butanol solution, as illustrated in Figure 4.

Box 4

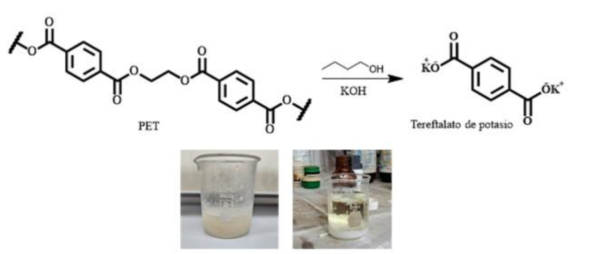

Figure 4
Basic Hydrolysis Process of PET

Source: Authors

The previously prepared mixture was maintained under constant stirring and heating at 110 °C for 50 minutes. Following this period, a white solid was obtained within the reaction mixture. To the previously described mixture, 40 mL of distilled water was added to dissolve the formed solid. From this new mixture, the aqueous phase was extracted and acidified with concentrated H_2SO_4 (Figure 5), resulting in the precipitation of the product. The product was subsequently isolated by filtration, washed with abundant distilled water, and analyzed using FTIR.

Box 5

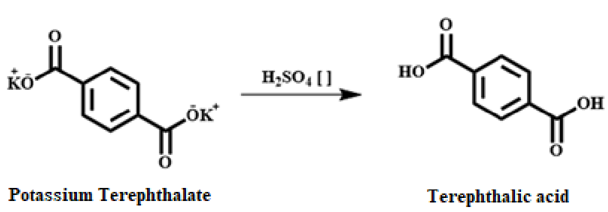

Figure 5
Neutralization equation of the dipotassium salt of terephthalic acid

Source: Authors

Acid Hydrolysis of PET

Following the methodology described by (Morales Palomino, 2010), acid hydrolysis of PET was carried out, a process shown in Figure 6. For this, approximately 1 g of PET was mixed with 40 mL of concentrated H_2SO_4. The resulting mixture was heated within a temperature range of 90-100 °C for 40 minutes, after which the complete dissolution of the plastic was observed. Subsequently, the mixture was cooled to room temperature and washed multiple times with distilled water until neutral pH was reached and no further precipitation of a white solid was observed. The obtained solid was filtered, washed with abundant distilled water, and finally analyzed using FTIR.

Box 6

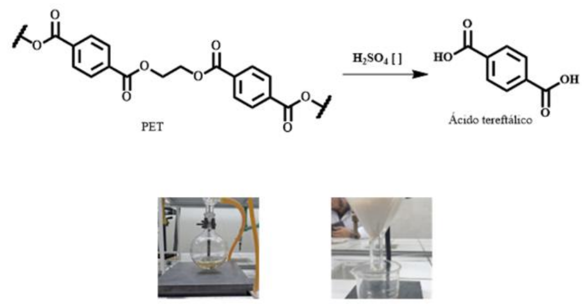

Figure 6
Acid hydrolysis process of PET

Source: Authors

FTIR Characterization

The Fourier-Transform Infrared (FT-IR) spectra of the terephthalic acid obtained from PET hydrolysis were recorded using a Thermo Scientific Nicolet iS5 equipped with an Attenuated Total Reflectance (ATR) unit (iD7), covering the range of 500 to 4000 cm^{-1}.

Statistical Analysis

Statistical analysis was performed using R (version 4.3.3, released in February 2024), an open-source software under the GNU General Public License. This environment was chosen due to its cost-effectiveness, flexibility, and the extensive tools it offers for conducting reproducible analyses and high-quality visualizations. Given the small sample size and the nature of the data, non-parametric methods were applied, specifically the sign test, to compare the medians of the yields obtained under acid and basic hydrolysis conditions.

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This statistical approach allowed for the evaluation of significant differences without requiring strict assumptions regarding data distribution. Furthermore, descriptive plots such as boxplots were generated, which clearly visualized the variability, interquartile ranges, and differences between both experimental conditions. The use of R not only ensured the transparency of the analysis but also favors its reproducibility.

