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# Mass Transfer Modeling During Marination of Anchovy Fillets in Ternary and Multicomponent Solutions

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#### ABSTRACT

We assessed the applicability of three mathematical models (i.e., the Peleg, Zugarramurdi and Lupin and Weibull models) for water loss and solute gain of marinated *Engraulis anchoita*. The marinating stage was performed in a ternary solution of water/salt/acid and in a multicomponent solution of water/salt/acid/ sorbitol. Based on the statistical parameters ( $R^2 > 0.96$ ; 0.001 < RMSE < 0.032), the models were able to predict the salt and acid gains upon marinating in the water/salt/acid solution. Water loss and solute gain could be estimated by all of the proposed models ( $R^2 > 0.89$ ; 0.003 < RMSE < 0.080) for the water/salt/acid/ sorbitol marinating solution. The Peleg, Zugarramurdi, and Lupin and Weibull models can thus be used to represent the kinetics of mass transfer for water and solutes for marinated anchovies.

#### **KEYWORDS**

*Engraulis anchoita;* marinating; ternary and multicomponent solutions; mass transfer modeling

# Introduction

Marinated fish are semipreserved fish products that are ready-to-eat without heating (Fuentes et al., 2010). Acetic acid and salt are added to the fish to retard the action of bacteria and enzymes. This process results in a product with a characteristic flavor and an extended but limited shelf life (McLay, 1972). Marination is an osmotic dehydration (OD) process in which simultaneous counterdiffusion of water and solutes occurs. Three types of mass transfer counterflows occur: water flows from the product to the solution, solute transfers from the solution to the product, and solute outputs (minerals, volatile basis, organic acids, and vitamins) from the food to the solution. The latter is neglected in modeling because it is very small compared to the other two types of flow (Ochoa-Martinez and Ayala-Aponte, 2005; Corzo and Bracho, 2009). Mass transfer during OD under atmospheric pressure is generally modeled using a Crank model based on Fick's law (Rodger et al., 1984; Gerla and Rubiolo, 2003; Gou et al., 2003; Telis et al., 2003; Graiver et al., 2006; Corzo and Bracho, 2007; Casales et al, 2009). Other alternatives to this modeling process include empirical models such as the Peleg, Zugarramurdi and Lupin (Z and L), and Azuara models (Corzo and Bracho, 2005; 2006a, 2006b; Czerner and Yeannes, 2010). These models are based on mathematical representations of experimental data, resolving some of the limitations of the Fick's law-based model, for which the analytical solutions are available only for plane sheets, cylinders, and spheres, and the equilibrium state must be experimentally determined (Schmidt et al., 2009).

Diffusive models depend on the estimation or experimental determination of the equilibrium values, which require long immersion times that can lead to food tissue changes (Schmidt et al., 2009). Estimation of the equilibrium values can be performed using empirical models. Some of these models have been developed from polynomial adjustments. Other models use mass balances and the relationships between process variables (Ochoa-Martinez and Ayala-Aponte, 2005). Water loss and salt gain in fish products have been modeled using Fick, Peleg, Weibull, and Z and L models (Zugarramurdi and Lupin, 1977, 1980;

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Rodger et al., 1984; Telis et al., 2003; Corzo and Bracho, 2005, 2006b, 2007, 2009; Casales et al., 2009; Czerner and Yeannes, 2010). Rodger et al. (1984) used Fick's law to model the salt and acid gain during the marination of herring. There is no available information for modeling sorbitol gain during the osmotic dehydration of fish products. Sorbitol was previously used as a plasticizer by Casales and Yeannes (2006) for marinating anchovies to avoid the deterioration of their texture and increase their shelf life.

While there is scarce scientific information pertaining to the application of empirical models for osmotic dehydration of fish in water/salt/acid solutions, there is no scientific information for the osmotic dehydration of fish in multicomponent solutions containing water, acid, salt, and sorbitol.

The aim of this work was to analyze the applicability of the Peleg, Zugarramurdi and Lupin, and Weibull models for modeling water loss and salt, acid, and sorbitol gain during marination of *Engraulis anchoita* in ternary and multicomponent solutions.

