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Effect of pulsed vacuum and laser microperforations on the potential acceleration of chicken meat marination

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Abstract

Chicken meat marination has become a fundamental process in the poultry industry since it provides a product with better sensory attributes. The objective of this study was to examine the effect of CO_2 laser microperforation coupled with vacuum impregnation treatment on the marination processing time of chicken breasts and to mathematically analyze the marinade diffusion process using Fick's second law and an anomalous diffusion model. Cylindrical cuts of unmarinated chicken meat were CO_2-laser microperforated with pores of 228 μm and marinated under vacuum pressure (15 kPa) with NaCl (3% wt/wt) and sodium tripolyphosphate (1% wt/wt) during 60 hr at 6 ° C. The chicken:brine ratio was 1:11 wt/wt. Mass gain, moisture content, and salt concentration were determined over time; furthermore, effective diffusion coefficient (D_{eff}) was obtained using Fick's second law and anomalous diffusion models. Marinade diffusion into chicken cuts was favored by microperforation combined with vacuum pulses, reduction processing time in 34%. The D_{eff} ranged between 1.46×10^{-10} and $2.08 \times 10^{-10} m^2/s$ for Fick's second law and between 2.27×10^{-10} and $4.23 \times 10^{-10} m^2/s^{\alpha}$ for anomalous diffusion model, with α–values close to 1. In conclusion, the results showed no significant difference between the models, which was attributed to the homogeneity of the chicken tissue.

Practical Applications

Chicken meat marination has become a fundamental process in the poultry industry since it provides a product with better sensory attributes and shelf life than unmarinated chicken meat. Nevertheless, marination is a process that requires a long processing time to obtain a specified salt content in the meat. In this sense, laser microperforation of the meat is a treatment that when coupled with vacuum impregnation, can accelerate the marinating process of poultry meat. When simultaneously applying both technologies, the processing time was reduced by 6 hr compared with the control (almost 34%), which will allow a significant increase in plant productivity.

1 | INTRODUCTION

Currently, chicken (*Gallus gallus domesticus*) is one of the most consumed animal proteins worldwide (OECD, 2019). This is because chicken meat is a low-cost protein; in addition, chicken meat has been considered a healthy alternative with respect to red meat due to its low fat content and its versatility (Katiyo, de Kock, Coorey, & Buys, 2020). Chicken marination has become a fundamental process in the poultry industry due to the rapid increase in demand by consumers, marketers, and catering chains, as it delivers a product with better flavor, texture, and juiciness and extends the shelf life compared to conventional unmarinated chicken (Yusop, O'Sullivan, Kerry, & Kerry, 2012). Several studies have focused on how the processing time is affected by the type of additives used as marinades (NaCl, phosphates, acids, proteins, etc.) (Kaewthong & Wattanachant, 2018; Kaewthong & Wattanachant, 2019; Lyon, Smith, & Savage, 2005; Sengun et al., 2021; U-chupaj, 2015; Yusop, O'Sullivan, & Kerry, 2011), the marination techniques (immersion, injection, and tumbling) (Alvarado & McKee, 2007; Rodrigues et al., 2016; Saha, Lee, Meullenet, & Owens, 2008), the process conditions and the equipment used in margination (Barbanti & Pasquini, 2005; Gamage, Mutucumarana, & Andrew, 2017; Yusop, O'Sullivan, Kerry, & Kerry, 2010).

Considering the slow diffusion rate in the marinating process mainly due to the lower temperatures at which the process is performed to avoid microbial spoilage, different emerging technologies have been studied with the aim of accelerating mass transfer and promoting an improvement in quality (Simpson et al., 2018). Some of these technologies are the use of ultrasound, electric fields, and high hydrostatic pressures (Deumier, Trystram, Collignam, Guédider, & Bohuon, 2003; Li et al., 2020; Núñez et al., 2020; Simpson et al., 2018). One of the newest technologies is the use of CO_2 laser microperforation. Formation of micropores in the sample favors the mass transfer process. This has been reported in salmon tissue brining (Olivares et al., 2021), pork marination (Figueroa, Ramírez, Nuñez, Jaques, & Simpson, 2020), tomato peeling with lye (Silva et al., 2020), fruit peeling (Panchev, Kirtchev, & Dimitrov, 2011), the freeze drying of blueberries (Fujimaru, Ling, & Morrissey, 2012; Munzenmayer et al., 2020), and dough browning (Blutinger et al., 2019). Independent of the product, microperforation had reported significant time reductions varying between 48 and 67% with respect to non-microperforated samples, depending on the matrix and mass transfer performed. In the case of tomato peeling, a significant reduction in the lye concentration was reported when micropores were applied.

The mass transfer process is usually represented and modeled by Fick's second law using the solutions proposed by Crank (1975). However, some authors, such as Simpson, Jaques, Núñez, Ramírez, and Almonacid (2013), have proposed that diffusion in food materials generally obeys non-Fickian diffusion (anomalous model). The anomalous diffusion model, which is based on the fractional calculus tool, has been applied to describe the diffusion process in different food materials with a high goodness of fit compared with Fick's second law model. Additionally, the model trough time exponent α can estimate if the diffusion process is subdiffusive ($\alpha < 1$), superdiffusive ($\alpha > 1$) or Fickean ($\alpha = 1$) (Núñez et al., 2020; Simpson et al., 2013; Simpson et al., 2015; Simpson, Ramírez, Núñez, Jaques, & Almonacid, 2017).