Results

Hydrolysis

Tables 1 and 2 show the results obtained from the acid and basic hydrolysis methodologies employed, recording the mass of product achieved as well as the respective yield percentages:

Box 7

Table 1

Yield obtained from acid hydrolysis

N°	Hydrolysis		
	Acid		
	PET (g)	TPA (g)	Yield (%)
1	1.0180	0.7267	71.39
2	1.0175	0.7147	70.24
3	1.0182	0.7193	70.69

Source: Authors

Box 8

Table 2

Yield obtained from basic hydrolysis

N°	Hydrolysis		
	Basic		
	PET (g)	TPA (g)	Yield (%)
1	1.0059	0.6574	65.35
2	1.0034	0.6715	66.92
3	1.0015	0.6604	65.94

Source: Authors

The percentage yield was calculated according to the following formula:

$$\%\text{yield TPA} = \frac{\text{g TPA}}{\text{g PET}} \times 100 \quad [1]$$

Acid hydrolysis yielded better results concerning the recovery of TPA. This may be associated with the fact that TPA is obtained in the bulk of the reaction, whereas, in basic hydrolysis, TPA is first obtained as the dipotassium salt. This requires a liquid-liquid extraction prior to the precipitation of the TPA, a procedure in which small amounts of the dipotassium salt may be lost, thus affecting the process yield.

Statistical analysis

To compare the two related samples, a non-parametric test was applied using the R software (version 4.3.3, released in February 2024): the sign test. This test relies on the direction of change between the data pairs, without considering the magnitude of the change. Each pair of yield results for the two types of hydrolysis were compared, and a simple count was made of how many times the acid hydrolysis sample had a higher value than the basic hydrolysis sample, and vice versa.

Subsequently, the sign statistic is used to determine if there is a significant difference between the two samples. The sign test relies on the null hypothesis that there is no median difference between the two groups (acid hydrolysis and basic hydrolysis), and the alternative hypothesis that a difference does exist.

Box 9

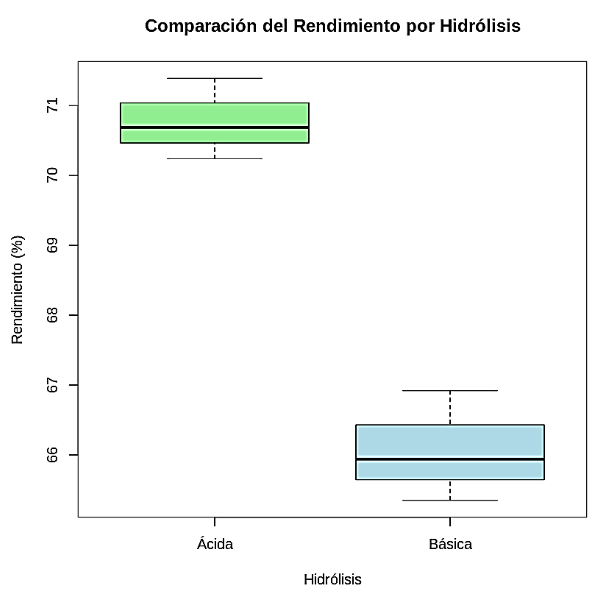

Figure 7

This graph allows a visual comparison of the medians, interquartile ranges, and possible outliers between the acid and basic hydrolysis conditions.

Source: Authors

For the yield results shown in Tables 1 and 2, three positive differences and zero negative differences were found between the acid yield and the basic yield.

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This indicates that, for all data pairs, the acid yield was consistently higher than the basic yield. Upon performing the sign test (using a binomial test, as the sign test can be considered a special case of the binomial test with p=0.5 under the null hypothesis), the resulting p-value was 0.25. This p-value represents the probability of obtaining a sign distribution as extreme as (or more extreme than) the one observed, assuming there is no true difference in the median between the two groups

Given that this p-value (0.25) is not generally considered sufficiently small to reject the null hypothesis (a threshold of 0.05 or 0.01 is typically used), there is insufficient evidence to conclude that there is a significant difference in the median yield between the acid and basic conditions with this small dataset.