#### Materials and methods

#### Raw materials

Two blocks of anchovies (*Engraulis anchoita*), each weighing 5 kg, were used in this study. The specimens were caught in the southwestern Atlantic Ocean between 38 and 41° LS in the coastal sector of Buenos Aires, Argentina, in September. The samples were frozen on board and stored at  $-30^{\circ}$ C for 2 months.

#### Marinating process

The cold marinating process consisted of thawing, washing, heading and gutting, filleting, washing, brining, and marinating. The brining stage was carried out in a brine bath with 10 g/100 g of salt (food grade) for 1 h with a fish:solution ratio of 1:1 at room temperature (18°C). At the end of the brining stage, the anchovy fillets were composed of 76.82 water, 4.6 lipids, and 14.64 proteins with an ash content of 3.94 (all expressed as g/100 g). The sodium chloride content was 3.36 g/100 g, and the pH was 6.5. The fillets were 9.69  $\pm$  0.29 cm length, 1.88  $\pm$  0.07 cm wide, and 0.61  $\pm$  0.02 cm thick. These values represent the averages of all of the measurements taken.

## Marinating stage

At the end of the brining stage, the fillets were immersed in the ternary and multicomponent marinating solutions.

The ternary marinating solution was composed of 88.2 water, 9.1 sodium chloride, and 2.7 acetic acid (expressed in g/100 g) with a pH of 2.5. The multicomponent marinating solution was composed of 66.6 water, 22.4 sorbitol, 8.5 salt, and 2.5 acetic acid (expressed in g/100 g) with a pH of 2.3. The marinating stage was carried out in closed receptacles at  $20 \pm 1^{\circ}$ C using a solution: fish ratio of 10:1 to avoid significant dilution of the marinating solution. The anchovies were marinated in the ternary solution without agitation and with agitation at 50 rpm on an orbital shaker. Because agitation did not improve the uptake of sodium chloride and acetic acid and the equilibrium times were longer in samples stirred during marination, the marination in the water/salt/acid/sorbitol solution was carried out without agitation. The anchovy fillets and the marinating solution was reached. The equilibrium values were determined by calculating the distribution coefficients (Casales et al., 2009). Equilibrium was reached when the distribution coefficients were close to unity.

The water, sodium chloride, acetic acid, sorbitol contents, and pH were determined for the anchovy fillets at the start of the marinating stage and at 0.5-h intervals. Two experimental runs were performed.

#### Physical and chemical analyses

The water content was determined by the standard gravimetric method at 105°C until a constant weight was reached (AOAC, 1990). The sodium chloride content was determined based on chloride using the Mohr method adapted for foods (Kirk et al., 1996). The acid content was determined by titration with sodium hydroxide (Kirk et al., 1996). The sorbitol content was determined by an enzymatic colorimetric method (Bergmeyer et al., 1974). The pH value was determined using a pH meter (Model Vega VI; Parsec, Buenos Aires, Argentina) with a fish:distilled water ratio of 1:1 (AOAC, 1990). These measurements were carried out in triplicate.

#### Calculations

To consider the changing solute contents during the osmotic process, the water and solute contents were expressed on a nonsalt, nonacid, nonsorbitol dry matter basis. The dry matter was calculated as the sample weight minus the weight of water and the weight of salt, acid, and sorbitol gain (Mujaffar and Sankat, 2006). Favetto et al. (1981) noted that for an osmotic process, the nonsolute dry matter is a measure of the true dry matter of the sample.

The water loss, which represents the total amount of moisture lost by the fillet from the beginning of the process up to the sampling time, was also expressed on a dry matter basis as g gdm<sup>-1</sup> and was calculated as  $[(H_2O)_0 - (H_2O)_t]/dm$ . The solute gain, which represents the total amount of solute uptake by the slabs from the beginning of the process up to the sampling time, was also expressed on a dry matter basis as g gdm<sup>-1</sup> and was calculated as  $[(Solute)_t - (Solute)_0]/dm$  (Mujaffar and Sankat, 2006).

