

ORIGINAL ARTICLE

Kinetic of peroxide index variation and shelf life of mayonnaise with microencapsulated oregano essential oil

Cinética de variación del índice de peróxido y vida útil de mayonesa con aceite esencial de orégano microencapsulado

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ABSTRACT

This work aimed to evaluate the influence of adding microencapsulated oregano (Origanum vulgare L.) essential oil (MOEO) on the stability of mayonnaise. Three formulations were developed with MOEO at different concentrations (0.0, 0.26, and 0.44%). Mayonnaises were stored at 35, 45, and 55 °C until deterioration. The titratable acidity and peroxide index (PI) were determined during the accelerated storage. PI was subjected to linear regression analysis as a function of time. The parameters of the Arrhenius model (k and Ea) for the variation of PI and the temperature acceleration factor (Q10) were also determined. Mayonnaise, with 0.44% MOEO, presented the highest stability in delaying the increase in the PI, indicating the deterioration of this type of product. In all cases, the PI variation was adjusted to a zero-order reaction. Linear models were obtained to estimate the shelf life of mayonnaises.

Keywords: mayonnaise, oregano essential oil, microencapsulation, peroxide index.

RESUMEN

Este trabajo tuvo como objetivo evaluar la influencia de la adición de aceite esencial de orégano (Origanum vulgare L.) microencapsulado (MOEO) en la estabilidad de mayonesas. Se desarrollaron tres formulaciones con MOEO a diferentes concentraciones (0,0; 0,26 y 0,44 %). Las mayonesas se almacenaron a 35, 45 y 55 °C hasta su deterioro. Durante el almacenamiento acelerado se determinó la acidez titulable y el índice de peróxidos (IP). El IP se sometió a un análisis de regresión lineal en función del tiempo. También se determinaron los parámetros del modelo de Arrhenius (k y Ea) para la variación del IP y el factor de aceleración de la temperatura (Q10). Las mayonesas con 0,44 % de MOEO presentaron la mayor estabilidad en términos de retardar el aumento del IP, como indicador del deterioro de este tipo de productos. La variación del IP se ajustó, en todos los casos, a una reacción de orden cero. Se obtuvieron modelos lineales para estimar la vida útil de las mayonesas.

Palabras clave: mayonesa, aceite esencial de orégano, microencapsulación, índice de peróxido.



INTRODUCTION

Lipid oxidation in food products such as mayonnaise results in the appearance of rancidity and deterioration, making these products unfit for human consumption. Mayonnaise is an oil-in-water emulsion with a low pH and an oil content between 65 and 85% (Mattia et al., 2015).

One of the most effective alternatives to delay lipid oxidation in this product is the addition of antioxidants such as butyl hydroxyanisole and butylated hydroxytoluene. However, their use in food has been restricted due to their adverse effect on enzymes in human organs (Bakkali et al., 2008). This has motivated the development of formulations with antioxidants obtained from natural sources (Amorati et al., 2013; García et al., 2014; Gavilanez & Rojas, 2024). This is how mayonnaise has been made with poly ε -caprolactone nanoparticles loaded with thyme essential oil (Passos et al., 2019), tocopherol, rosemary essential oil, and Ferulago angulata (Schlecht) Boiss extract (Alizadeh et al., 2019) and microencapsulated phenolic compounds from lemon waste (Shaygannia et al., 2021).

Although various research has been done on using natural antioxidants in the formulation of mayonnaise, the actual effect of these phytochemical compounds should continue to be studied in depth. Although they show antioxidant activity in vitro and in vivo, they could not favor the preservation of the product. Thus, for example, it has been reported that gallic acid showed a pro-oxidant activity in mayonnaise, possibly due to its ability to reduce metal ions to their most active form, such as Fe3+ to Fe2+ (Jacobsen et al., 2014).