On the other hand, over the last 20 years, several authors have studied the application of pulsed vacuum impregnation in the brining or salting processes: Chiralt et al. (2001) reported on the salting of Manchego cheese, the marination of turkey meat was carried out by Deumier, Trystram, et al. (2003), the use of vacuum for marination of salmon was performed by Larrazábal-Fuentes, Escriche-Roberto, and Camacho-Vidal (2009), the salting of Russian sturgeon (*Acipenser gueldenstaedtii*) by Shen et al. (2020), and NaCl was replaced with KCl during the salting of beef cuts by Bampi, Domschke, Schmidt, and Laurindo (2016). In these studies, the results showed a significant reduction in the processing time and an improvement in the quality of the food studied. Pulsed vacuum treatment applies alternating short periods of vacuum and atmospheric pressure to a product immersed in a solution, causing an acceleration in the impregnation of salt into the product and greater homogeneity in its distribution and reduces water loss (Deumier, Bohuon, Trystram, Saber, & Collignam, 2003). The porosity of the solid matrix is the most relevant structural property in terms of the effectiveness of vacuum impregnation (Chiralt et al., 2001). However, the complex microstructure of meat products and their mechanical properties lead to important changes in the physical properties of the product under different treatments (Deumier, Trystram, et al., 2003).

Therefore, we have two technologies (Co_2 laser microperforation and vacuum impregnation) that individually have shown a potential impact in accelerating the mass transfer process. Therefore, our hypothesis is that coupling both techniques can significantly reduce the processing time during the cold marination of chicken breast compared with the control and with each technique performed individually.

Based on this hypothesis, the objective of this research was to study the effect of Co_2 laser microperforation coupled with vacuum impregnation treatment on the marination processing time of chicken breasts and to mathematically analyze the marinade diffusion process using Fick's second law and an anomalous diffusion model.

2 | MATERIALS AND METHODS

2.1 | Materials

Fresh, deboned, skinless, and unmarinated chicken breasts were purchased from a local market in the city of Valparaíso, Chile and were stored refrigerated (2 ± 0.5 °C) until use. Sodium chloride (98% purity NaCl) and food-grade sodium tripolyphosphate (STPP), purchased from G. A. Ventas (Viña del Mar, Chile), were used to prepare the marinade solution.

2.2 | Marination and sampling

Cylindrical meat chicken sections of 0.0025 ± 0.001 m in diameter (2R) and 0.025±0.001 m in height (L) were formed perpendicular to the fibers, promoting bidirectional diffusion. The average weight of the samples was 0.0155 ± 0.0005kg.

The amount of brine required was calculated based on an 11:1 (wt/wt) brine: chicken ratio to minimize changes in the concentration of the solution during the process (Simpson et al., 2018); 45 g NaCl and 15 g STPP were dissolved in in 1.5 L water to obtain 3 and 1% NaCl and STPP solutions, respectively (values used in commercial operations). The brine was refrigerated (2 °C) until use (Kaewthong &

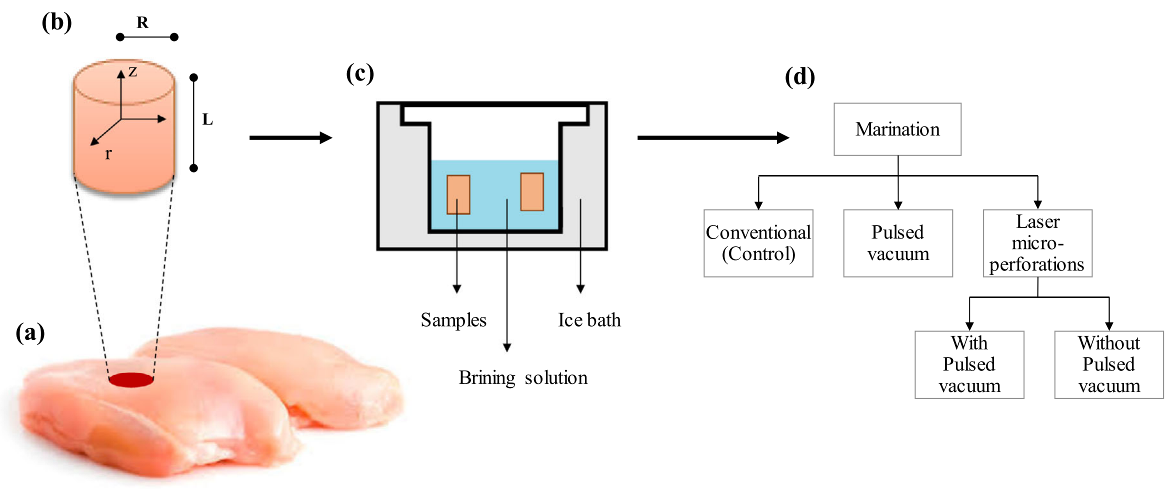

FIGURE 1 Sampling and experimental design: (a) section of the chicken breast used to obtain cylindrical samples; (b) sample geometry: diameter (2R), 0.025 m; height (L = 2R), 0.025 m; (c) experimental setup of the marination; and (d) proposed experimental design

Wattanachant, 2018; Komoltri & Pakdeechanuan, 2012; Muñoz, Restrepo, & López, 2014; Yusop et al., 2011).

To evaluate the effect of microperforation and vacuum impregnation on the processing time, four treatments were carried out: (a) standard marination as the control process (no vacuum or microperforation treatment), (b) marination with pulsed vacuum impregnation, (c3) CO_2 laser microperforation treatment followed by standard marination, and (d) CO_2 laser microperforation treatment followed by marination coupled with pulsed vacuum impregnation (Figure 1). All treatments were performed at 6 ± 2 ° C for 60 hr. All treatments were carried out in duplicate.

2.3 | Microperforation and vacuum impregnation setup

Microperforations were made to the chicken cylinders in the longitudinal direction in a 5 × 5 square arrangement (25 perforations in total) with a 5 - mm separation between perforations. The laser was operated using 35% of the total 100-W power, and 300 pulses with a 2 - μs duration and a delay of 1μs between pulses were applied using a SYNRAD TI100 model CO_2 laser with a 10.6 - μm wavelength and a 125 - nm flat field lens. This configuration was established to avoid burning the borders of the samples. The pore sizes were 228 ± 15.75μm. The pore size generated was measured by optical microscopy (Hund Wetzlar brand 112 microscope, Germany) using uEye Cockpit software from Imaging Development Systems 113 (IDS) and Image-Pro Plus, which allowed a microscopic photo to be captured for observation.