# Mathematical models

#### Peleg model

Peleg (1988) proposed a two-parameter sorption equation, which is expressed as:

$$x_t = x_0 \pm \frac{t}{K_1 + K_2},\tag{1}$$

where  $x_t$  is the water, salt, sorbitol, or acid (expressed on a nonsalt, nonacid, nonsorbitol dry matter basis in g gdm<sup>-1</sup>) at time t (h) and  $k_1$  and  $k_2$  are the Peleg parameters. The Peleg model can be rewritten to describe water loss and solutes gain as:

$$x_{\tau} = \frac{t}{K_1 + K_2 t},$$
 (2)

where  $x_t$  represents the water loss or solute gain,  $k_1 [h (g gdm^{-1})^{-1}]$  is Peleg's rate constant related to the mass transfer rate at the beginning of the OD process at  $x_t = x_0$  (Equation (3), and  $k_2 (g gdm^{-1})^{-1}$  is the capacity constant related to the equilibrium water and solute content at time  $t \rightarrow \infty$  (Equation 4):

$$\frac{\partial x_t}{\partial t} = \frac{1}{K_1},\tag{3}$$

$$x_{eq} = \frac{1}{K_2}.$$
(4)

#### Zugarramurdi and Lupin model (Z and L model)

Zugarramurdi and Lupin (1977, 1980) proposed an exponential model for the salt and water equilibrium values, which can be expressed as:

$$\frac{dx_t}{dt} = k(x_{eq} - x_\tau),\tag{5}$$

where  $x_t$  and  $x_{eq}$  are the water loss or solute gain (g gdm<sup>-1</sup>) at time t(h) and at equilibrium, respectively; and k (h<sup>-1</sup>) is the specific rate constant. Integrating Equation 5 with the initial condition  $x_{t=0} = x_o$ , we can obtain the following expression:

$$x_t = x_0 e^{-Kt} + x_{eq} (1 - e^{-Kt}).$$
(6)

#### Weibull model

The Weibull model describes the behavior of systems or events that have some degree of variability, such as the osmotic dehydration kinetics. The probability density function of the Weibull distribution may be written as:

$$\frac{x_t - x_{eq}}{x_0 - x_{eq}} = \exp\left[-\left(\frac{t}{\alpha}\right)^{\beta}\right],\tag{7}$$

where  $x_0$ ,  $x_t$ ,  $x_{eq}$  are the water loss or solute gain (g gdm<sup>-1</sup>) at t = 0, at time t, and at equilibrium, respectively;  $\alpha$  is the scale parameter (h);  $\beta$  is the shape parameter (dimensionless); and t is the sampling time (Corzo and Bracho, 2008, 2009).

#### Statistical analysis

All treatments were repeated two times, and all samples were analyzed three times.

The fitting of the models with the experimental data was performed by nonlinear regression using OriginPro 8 software (OriginLab Corp., Northampton, MA, USA). To evaluate the goodness of fit of the experimental data to the models, the determination coefficient ( $R^2$ ) and root mean square error (RMSE) were used according to:

$$RMSE = \sqrt{\frac{1}{n} \sum_{i=1}^{n} (x_i - x_{pi})^2},$$
(8)

where  $x_i$  is the experimental value,  $x_{pi}$  is the predicted value, and n is the number of data pairs.

The analysis of variance (ANOVA) was used to estimate the statistical significance, with a confidence level of 95% (p < 0.05). The differences between the means were analyzed using Tukey's test for post-hoc comparison. The analyses were performed using OriginPro 8 software (OriginLab).

#### **Results and discussion**

The osmotic process was studied in terms of water loss and solute gain (Figures 1–4). An initial high rate of water removal (and solutes uptake) followed by slower removal (and uptake) in the later stages was observed. The estimated parameters for the Peleg, Z and L, and Weibull models are shown in Table 1. The major advantage of the Peleg model is that the equilibrium values can be estimated using short time point experimental data (Turhan et al., 2002). In the Z and L model, the equilibrium value is a parameter of the model (Zugarramurdi and Lupin, 1977, 1980). The Weibull probabilistic distribution model is quite simple and generally gives a good description of complex processes with high variability, as is the case for osmotic dehydration (Cunha et al., 2001). The main disadvantage of these models is the limited validity within the experimental range for which the parameters of the model are obtained. In addition, these models do not take into account the size, shape, or structure of the material (Schmidt et al., 2009).

The inverse of  $k_1$  in the Peleg model, k in the Z and L model, and the inverse of  $\alpha$  in the Weibull model are related to the initial mass transfer rate. Moreover, the equilibrium content is related to the Peleg capacity constant ( $k_2$ ) and parameter  $x_{eq}$  of the Z and L model. The kinetic parameters of the



Figure 1. Fit of experimental water loss values to models during osmotic dehydration in different marinating solutions.

models for the water loss and solute gain in the multicomponent solution are significantly different (p < 0.05) from the ternary solution.