Nevertheless, we can descriptively analyze and visualize the data conditions for acid and basic hydrolysis. See Figure 7. The box plot shown for the acid hydrolysis yield clearly demonstrates a higher median than the basic hydrolysis yield, which generally indicates a superior yield under acid conditions.

Analysis by FTIR-ATR Spectroscopy

The TPA obtained from the hydrolysis process was analyzed using Infrared Spectroscopy. The objective was to identify characteristic signals indicating the disappearance of the ester carbonyl bond and the emergence of a signal for the carboxylic acid group.

Consistent with similar research, these signals are expected to show a shift to lower wavenumbers for the C=O vibration of the carboxylic acid (1682 cm^{-1}) compared to the ester (1730 cm^{-1}). Additionally, the emergence of a broad band with overtones from 3300 to 2500 cm^{-1} is expected, corresponding to the O-H stretching vibration of a carboxylic acid (Ramírez y col., 2010; Sammon et al, 2000)

Figure 8 shows the IR spectra of the products obtained from acid (C) and basic (B) hydrolysis, as well as the IR spectrum of the collected PET (A).

Box 10

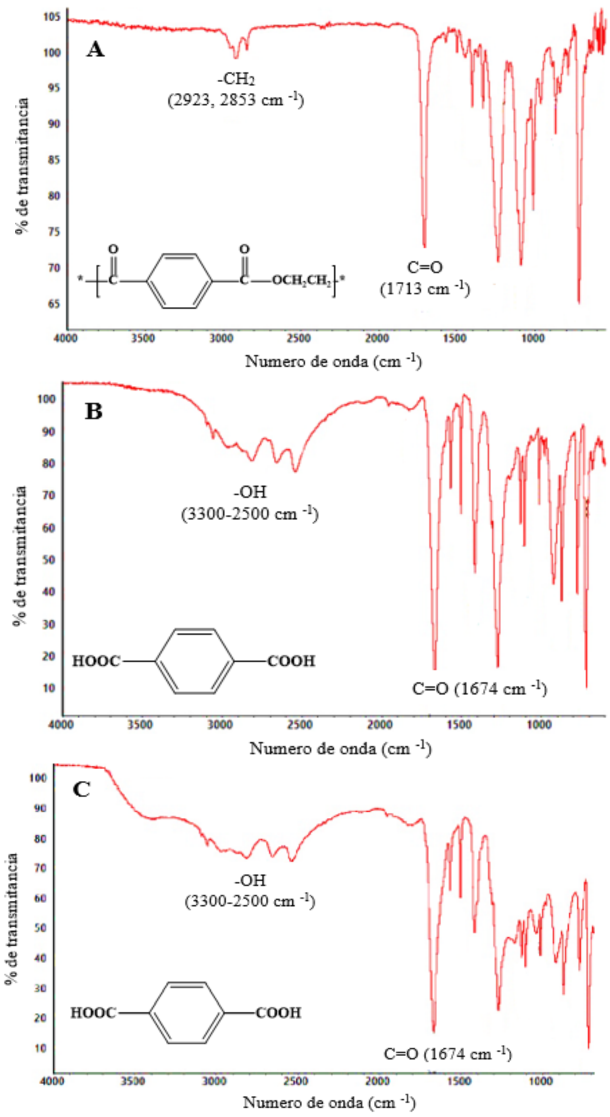

Figure 8

IR spectra of the collected PET sample (A), Basic hydrolysis product (B), and Acid hydrolysis product (C).