Vacuum impregnation was performed according to the methodology proposed by Deumier, Bohuon, et al. (2003) with slight modifications. For marination, the cylindrical samples were placed inside a glass desiccator that contained two thermocouples, and the container was hermetically sealed and connected to a vacuum pump using a plastic hose (Dosivac, model DVP-1, Buenos Aires, Argentina). A vacuum pressure of 15 kPa was applied in a three-stage cycle: the time required to reach the vacuum pressure (t_0 = 90s), the time in which the vacuum was maintained (t_1 = 150s) and the time required to reestablish the atmospheric pressure conditions (t_2 = 4s). Vacuum pulses were implemented for 0, 5, 10, 20, 30, and 45 hr of marination.

2.4 | Sampling and analytical determinations

Marination sampling was performed at 0, 5, 10, 20, 30, and 60 hr, choosing four random samples at each time to determine the moisture and salt contents. Additionally, two properly labeled samples were used to measure the weight change during the processing time, and those samples were excluded from the moisture and salt content determination. The measurements were carried out in duplicate.

The weight of the samples was determined on an analytical balance, and the variation in the mass (ΔM_t^{0}) was calculated using Equation (1):

$$\Delta M_t^0 = \frac{M_t^0 - M_0^0}{M_0^0} \quad (1)$$

where M_0^0 and M_t^0 correspond to sample weights (g) at the beginning (t = 0) and at each sampling time (t), respectively.

The moisture content was determined by drying 5 g of the sample in an oven at 105 ° C for 24 hr and recording the final constant weight (AOAC, 2000). The variation in moisture $\left(\Delta M_t^W\right)$ was determined using Equation (2):

$$\Delta M_t^W = \frac{M_t^0 x_t^W - M_0^0 x_0^W}{M_0^0} \quad (2)$$

where x_0^w and x_t^w are the water mass fractions in a sample at the beginning (t = 0) and at each sampling time (t), respectively.

The salt content was determined using the Dicromat II salt analyzer (PCL Control Instruments, Leicester, UK): a 20-g sample of chicken was taken, mixed, and homogenized with 200 ml of distilled water. The measurement was carried out by introducing the electrode into the formed solution and reading the value reported by the device. The variation in the salt (ΔM_t^S) was calculated using Equation (3):

$$\Delta M_t^S = \frac{M_t^0 x_t^S - M_0^0 x_0^S}{M_0^0} \quad (3)$$

where x_0^s and x_t^s are the salt mass fractions in a sample at the beginning (t = 0) and at each sampling time (t), respectively.

2.5 | Mathematical models

2.5.1 | Fick's second law of diffusion

The chicken meat samples corresponded to a finite cylinder geometry with bidirectional diffusion, where the salt concentration profile towards the inside of the cylinder can be determined using the solution given by Crank (1975). The following assumptions were considered: the diffusion process was isothermal (6 °C), soluble solids were initially uniformly distributed, neither contraction nor shrinkage of the sample was considered, there were simultaneous countercurrent flows (water flowed out and salt entered the chicken), and the effective diffusion coefficient (D_{eff}) was constant.

The analytical expression for diffusion in a finite cylinder is given by Equation (4): (Crank, 1975).

$$\frac{\partial C}{\partial t} = D_{eff}\left[\frac{1}{r}\frac{\partial}{\partial r}\left(r\frac{\partial C}{\partial r}\right) + \frac{\partial^2 C}{\partial z^2}\right] \quad (4)$$

The solution of Equation (4) depends on the initial and boundary conditions of the system:

$$C(r,z,t=0) = C_0 \quad (5)$$

$$C(r=R,z,t) = C_{e,}\ t>0 \quad (6)$$

$$C(r,z=\pm L/2,t) = C_{e,}\ t>0 \quad (7)$$

By integrating Equation (4), we obtained the solution of the average dimensionless concentration for a finite cylinder in the following form:

$$C(t) = C_e + (C_0 - C_e)\left\{\frac{8}{\pi^2}\left[\sum_{n=0}^{\infty}\frac{1}{(2n+1)^2}\exp\left(-D_{eff}\left(\frac{(2n+1)\pi}{L}\right)t^2\right)\right] * \left[\sum_{m=1}^{\infty}\frac{4}{\varepsilon_m^2}\exp\left(\frac{-\varepsilon_m^2 D_{eff} t}{R^2}\right)\right]\right\} \quad (8)$$

where $C^*(t)$ is the dimensionless concentration of the salt in the chicken at time t, $C(t)$ is the concentration of salt in the chicken at time t, Ce is the concentration of salt at equilibrium, C_0 is the initial concentration of salt (t = 0), D_{eff} is the effective diffusion coefficient (m^2/s), L and R are the half thickness and the radius of the sample, respectively (m), t (s) is the time, and ε_m is the mth zero of the zero-order Bessel function.

2.5.2 | Anomalous diffusion model

For diffusion in porous media, previous studies (Ramírez, Astorga, Núñez, Jaques, & Simpson, 2017; Simpson et al., 2013; Simpson et al., 2015; Simpson et al., 2017) have shown that it is possible to use a fractional order for the temporal derivative (α) and second order for the spatial derivative to represent the evolution of mass transport, avoiding underestimation of the complexity of a problem. Equation (9) corresponds to the finite cylinder geometry in fractional terms, and Equation (10) corresponds to the solution of Equation (9):

$$\frac{\partial^\alpha C}{\partial t^\alpha} = D^*{}_{eff}\left[\frac{1}{r}\frac{\partial}{\partial r}\left(r\frac{\partial C}{\partial r}\right) + \frac{\partial^2 C}{\partial z^2}\right] \quad (9)$$

$$C(t) = C_e + (C_0 - C_e)\left\{\frac{8}{\pi^2}\left[\sum_{n=0}^{\infty}\frac{1}{(2n+1)^2}E_\alpha\left(-D^*{}_{eff}\left(\frac{(2n+1)\pi}{L}\right)^2 t^\alpha\right)\right] * \left[\sum_{m=1}^{\infty}\frac{4}{\varepsilon_m^2}E_\alpha\left(\frac{-\varepsilon_m^2 D^*{}_{eff} t^\alpha}{R^2}\right)\right]\right\} \quad (10)$$