According to the  $R^2$  and RMSE statistical parameters (Table 2), the Peleg, Z and L, and Weibull models are able to predict the salt and acid gain for marinating anchovies in the water/salt/acid solution. The water loss fit to the Peleg and Weibull models for marinating in the ternary solution with agitation and fit to the Peleg, Z and L, and Weibull models for marinating without agitation. The  $R^2$  values were lower. The Peleg rate constant k1 for salt was similar to the value obtained by Corzo and Bracho (2005) in the osmotic dehydration of anchovy sheets in a water/salt solution. Marination in the multicomponent solution for water loss and salt, acid, and sorbitol gain could be modeled using the three proposed models. The high molecular weight of sorbitol reduces the salt and acid uptake, similar to the observation of Collignan et al. (2001) for simultaneous salting and drying of fish in a ternary aqueous solution containing salt and sugar. The Peleg model parameter  $1/k_1$  describes the initial rate of mass exchange. Higher values of  $1/k_1$  were obtained in the ternary solution (Table 1). These results are in accordance with the results from Azoubel and Murr (2004) for the osmotic process of cherry tomato marination in salt/sucrose solutions. The rate constants for the Z and L model (Table 1) showed the same tendency as  $1/k_1$ , which indicated that the addition of sorbitol in the marinating solution significantly decreased (p < 0.05) the uptake rate of acid and salt. According to Collignan et al. (2001), a few studies have focused on the simultaneous salting and drying of fish or meat products in a ternary aqueous solution containing salt and sugar. It has been demonstrated that the presence of sugar in the liquid phase boosts the gradient concentration between the food and the solution and enhances the water release (Collignan et al., 2001). Our results support this notion because the water loss in the multicomponent solution is higher than in the ternary solution.



Figure 2. Fit of experimental salt gain values to models during osmotic dehydration in different marinating solutions.

Furthermore, the salt uptake is limited by the presence of sugar due to the formation of a highly concentrated coating on the food (Collignan et al., 2001). In this work, the equilibrium salt gain in the multicomponent solution is higher than in the ternary solution (Table 3). This could be explained by the greater equilibrium times (Table 1).

Figures 1–4 show the fit of the experimental data to the Peleg, Z and L, and Weibull models. For most of the conditions for water loss and salt, acid, and sorbitol gain, smaller differences between the predicted and experimental data were observed. This was verified through the  $R^2$  and RMSE statistical parameters presented in Table 2.

Table 3 shows the equilibrium time, the experimental water loss, and the solute equilibrium values and the corresponding values predicted by the models. The predicted equilibrium values were obtained from Equation 4 according to the Peleg model and were also obtained directly from the parameter  $x_{eq}$  of the Z and L model.

In the ternary solution, the experimental equilibrium values with and without agitation were not significantly different (p > 0.05). For water loss and salt gain, the experimental equilibrium values in the multicomponent solution were significantly different (p < 0.05) from those in the ternary solution.

In general terms, the predicted equilibrium values agreed with the experimental results. Taking into account the experimental equilibrium values, the Peleg model overestimates the equilibrium values for water loss and acid and sorbitol gain in the multicomponent solution. On the other hand, the equilibrium values predicted by the Z and L model are more similar to the experimental values for water loss in the



Figure 3. Fit of experimental acid gain values to models during osmotic dehydration in different marinating solutions.

multicomponent solution, for acid and sorbitol gain in the ternary and multicomponent solutions, respectively, and for salt in the ternary solution, as verified by the lower E (%).

## Conclusions

The Peleg, Z and L, and Weibull models can be used to represent the kinetics of mass transfer for water, salt, acid, and sorbitol during anchovy marination. The equilibrium values predicted by the Z and L model are closer to the experimental values than those calculated by the Peleg model. The use of sorbitol in the multicomponent marinating solution decreased the rate of salt and acid uptake. These are the first reported for the use of sorbitol in a marinating solution for fish.

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Figure 4. Fit of experimental sorbitol gain to models during osmotic dehydration in different marinating solutions.