Source: Authors

In Spectrum A, the significant characteristic bands of PET are highlighted: the $-CH_2$ bond shows asymmetric stretching at 2923 cm^{-1} and symmetric stretching at 2853 cm^{-1}; the ester C=O stretching is found at 1713 cm^{-1}; the C=C stretching of the aromatic ring appears at 1408 cm^{-1}; and the in-plane C-H bending is observed in the region of 1241 to 1016 $cm^{-1.}$

In Spectra B and C, characteristic signals of TPA are shown. Both exhibit a broad band ranging from 3300 to 2500 cm^{-1}, corresponding to the O-H stretching vibration for acids; the C=O stretching vibration shifted to 1674 cm^{-1}; C=C stretching vibrations of the aromatic ring between 1574 and 1422 cm^{-1}; C-OH in-plane bending around 1423 cm^{-1}; and C-O stretching around 1279 and 1276 cm^{-1}.

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Conclusions

The collected PET, when subjected to acid and basic hydrolysis, yielded TPA as the final product, with average yields of 70.77% and 66.7%, respectively. Although the yields are very similar, the statistical analysis performed using the sign test found no significant difference between the medians of the yields from both treatments.

This indicates that, based on the available data, it cannot be asserted that one methodology is superior to the other in terms of yield. However, based on the experimental experience performing both methodologies, it can be stated that TPA from basic hydrolysis is obtained more easily and safely compared to TPA from acid hydrolysis. The products were confirmed through IR spectra, which highlighted characteristic bands showing the changes in functional groups from PET to TPA.

Obtaining TPA through chemical recycling is relevant from an environmental perspective, as it presents a distinct option for the treatment of plastic waste in El Salvador. Furthermore, this work can establish a precedent for potential chemical recycling feasibility studies in the country, offering an alternative to the mechanical recycling currently in practice.

Declarations

Conflict of interest

The authors declare no interest conflict. They have no known competing financial interests or personal relationships that could have appeared to influence the article reported in this article.

Author contribution

De la Cruz, Boris: Literature search, methodological design of basic hydrolysis, experimental process of basic hydrolysis, analysis of spectroscopic and yield data, and manuscript writing.

Domínguez, Ricardo: Literature search, methodological design of acid hydrolysis, experimental process of acid hydrolysis, analysis of spectroscopic and yield data, and manuscript writing.

Moreno, Juan: Literature search, methodological design of statistical analysis, application of statistics for data analysis, and manuscript writing.

Availability of data and materials

All data generated or analyzed during this study are included in this published article and its supplementary information files.

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Abbreviations

ATR	Attenuated Total Reflection
EG	Ethylene Glycol
FTIR	Fourier Transform Infrared Spectroscopy
HCl	Hydrochloric acid
H_3PO_4	Phosphoric acid
HNO_3	Nitric acid
H_2SO_4	Sulfuric acid
KOH	Hidróxido de potasio
NaOH	Potassium Hydroxide
NYL	Nylon
PE	Polyethylene
PET	Poly(ethylene terephthalate)
PP	Polypropylene
PS	Polystyrene
PVC	Polyvinyl Chloride
TPA	Terephthalic acid

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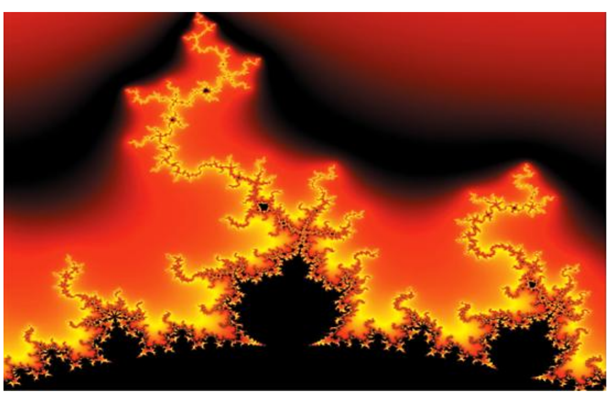

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Develop give the meaning of the variables in linear writing and important is the comparison of the used criteria.

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Clearly explain the results and possibilities of improvement.

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Specify the contribution of each researcher in each of the points developed in this research.

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Indicate the availability of the data obtained in this research.

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Indicate if the research received some financing.

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Abbreviations

List abbreviations in alphabetical order.

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ANN	Artificial Neural Network

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