Thus, the solution for diffusion with fractional calculus was analogous to Equation (8), with the same initial and boundary conditions, where E_α corresponds to the Mittag–Leffler function, which is commonly used in solving equations with derivatives of fractional order, the function converges to the exponential function of Equation (8) when α = 1, and D^*_{eff} is the fractional diffusion coefficient (m/s^α). The α value indicates the transport mechanism that dominates the mass transfer process. Thus, if $0 < \alpha < 1$, the diffusion mechanisms can be assumed to be subdiffusive. In contrast, if $\alpha > 1$, the mechanisms can be considered superdiffusive. If α converges to unity, the anomalous diffusion model converges to Fick's second law.

Experimental data fitting was performed using MATLAB R2019b software version 9.6 (the MathWorks Inc., Natick, MA), with programming for n terms of the sum (n = 150). The developed code allowed us to obtain the values of the effective diffusion coefficient (D_{eff}), the sum of squared errors (SSE), the coefficient of determination (R^2) and the parameter alpha (α).

2.6 | Statistical analysis

To analyze the data, a design of free randomized experiments was carried out, taking as an independent variable the type of process with four levels: (a) "conventional," (b) "vacuum," (c) "laser," and (d) "Laser + vacuum." The dependent variables evaluated were Fick's diffusion coefficient, anomalous diffusion coefficient, alpha value, and marinating time.

To establish whether the type of process affected the dependent variables, an analysis of variance was performed with a confidence level of 95%, and to establish whether there were differences between the treatments, a multiple-range LSD-Fisher test was performed with a 95% confidence level using Statgraphic Centurion XV software (v15.1.02, Statgraphic Technologies Inc.).

To verify the assumptions of normality and independence of the data, Kolmogorov–Smirnov lack of fit and chi-square tests were performed, respectively, with 95% confidence. The normality test indicates whether the data fit the mathematical model of the design (Equation (11)). The independence test shows that the experiment was performed randomly and that each experiment and experimental unit did not have a linear dependency. Finally, to establish if the number of treatments was adequate for the experiment, power tests were performed for each dependent variable.

$$Y_{tj} = \mu + \tau_t + \varepsilon_{tj} \tag{11}$$

where Y_{ij} is the ith measurement in treatment j, μ is the scale parameter common to all treatments, referred to as the global mean, τ_i is a parameter that measures the effect of each treatment i on the response variable, and ε_{ij} is the error attributable to measurement Y_{ij}.

3 | RESULTS AND DISCUSSION

3.1 | Statistical analysis

The analysis of normality, power, and independence of the data are presented in Table 1. From the normality analysis, it can be seen that the data correspond to a normal distribution, since the p-value of the Kolmogórov–Smirnov test was greater than .05 for all cases, indicating that all the data of the response variables fit the model specified in Equation (11). Finally, once the power analyses were performed, it was evident that the number of experiments was valid for the Fick's diffusion coefficient, the α– value and the marinating time, since in general values close to and greater than 80% were obtained, indicating the reliability of the data (Howell, 2014; Montgomery, 2017). In the case of the anomalous diffusion coefficient, although high power was not obtained, it is adequate to know that the data come from a normal distribution. Regarding independence, a p-value greater than .05 was obtained, confirming that the experiments were carried out randomly and that each sample or experimental unit was independent of each other; that is, they did not have a linear dependence.

3.2 | Effect of the treatments on the total mass weight, water, and salt changes

In general, the marination absorption rate was higher in the first 5 hr of immersion, decreasing gradually until becoming stable after 30 hr of marination, indicating that the brine compounds migrated into the product and that, in turn, the water retention capacity increased. These results are in agreement with those obtained in previous studies carried out by Totosaus (2007), Cori, Michelangeli, de Basilio, Figueroa, and Rivas (2014), Cando, Borderías, and Moreno (2017), and Thangavelu, Kerry, Tiwari, and McDonnell (2019) since salt increased the ionic strength, facilitating the solubilization and dispersion of myofibrillar proteins, which made it possible for these proteins to bind water and form a well-organized and stabilized protein network, improving the binding properties of poultry meat.

Figure 2 shows the variation in the water content of the chicken meat cylinders immersed at 6 ° C in a 3% NaCl and 1% STPP solution. For all the treatments, an increase in the water content as marination elapsed was observed, with the treatments that received laser microperforation having the highest water content values throughout the

TABLE 1 Normality (Kolmogorov–Smirnov), power and independence (chi-square) tests results applied for every response variable

Treatment	Fick's diffusion coefficient (D_{eff})	Anomalous diffusion coefficient (D_{eff}*)	α-value	Marinating time for 0.2% salt (hr)
Conventional	1.448a	7.029a	0.826a	16.60a
	1.473a	1.429a	1.000a	18.40a
Vacuum	1.701a	2.517a	0.957a	14.95a
	1.462a	2.031a	0.965a	15.05a
Laser	1.594a	4.140a	0.899a	15.40a
	1.405a	1.959a	0.964a	15.60a
Laser + vacuum	2.389a	2.390a	1.000a	10.00b
	1.778a	2.251a	0.975a	13.00b
Normality test p-value*	.795	.289	.442	.642
Power test (%)	79	30	98	95
Independence test	0.998			

Note: Different letters indicate that there were significant differences ($p < .05$) between treatments.
*Estimated parameter significance ($p < .05$).