Spray-drying has been used earlier to microencapsulate oregano essential oil (MOEO) (Rojas-Molina et al., 2022; Plati et al., 2021). However, other wall materials were used in these studies, which differed from maltodextrin-gum Arabic combinations. Considering the above, this work aimed to estimate the kinetic parameters of the variation of the peroxide index (PI) and shelf life of mayonnaise with microencapsulated oregano (Origanum vulgare L.) essential oil as a natural antioxidant during its accelerated storage.

METHODOLOGY

The emulsion consisted of 7% m/m of oregano (O. vulgare) essential oil, 9.3% m/m gum Arabic, 18.7% m/m maltodextrin, and 65% deionized water (35% m/m total solids feed). According to Rojas-Molina et al. (2022), the microcapsules were obtained by spray drying.

The mayonnaises of each treatment (10kg) were prepared under the same conditions. Ethylenediaminete tracetic acid was dissolved in an aliquot of the control treatment formulation oil (T1) and then mixed with the rest. The MOEO was added with the soybean oil during the emulsion formation to obtain concentrations of 0.26 and 0.44% for the treatments T2 and T3, respectively. The determination of the maximum concentration of MOEO to be used in the formulation of mayonnaise, without affecting the color, odor, and taste, was carried out through an acceptance/rejection test in group sessions with 10 semi-trained judges. The concentration of MOEO varied between 0 and 1.0% (m/m).

All the solid ingredients (white sugar, common salt, starch, citric acid, Guar gum, xanthan gum, mustard flour) were mixed for about 1 min, except the MOEO, to facilitate its homogenization during emulsification. Chlorinated water was then added and mixed for 2 to 3 min until the paste was homogenized due to the hydration of the starches. The eggs were then added, and, at the end, the refined soybean oil at 12 $^{\circ}$ C and the pasteurized white vinegar were slowly incorporated, homogenizing the mixture until the emulsion was obtained.

The mayonnaises were packed in the presence of air in the headspace of 270 g glass jars with a metallic twist off top (\emptyset = 63 mm) and then stored at 35, 45, and 55 °C for a variable time, depending on the formulations and temperatures, until the samples were considered objectionable.

A sample was dissolved in a mixture of ethanol/petroleum ether with an ethanolic hydroxide of potassium (AOAC, 2012) to determine the titratable acidity (% m/m acetic acid). The PI (meq O2/kg of oil) was determined by an iodometric method with a visual endpoint detection after treating the mayonnaise to separate the oil from the emulsion (ISO 3960, 2017). The parameters of the Arrhenius model (k and Ea) for the variation of the PI during the storage were estimated. The temperature acceleration factor (Q10) was also determined according to Nascimento et al. (2013).

ANOVA was performed with the Statistica program (v. 7, 2004, StatSoft. Inc., Tulsa, USA), and when a significant difference was detected ($p \le 0.05$), Duncan's multiple range test was applied.

PI during storage was subjected to linear regression analysis as a function of time. The ANOVA's R2 and the lack of fit test were used to choose the best-fit kinetic model, with a significance level of 5%.

RESULTS AND DISCUSSION

The kinetics of the variation in IP during accelerated storage of mayonnaise sauces showed a better fit, in all cases, for a pseudo-zero-order reaction, with adjusted R^2 values between 0.8945 and 0.9791. Table 1 presents the results of the variance analysis of the kinetic models of this variation in mayonnaise sauces.