Table	1. Kinetic	parameters	for	water	loss	and	solutes	gain.

	Water loss							
Model	Peleg			Z and L	Weibull			
parameters	k <sub>1</sub> h (g gdm <sup>-1</sup> ) <sup>-1</sup>	k <sub>2</sub> (g gdm <sup>-1</sup> ) <sup>-1</sup>	k (h <sup>-1</sup> )	x <sub>eq</sub> (g gdm <sup>-1</sup> )	a (h <sup>-1</sup> )	β		
Ternary solution								
With agitation	0.561ª	2.556ª	*	*	0.323ª	0.390 <sup>a</sup>		
Without agitation	0.734 <sup>a</sup>	2.694ª	2.340 <sup>a</sup>	0.336 <sup>ª</sup>	0.403 <sup>b</sup>	0.588 <sup>b</sup>		
Muticomponent solution	3.217 <sup>b</sup>	0.975 <sup>b</sup>	0.392 <sup>b</sup>	0.719 <sup>b</sup>	3.312 <sup>c</sup>	0.912 <sup>c</sup>		
			Salt gain					
Model	Peleg			Z and L	Weibull			
parameters	k <sub>1</sub> h (g gdm <sup>-1</sup> ) <sup>-1</sup>	k <sub>2</sub> (g gdm <sup>-1</sup> ) <sup>-1</sup>	$k (h^{-1})$	x <sub>eq</sub> (g gdm <sup>-1</sup> )	$a (h^{-1})$	β		
Ternary solution								
With agitation	6.054 <sup>a</sup>	10.958 <sup>a</sup>	1.409 <sup>a</sup>	0.079 <sup>a</sup>	0.736 <sup>a</sup>	0.860 <sup>a</sup>		
Without agitation	6.716 <sup>ª</sup>	9.125 <sup>b</sup>	1.210 <sup>b</sup>	0.090 <sup>ª</sup>	0.877 <sup>a</sup>	0.900 <sup>ª</sup>		
Muticomponent solution	10.836 <sup>b</sup>	7.189 <sup>c</sup>	0.660 <sup>c</sup>	0.110 <sup>b</sup>	2.144 <sup>b</sup>	0.722 <sup>b</sup>		
	Acid gain							
Model	Peleg		Z and L		Weibull			
parameters	k <sub>1</sub> h (g gdm <sup>-1</sup> ) <sup>-1</sup>	k <sub>2</sub> (g gdm <sup>-1</sup> ) <sup>-1</sup>	k (h <sup>-1</sup> )	x <sub>eq</sub> (g gdm <sup>-1</sup> )	a (h <sup>-1</sup> )	β		
Ternary solution								
With agitation	8.979 <sup>a</sup>	11.997 <sup>a</sup>	1.130 <sup>a</sup>	0.070 <sup>a</sup>	1.046 <sup>a</sup>	0.752 <sup>a</sup>		
Without agitation	9.683 <sup>ª</sup>	11.688 <sup>ª</sup>	1.100 <sup>ª</sup>	0.070 <sup>a</sup>	0.986 <sup>ª</sup>	0.816 <sup>ª</sup>		
Muticomponent solution	20.875 <sup>b</sup>	8.010 <sup>b</sup>	0.453 <sup>b</sup>	0.092 <sup>b</sup>	1.961 <sup>b</sup>	1.043 <sup>b</sup>		
	Sorbitol gain							
Model	Peleg		Z and L		Weibull			
parameters	$k_1 h (g g d m^{-1})^{-1}$	$k_2 (g \ gdm^{-1})^{-1}$	k (h <sup>-1</sup> )	$x_{eq}$ (g gdm <sup>-1</sup> )	a (h <sup>-1</sup> )	β		
Muticomponent solution	4.343	0.824	0.285	0.778	2.410	1.500		

\*The fit with this model is not good. Mean values followed by different letters in the same column are significantly different at p < 0.05.