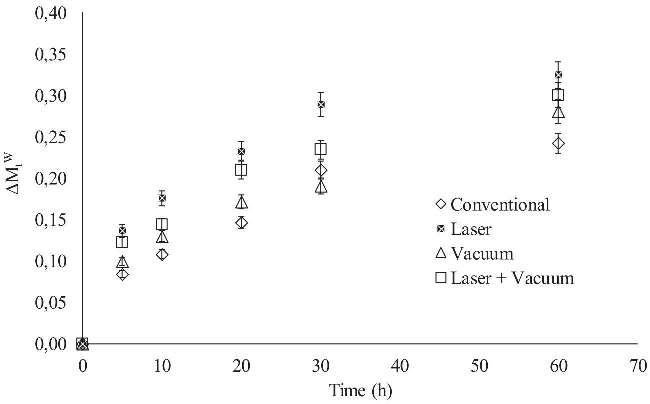

FIGURE 2 Variation in the moisture content over time for all treatments

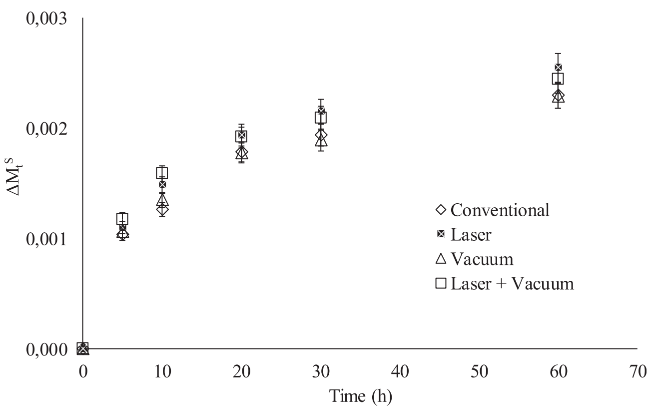

FIGURE 3 Variation in the salt content over time for all treatments

whole process. The results show that marination treatment with vacuum and microperforation significantly increased the water weight change in the samples. After 60 hr, the water retention capacity was greater with the microperforation pretreatment and the vacuum impregnation treatment alone, but coupling both treatments resulted in a lower water retention capacity, which can be explained by the increased porosity caused by the laser, which favors the migration of solution within the chicken samples; this effect has also been reported in salmon (Olivares et al., 2021) and pork meat (Figueroa et al., 2020). Figure 3 shows the salt gain with increasing marination time for all treatments. The results showed no significant differences among the different curves obtained for each treatment (p > .05). Furthermore, it was possible to observe that the mass gained by salt uptake was relatively low, with an increase close to 0.3% (Angel, Weinberg, & Jaffe, 1989; Desmond, 2007), indicating that the mass gain was mainly caused by water retention due to phosphate present in the marinade solution. The use of STPP increases the pH of myofibrillar proteins away from their isoelectric point, which reduces the interaction of proteins and, thus, increases the capacity for water retention (Warner, 2017).

Figure 4 shows the total weight of the chicken meat over the marination time for all the treatments. The results showed an increase

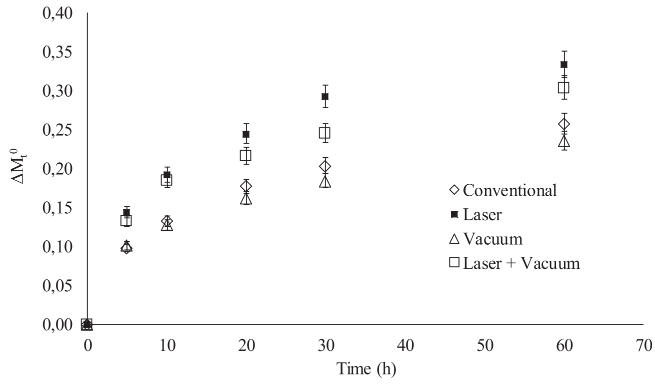

FIGURE 4 Variation in the mass over time for all treatments

in the total weight change as the marination time increased for all treatments. However, in the microperforated samples and microperforated and vacuum-treated samples, a significant increase in ΔM_0^t with respect to conventional or vacuum-assisted processes was observed, since the increase in porosity caused by the laser favors the migration and retention of the solution inside the chicken meat. According to Fujimaru et al. (2012), laser perforation contributed significantly to the weight gain of infused blueberries.

Several authors have reported the effects of incorporating vacuum treatments in the salting and marinating processes of different kinds of foods, obtaining favorable yields. Nevertheless, they use higher temperatures, salt solution concentrations, and contact areas than those used in this study, among other variables, corroborating the marked influence of the process conditions on the effect of the applying vacuum treatment (Bampi, Schmidt, & Laurindo, 2019; Chiralt et al., 2001; Corzo & Bracho, 2007; Jin et al., 2014; Mújica-Paz, Argüelles-Piña, Pérez-Velázquez, Valdez-Fragoso, & Welti-Chanes, 2006; Serio et al., 2017; Tomac, Rodríguez, Perez, Garcia, & Yeannes, 2020; Wang, Xu, Kanga, Shen, & Zhanga, 2016). Deumier, Bohuon, et al. (2003) reported that the number of pulses affected the water and salt mass gain differently. The salt mass gain was constant and had an upward trend as the number of pulses increased, while the water mass gain increased significantly during the first few pulses and plateaued as the number of pulses increased (Deumier, Trystram, et al., 2003).

With respect to the treatments used, the results show that the application of vacuum pulses had no greater effect on the mass gain compared to that of conventional marination; this could be related to the high porosity of chicken (30–60%) (ASHRAE, 2006; Boukouvalas, Krokida, Maroulis, & Marinos-Kouris, 2006), which causes the activation of nondiffusion solution infiltration mechanisms in meat spaces occupied by occluded and/or dissolved gases (capillary forces) before the application of the vacuum, which occurs similarly in plant and dairy products under atmospheric pressure conditions (Deumier, Trystram, et al., 2003; Hofmeister, Souza, & Laurindo, 2005). In other words, the moment the sample was immersed in the brine, hydrodynamic forces began to act, promoting the solution to enter the interior of the sample according to the porosity of the material, and this occurred earlier and faster than diffusional transport and could explain why there were no significant differences in the mass gain when applying a vacuum with respect to that of conventional marination. In this sense, saturated brine solutions would be recommended to accelerate the process, that is, modifying the concentration of salt used in conventional commercial operations while still evaluating the final functional properties to enhance the effect of the evaluated technologies.