– <i>i</i>		Pseudo-					
lemperature (°C)	Treatment*	order of reaction	Model**	R	R ²	R ² adjusted	р
		0	PI = 0.1434t-0.9835	0.976	0.952	0.945	0.000007
	T1	1	ln PI = 0.0343t-0.0339	0.968	0.938	0.931	0.000001
		2	1/PI = -0.0103t+0.763	0.945	0.892	0.881	0.000012
		0	PI = 0.0952t+0.7105	0.972	0.944	0.939	0.000000
35	Т2	1	ln PI = 0.0217t+0.4515	0.970	0.942	0.937	0.000000
		2	1/PI = -0.0062t+0.5714	0.908	0.824	0.810	0.000007
		0	PI = 0.0847t+0.6003	0.983	0.967	0.964	0.000000
	Т3	1	ln PI = 0.0198t+0.3438	0.981	0.963	0.960	0.000000
		2	1/PI = -0.0056t+0.6005	0.952	0.906	0.899	0.000000
		0	PI = 0.2615t+1.2349	0.983	0.966	0.957	0.000445
	T1	1	ln PI = 0.0616t+0.4415	0.935	0.873	0.842	0.006302
		2	1/PI = -0.0207t+0.6739	0.833	0.694	0.618	0.039353
	T2	0	PI = 0.217t+1.7762	0.986	0.971	0.966	0.000048
45		1	ln PI = 0.0458t+0.6982	0.922	0.850	0.820	0.003124
		2	1/PI = -0.0132t+0.5307	0.802	0.643	0.572	0.030051
	Т3	0	PI = 0.1873t+2.1466	0.955	0.912	0.895	0.000804
		1	ln PI = 0.0385t+0.7545	0.903	0.816	0.779	0.000528
		2	1/PI = -0.0105t+0.5067	0.827	0.683	0.620	0.021869
		0	PI = 0.2171t+4.2667	0.993	0.986	0.979	0.007005
	T1	1	ln PI = 0.0282t+1.5701	0.981	0.962	0.942	0.019423
		2	1/PI = -0.0038t+0.1962	0.962	0.926	0.889	0.037689
		0	PI = 0.1648t+4.4333	0.979	0.958	0.944	0.003745
55	Т2	1	ln PI = 0.022t+1.5789	0.960	0.922	0.896	0.009528
		2	1/PI = -0.003t+0.1974	0.932	0.868	0.824	0.021312
		0	PI = 0.2299t+1.2977	0.965	0.931	0.907	0.007942
	Т3	1	ln PI = 0.0497t+0.5975	0.950	0.902	0.869	0.013468
		2	1/PI = -0.0143t+0.5421	0.848	0.719	0.625	0.069680

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Note. *T1: control treatment; T2: treatment with adding oregano EO microcapsules at 0.26% m/m; T3: treatment with adding oregano EO microcapsules at 0.44% m/m.

**Estimated by determining the peroxide index (PI) expressed as 0, in meq kg⁻¹; t: time expressed in days.

Table 2 shows the models for estimating the shelf life of the mayonnaise sauces for each treatment. A high significance of the linear models for estimating the shelf life is observed, with a confidence level of 95.0% and adjusted R^2 values of 0.939, 0.994, and 0.914 for T1, T2, and T3, respectively.

Treatment	Model	R ²	R ² adjusted
T1	ln t = 0.018T + 3.7085	0.970	0.939
T2	ln t = 0.011T + 3.1239	0.997	0.994
Т3	ln t = 0.018T + 2.6657	0.957	0.914

Note. T1: control treatment; T2: treatment with adding oregano EO microcapsules at 0.26% m/m; T3: treatment with adding oregano EO microcapsules at 0.44% m/m. t: shelf life (d), T: temperature (°C).

These values indicated that the adjusted models explain more than 91.4% of the variability in estimating the shelf life of the mayonnaise sauce. Table 3 shows the specific rate constants and lifetime of the different treatments.

Temperature	Treatment	k	Estimated shelf life	
(°C)	incutinent	(meq O ₂ /kg/d)	(d)†	
	T1	0.143	76	
35	Т2	0.095	97	
	Т3	0.085	110	
	T1	0.262	33	
45	T2	0.217	37	
	Т3	0.187	41	
	T1	0.217	26	
55	Т2	0.165	33	
	Т3	0.230	37	

 Table 3. Specific rate constant and shelf life of mayonnaise sauces.

Note. T1: control treatment; T2: treatment with adding oregano EO microcapsules at 0.26% m/m; T3: treatment with adding oregano EO microcapsules at 0.44% m/m.

k: specific rate constant.

 $^{\scriptscriptstyle \dagger}$ Considering rejection for a limiting peroxide index of 10 meq/kg (NC 50, 2015).