Solute		Peleg	Z and L	Weibull			
	Marinating in ternary solution						
		With agitation					
Salt	R <sup>2</sup>	0.957	0.976	0.975			
	RMSE	0.0024	0.0019	0,024			
Acid	R <sup>2</sup>	0.987	0.962	0.987			
	RMSE	0.0013	0.0023	0.019			
Water	R <sup>2</sup>	0.793	*	0.848			
	RMSE	0.015		0.033			
		Without	agitation				
Salt	R <sup>2</sup>	0.984	0.996	0.994			
	RMSE	0.002	0.001	0.013			
Acid	R <sup>2</sup>	0.964	0.953	0.964			
	RMSE	0.0024	0.0027	0.032			
Water	R <sup>2</sup>	0.819	0.802	0.806			
	RMSE	0.0162	0.0169	0.047			
		Marinating in multicomponent solution					
		Without agitation					
Salt	R <sup>2</sup>	0.982	0.959	0.987			
	RMSE	0.003	0.005	0.022			
Acid	R <sup>2</sup>	0.942	0.945	0.940			
	RMSE	0.005	0.005	0.061			
Sorbitol	R <sup>2</sup>	0.894	0.902	0.930			
	RMSE	0.063	0.061	0.080			
Water	R <sup>2</sup>	0.977	0.981	0.975			
	RMSE	0.027	0.025	0.034			

Table 2. Statistical parameters of Peleg, Z and L, and Weibull models.

\*The fit with the model is not good.

#### Table 3. Equilibrium values.

	Experimental		Peleg		Z and L	
Water loss	t <sub>eq</sub> (h)	x <sub>eq</sub> (g gdm <sup>-1</sup> )	x <sub>eq</sub> (g gdm <sup>-1</sup> )	E (%)	x <sub>eq</sub> (g gdm <sup>-1</sup> )	E (%)
Ternary solution						
With agitation	4.5	0.401 <sup>a</sup>	0.391 <sup>a</sup>	2.49	*	*
Without agitation	4.2	0.355ª	0.371 <sup>a</sup>	4.51	0.336 <sup>a</sup>	5.35
Multicomponent solution	8.0	0.818 <sup>a</sup>	1.030 <sup>b</sup>	25.92	0.719 <sup>a</sup>	12.10
	Ex	perimental	Peleg		Z and L	
Salt gain	t <sub>eq</sub> (h)	x <sub>eq</sub> (g gdm <sup>-1</sup> )	$x_{eq}(g \ gdm^{-1})$	E (%)	$x_{eq}(g \ gdm^{-1})$	E (%)
Ternary solution						
With agitation	5.0	0.081 <sup>a</sup>	0.091 <sup>a</sup>	12.35	0.079 <sup>a</sup>	2.47
Without agitation	4.2	0.093 <sup>a</sup>	0.110 <sup>a</sup>	18.28	0.090 <sup>a</sup>	3.33
Multicomponent solution	7.0	0.127 <sup>a</sup>	0.139 <sup>a</sup>	9.45	0.110 <sup>a</sup>	13.39
	Ex	perimental	Peleg		Z and L	
Acid gain	t <sub>eq</sub> (h)	x <sub>eq</sub> (g gdm <sup>-1</sup> )	x <sub>eq</sub> (g gdm <sup>-1</sup> )	E (%)	x <sub>eq</sub> (g gdm <sup>-1</sup> )	E (%)
Ternary solution						
With agitation	5.0	0.071 <sup>a</sup>	0.083ª	16.90	0.070 <sup>a</sup>	1.41
Without agitation	4.0	0.073 <sup>ab</sup>	0.086 <sup>a</sup>	17.81	0.070 <sup>bc</sup>	4.11
Multicomponent solution	6.0	0.086 <sup>a</sup>	0.125 <sup>b</sup>	45.35	0.092 <sup>a</sup>	6.98
	Ex	Experimental Peleg			Z and L	
Sorbitol gain	t <sub>eq</sub> (h)	$x_{eq}$ (g gdm <sup>-1</sup> )	$x_{eq}$ (g gdm <sup>-1</sup> )	E (%)	x <sub>eq</sub> (g gdm <sup>-1</sup> )	E (%)
Multicomponent solution	8.0	0.643ª	1.21 <sup>b</sup>	88.18	0.778 <sup>a</sup>	20.99

\*The fit with this model is not good. Mean values followed by different letters in the same row are significantly different at p < 0.05.

 $E = \left|\frac{x_i - x_{pi}}{x_i}\right|$  100, where E is relative error,  $x_i$  is experimental value,  $x_{pi}$  is calculated value.

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