The laser microperforations presented positive results in the variation of the mass and moisture content of the marinated chicken samples, and this was mainly attributed to two effects related to the structural alteration of the original sample: first, the contact area between the sample and the solution increased, and second, the distance the solute had to travel to access the interior of the sample was significantly reduced, after which the solute had to diffuse only the average distance that separated a pore from an adjacent pore. Regarding the first postulate, a simple calculation showed that the increase in the contact area was minimal (less than 0.1%). Thus, the reduction in the diffusion distance was very high, and depending on the number of microperforations made per square meter, this distance could be reduced between 5- and 10-fold compared to that of the original sample. In summary, the second postulate could explain, with relatively good precision, the effect of microperforations on the diffusion of the brine in the samples, achieving greater homogeneity than that achieved without microperforations (Figueroa et al., 2020).

3.3 | Effect of the treatments on the salt content in chicken meat cylinders

Regarding the concentration of salt attained in chicken meat cylinders (Figure 5), no significant differences were observed between treatments throughout the marinating process ($p > .05$). These results can be attributed to the low salt concentrations used, both in the chicken (from 0.07 to 0.25%) and in the marination solutions (3%, value based on commercial operations). Different authors have studied the influence of marinade concentration on the salt and water diffusion coefficients in meat using multiple regression models and have demonstrated that the rates of salt and water diffusion are positively correlated with an increase in the salt concentration of the brine (Chiralt et al., 2001; Gómez et al., 2015; Volpato, Michielin, Ferreira, & Petrus, 2007). This argument was consistent with the results of similar studies, such as those carried out by Simpson et al. (2018), where salting Atlantic salmon was evaluated using solution concentrations of 6 and 24% salt. In their study, they found relatively steep slopes by directly relating the concentration of salt in the salmon samples to the salting time and achieving high final concentrations of salt in the salmon since a directly proportional relationship was observed between the equilibrium concentration and the concentration of the solution; that is, a higher brine concentration resulted in a higher salt concentration in the samples. Moreno et al. (2020) studied marination applied to chicken breasts at concentrations of 3, 5, and 7% (salt and polyphosphate), and the results showed that the 7% concentration improved the mass transfer of ions in the treated samples.

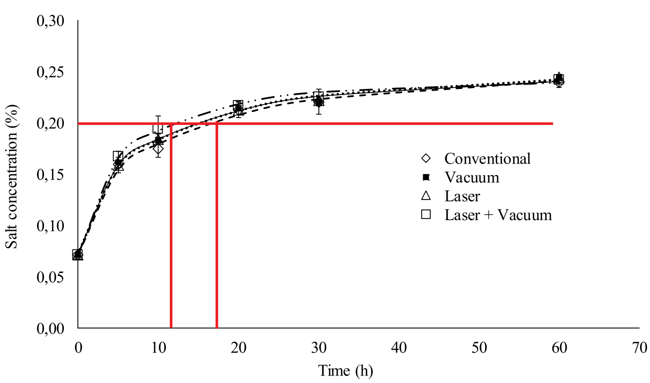

FIGURE 5 Comparison of the time and final salt concentrations for all treatments. *The red line indicates the marinating time required to achieve the standard salt concentration of 0.20% (commercial marinated chicken)

3.4 | Reduction in the marination processing time

The concentration of salt in food is commonly reported using the sodium content of the food, which, according to the World Health Organization, can be related using the formula in Equation (12) (WHO, 2012):

$$\mathrm{g Na} * 2.5 = \mathrm{g Nacl} \quad (12)$$

However, unmarinated chicken has an average of 41 mg of Na per 100 g of product, and once marinated, the Na concentration can reach 90 mg of Na per 100 g of product, that is, a salt concentration of approximately 0.225% (Probst, 2009). To evaluate the reduction in the marination time in this study, a standard salt concentration of 0.20% in the marinated chicken samples was considered. Figure 5 shows the marination time required to achieve the previously established standard salt concentration in the marinated chicken.

The processing time needed to reach the reference concentration using each treatment is presented in Table 2; likewise, the numerical values of the reduction times are reported concerning conventional or control marination, showing that all the treatments were able to reduce marination time at different rates. The reduction in marination

TABLE 2 Reduction in marination time for all treatments

Treatment	Marinating time for 0.2% salt (hr)	Reduction time (hr)
Conventional	17.5 ± 1.27^A	–
Vacuum	15 ± 0.07^A	2.5
Laser	15.5 ± 0.14^A	2
Laser + vacuum	11.5 ± 2.12^B	6

Note: Different capital letters express significant differences between the values ($p < .05$).

time was significantly more effective when coupling conventional marination with laser microperforations and vacuum pulses than when implementing conventional marination alone ($p < .05$).

The reference salt concentration for conventional marination was attained in 17.5 hr, while for laser microperforations and pulsed vacuum, the reference concentration was attained at 15.5 and 15 hr, respectively. It should be noted that vacuum pulses did not have a significant effect on the mass increase or moisture or salt content (Figures 2–4), but vacuum pulses did accelerate the marination, indicating that there was an effect on the migration of salt into the interior of the meat, causing it to penetrate the tissue faster.

By coupling the laser microperforations with vacuum pulses, a reduction in the processing time of approximately 34% (approximately 6 hr) was obtained. This treatment was the most effective in accelerating chicken meat marination compared to the conventional marination process. This time reduction revealed a synergy between the reduction in the diffusion distances, which allowed for the solute to enter and become distributed inside the tissue, and the subsequent extraction of the air present in the pores that were in contact with the marinade, which was replaced by the marination solution during the application of the pulsed vacuum, which improved the rate of diffusion.