During accelerated storage, the protective effect of microencapsulated oregano EO was observed, with an increase of 34, 8, and 11 days at 35, 45, and 55 °C, respectively, in the treatment with the addition of oregano EO microcapsules at 0.44% m/m compared to the control batch and considering the product objectionable when it reached a limit value of the IP equal to 10 meq/kg (NC 50, 2015). This represents an increase in durability of 45, 24, and 42% m/m for 35, 45, and 55 °C, respectively. The rate constant generally increased with temperature, so the highest formation of peroxides occurred at 55 °C.

These rate constants can be used to determine the influence of temperature variations through the acceleration factor Q_{10} (Pedro & Ferreira, 2006). This factor indicates the number of times the rate of a deterioration reaction changes when the temperature is varied by 10 °C and is a practical and reliable way of predicting the effect of variations in storage temperature on food.

A tendency was found for the T3 treatment to be more susceptible to temperature variation in the range of 35 to 45 °C (Table 4). However, when the higher temperature range (45 to 55 °C) is analyzed, the opposite behavior is observed and could be related to the presence of microencapsulated EO.

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Trootmont	Q ₁₀ [§]	
meatment	35 a 45 °C	45 a 55 °C
T1	2.29	1.27
Т2	2.57	1.12
Т3	2.65	1.11

Note. T1: control treatment; T2: treatment with adding oregano EO microcapsules at 0.26% m/m; T3: treatment with adding oregano EO microcapsules at 0.44% m/m.

⁵Values estimated through the relationship between shelf life (=) at a temperature (T) and shelf life at T+10 °C.

Figure 1 presents the graphs using the Arrhenius equation for the three studied temperatures. The Arrhenius equation was established by the relationship of $\ln k$ and 1/T, where the ordinate at the origin is $\ln A$ and the slope is -Ea/R.





The activation energy of chemical reactions in foods is usually between 24,000 and 150,000 J/mol. Ea values higher than 65,000 J/mol are related to the high susceptibility of the attribute to deterioration due to increased temperature (Touati et al., 2016). As seen in Table 5, the Ea values for the variation of the IP were between 13,886.84 and 22,243.38 J/mol. As a trend, as the concentration of microencapsulated EO increased, Ea decreased, with treatment T3, with 0.44% m/m EO, having the lowest Ea. This is also related to the highest Q_{10} value found for that treatment.

Table 5.	Arrhenius	equations	for the	variation o	f the	peroxide v	/alue in	mayonnaise	sauces
			,		,				

Treatment	Arrhenius equations	R ²	R ² adjusted	Ea
				(J/mol)
T1	ln k = 2676.7 (1/T) - 10.673*	0.917	0.834	22 243.38
T2	ln k = 1688.8 (1/T) - 6.826*	0.998	0.995	14 033.93
Т3	ln k = 1671.1 (1/T) - 6.856*	0.862	0.725	13 886.84

Note. T1: control treatment; T2: treatment with adding oregano EO microcapsules at 0.26% m/m; T3: treatment with adding oregano EO microcapsules at 0.44% m/m. T: temperature (K); k: rate constant (meq 0,/kg/d).

CONCLUSIONS

Mayonnaise, with 0.44% MOEO, presented the highest stability during accelerated storage in terms of delaying the increase in the PI, which indicates the deterioration of this type of product. Adding the MOEO did not affect (p > 0.05) the product's sensory attributes. However, the judges reported its influence on the typical odor and flavor compared to the control mayonnaise. The variation of the PI in the mayonnaises was adjusted, in all cases, to a zero-order reaction. Linear models were obtained to estimate the shelf life of mayonnaise for each treatment.

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Conflicts of Interest:

The authors declare no conflicts of interest. Author Contributions:

Jaime O. Rojas-Molina, Mario A. García y Jorge A. Pino: data curation, formal analysis, research, methodology, supervision, validation, visualization, writing of the original draft and writing, review, and editing. **Disclaimer/Editor's Note:**

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