This result indicates that vacuum treatment coupled with CO_2 laser microperforations causes a reduction in time greater than the sum of the reduction times of the two treatments used separately, giving rise to a synergistic effect between the methods.

Munzenmayer et al. (2020) reported reductions in the primary drying time of freeze-dried blueberries when skin microperforations were made, which was in addition to significant quality improvements; the same was observed with tomato peeling performed by Silva et al. (2020), and skin microperforations not only accelerated of the process but also decreased the energy requirements. Figueroa et al. (2020) found that the application of pulsed vacuum impregnation and CO_2 laser microperforations accelerated the pork marination process with a maximum processing time reduction of 47%. Olivares et al. (2021) also showed the positive effect of micropores on the mass transfer process, attaining a 30% reduction in the processing time. Mújica-Paz et al. (2006) found a 50% reduction jalapeño pickling times when using vacuum pulses, and these authors also observed that vacuum pulses enhanced the solute concentration and weight gain of the product. In addition, a notable reduction in salting times and an increase in processing yields were reported by Chiralt et al. (2001) in meat and fish processing with vacuum impregnation pretreatments. The results obtained by these authors agree with the results found in the present study.

3.5 | Mathematical analysis based on Fick's second law and the anomalous diffusion model

3.5.1 | Fick's second law

The results obtained for D_{eff} determined by Fick's second law adjustments are presented in Table 3. The effective salt diffusion coefficient (D_{eff}) values for chicken meat marination were between 1.46×10^{-10} and $2.08 \times 10^{-10} m^2/s$ (Table 3), and the experimental values were similar to those reported in the literature for diffusion coefficients of salt in meat products, as in the results reported by Volpato et al. (2007) with diffusivities of sodium chloride in chicken breasts in the range of 8.99×10^{-10} and $9.55 \times 10^{-10} m^2/s$ (with brining solutions consisting of 2% NaCl, 3% STPP, and 4% dextrose). Chiralt et al. (2001) found diffusivity coefficient values between 1.61×10^{-10} and $4.15 \times 10^{-10} m^2/s$ for beef and salmon, which were similar to those found by Gallart-Jornet et al. (2007), with values close to $9.90 \times 10^{-10} m^2/s$ for salting Atlantic salmon. The determination of diffusion coefficients in salted pork studied by Graiver, Pinotti, Califano, and Zaritzky (2006) was accompanied by diffusivity values on the order of $0.6 \times 10^{-10} m^2/s$ (using a salt concentration of 20 g/L), and Graiver, Pinotti, Califano, and Zaritzky (2009) reported a D_{eff} value of $8.5 \times 10^{-10} m^2/s$ for an experiment performed using brining solution with 140 g/L NaCl. There were no significant differences between the coefficients of the conventional treatment and the vacuum and laser coupled treatments ($p > .05$); however, when using the combined treatment of vacuum pulses with laser microperforations, the D_{eff} value ($2.083 \times 10^{-10} m^2/s$) tended to increase due to the structural modification of the tissue and the effect of capillary forces produced by the vacuum, improving the rate of diffusion of the solution into the material and, therefore, improving the mass transfer and the process performance.

3.5.2 | Anomalous diffusion model

Table 3 shows the results of fitting the experimental data to the anomalous diffusion model solved by fractional calculus. For almost all treatments used, R^2 increased and SSE decreased compared to those

TABLE 3 Effective diffusion coefficients for all treatments at 6 °C for 60 hr, with 3% NaCl in the brine according to Fick's second law and anomalous diffusion models

	Fick's second law model			Anomalous diffusion model			
Treatment	$D_{eff}(m^2/s)\ (\times 10^{10})$	SSE	R^2	$D_{eff}\,(m^2/s^{\alpha})\left(\times 10^{10}\right)$	α	SSE	R^2
Conventional	1.460 ± 0.018^A	0.0002	.991	4.229 ± 3.960^B	0.913 ± 0.123^C	0.0001	.993
Vacuum	1.582 ± 0.169^A	0.0001	.995	2.274 ± 0.343^B	0.961 ± 0.006^C	0.0001	.995
Laser	1.499 ± 0.133^A	0.0003	.998	3.049 ± 1.542^B	0.932 ± 0.046^C	0.0002	.999
Laser + vacuum	2.083 ± 0.433^A	0.0007	.997	2.321 ± 0.098^B	0.988 ± 0.017^C	0.0007	.984

Note: Different capital letters express significant differences between the values of the same column ($p < .05$).

determined using Fick's second law; however, this was an expected result, as the anomalous model incorporated an additional parameter into the equation. Nevertheless, as mentioned, Fick's second law was shown to be an appropriate model. Moreover, when using the anomalous model, the most relevant results were the values obtained for the alpha parameter. In all treatments, alpha values were not significantly

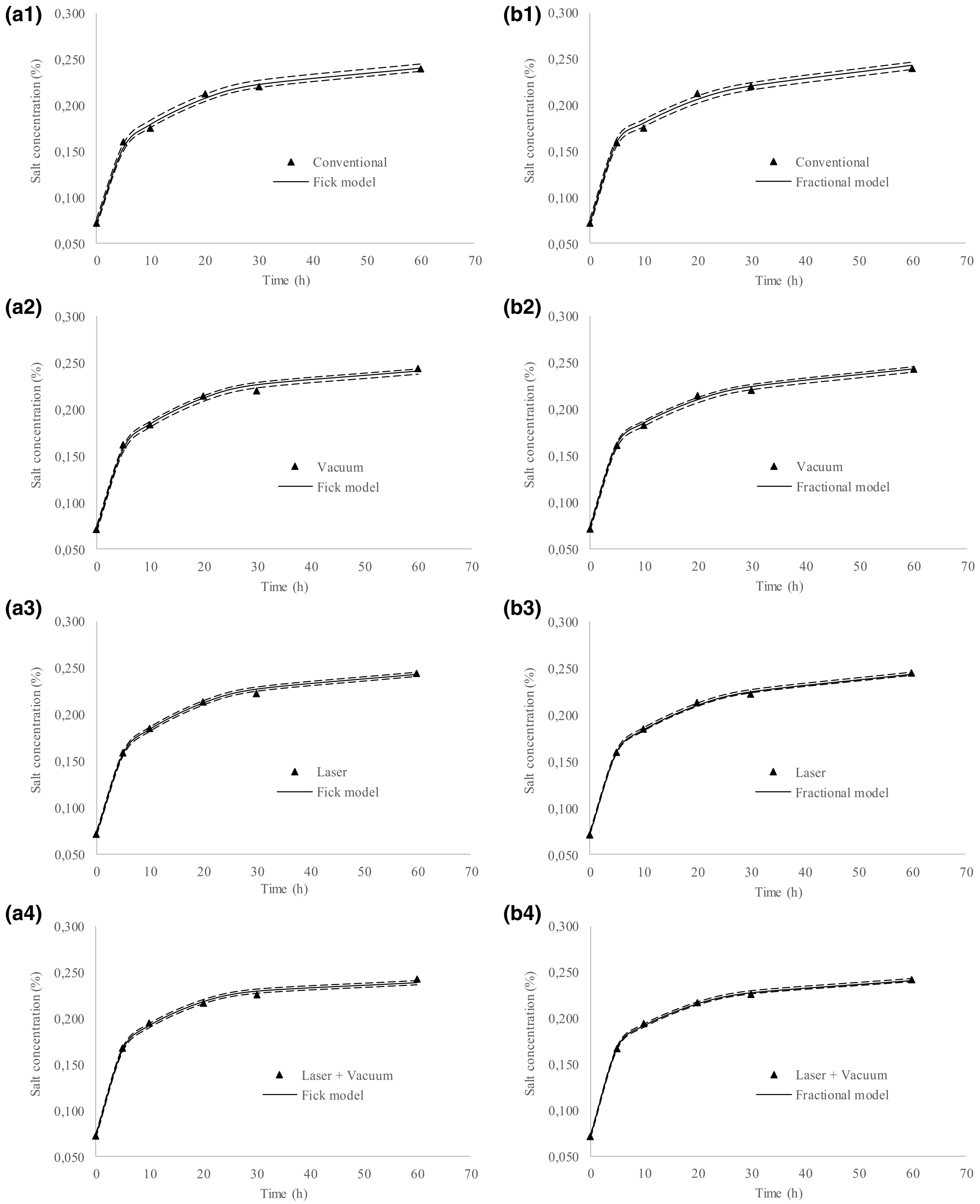

FIGURE 6 Adjustment of the experimental data for all the marination treatments to the Fick (1) and anomalous (2) models: (a) conventional, (b) vacuum, (c) laser, and (d) laser + vacuum

different from 1, indicating that the diffusion phenomenon tended to be Fickian since, when α = 1, the anomalous diffusion equation converges to Fick's second law. In the same way, none of the treatments caused changes at the microstructural level, so the α-value should not change, which is shown in Table 3.

The diffusion orders of magnitude obtained with the anomalous model were similar to those obtained with Fick's second law model, which makes sense given that the alpha values were close to 1. Furthermore, in the anomalous model, the values of diffusion did not vary statistically when coupling the pretreatments (p > .05). For salting salmon, Olivares et al. (2021) found that the fractional component of time (α < 1) showed subdiffusive behavior for microperforation treatments. On the other hand, for marinated pork, Figueroa et al. (2020) showed that in the case of microperforated pork coupled with VI, α – values were higher than 1, which suggests superdiffusive behavior for both treatments. Similar investigations have consistently shown that some food matrices are better modeled with the anomalous model than with other models (Ramírez et al., 2017; Simpson et al., 2015).

Figure 6 graphically depicts the adjustment of each treatment to both proposed models, where it is observed that the adjustment tends to improve with the anomalous diffusion model, but this improvement is not statistically significant. In addition, a steeper slope is observed when vacuum pulse and laser microperforation treatments are coupled with conventional marination (Figure 6).

However, the porosity of the solid matrix is a structural property of great relevance in food operations and research. Plant tissues, such as aubergines, apples, or even orange peels, have high porosity values (20–60%), and food matrices with compositions of greater complexity, such as the tissues of meat, fish, and even some cheeses, have much smaller porosities (1–10%), and few spaces inside are occupied by occluded and dissolved gases (less than 1%), making it difficult to manipulate and model these tissues in industrial processes. The problem is magnified when the heterogeneous structure of foods is added, which is frequently not included in mathematical models since they are usually based on ideal cases (Boukouvalas et al., 2006; Chiralt et al., 2001).

In the same way, the application of the new technologies used in this study promotes diffusion through a relatively homogeneous medium due to its effect on the porosity and structure of the food.

4 | CONCLUSIONS

Under the evaluated conditions, the results showed that all the treatments used separately accelerated the chicken marinating process, but a greater effect was achieved by coupling the laser microperforation and vacuum pulse treatments in the marinating process.

The application of laser microperforations coupled with vacuum pulses favored a significant reduction in chicken marinating time and showed a synergistic effect compared to that of conventional or control marinating.

In the future, evaluating the salt distribution throughout the sample at the end of the process is recommended to more precisely understand the phenomena that occur, in addition to evaluating the microstructural effects on the chicken muscle using SEM analysis.

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AUTHOR CONTRIBUTIONS

Natalia Ramírez: Formal analysis; methodology; writing-original draft. **Oscar Vega-Castro**: Formal analysis; investigation. **Ricardo Simpson**: Supervision; validation; writing-review and editing. **Cristian Ramirez**: Formal analysis; validation; writing-review and editing. **Helena Nuñez**: Conceptualization; investigation; methodology; validation; writing-review and editing.

DATA AVAILABILITY STATEMENT

Data available on request from the authors

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