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Use of Orange Peel (*Citrus X sinensis*) for Obtaining Essential Extract as a Natural Degreaser

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ABSTRACT

The growth of industries encourages the increase of waste, and in turn the implementation of eco-friendly techniques and technologies for the treatment and use of them. The objective of the research was to take advantage of the epicarp of *Citrus sinensis* L. Osbeck to obtain an essential extract and formulate a natural degreaser. A diagnosis was made on the production of organic waste by interviewing a company, resulting that, of the total amount of different fruits processed, half were waste without any type of use the essential extract was obtained with the technique of direct extraction to reflux and its quality was determined by measuring the parameters: pH, total solids, percentage of acidity, relative density, and determination of limonene. The essential extract (100–125–150 mL) and betaine (11–15%) were used as study factors and as substances not harmful to the environment for the formulation of the natural degreaser through a bifactorial design and ANOVA and Tukey test at 5% significance. The pH showed significant differences because of the interaction of essential extract*betaine. The use of 150 ml of orange extract and 15% of betaine (A_2B_1) obtained a greater detachment of lubricating grease, corresponding to the T_6, with a value of 2471 NTU and a pH of 5.46 values that were analyzed by spectrophotometry.

Keywords: betaine, epicarp, natural extract, degreaser, glycerin.

INTRODUCTION

In the world, a large amount of organic waste is generated during the agro-industrial process, 30% of the production is lost during collection, processing, and final consumption, that is, around 1,300 million tons/year [Gómez, 2018]. In South America, the growing generation of organic waste has become an environmental and economic problem for industries, being responsible for covering the high costs of treatments and final disposal [Peñaranda et al., 2017]. For his part, Solíz [2015] reports that, in Ecuador, approximately 58,829 tons of organic waste originate per week, of which only 20% is disposed of in adequate conditions and the rest is distributed in adverse dispositions for the ecosystem. Therefore, Rojas et al. [2019] mention that the non-use of these residues negatively affects the environment due to the high concentration of organic matter. The environmental impact of the accumulation of organic matter from these wastes is usually due to improper handling in the final disposal, where, through the different chemical and biological processes, they generate alterations to the soil and water resources through runoff [Cury et al., 2027].

The techniques of use of this waste applying the circular economy, requires knowledge of the properties and components that constitute it. Based on this, Cabrera et al. [2016] indicate that more appropriate technologies can be developed to achieve greater effectiveness in their use; as proposed by Barrantes et al. [2019] to obtain essential extract by the method of direct reflux extraction. In addition, Rincón et al. [2016] indicate that citrus residues have high bioactive amounts (limonene), which can be used for various applications, such as cleaning products.

The use of chemical degreasers has improved the life scenarios of the population, but at the same time, they have caused adverse effects, due to pollutants and chemical or synthetic substances that contain concentrations high enough to cause damage and negative effects on human health and/or the environment according to the Pan American Health Organization (PAHO) [Euskadi, 2019]. With this background, this research proposes to apply a method that facilitates the obtaining of the essential extract and transform it into a natural degreaser, to replace degreasers of chemical and polluting nature, to promote technology transfer, innovation, and entrepreneurship, promoting change of the productive matrix through the link between the public and private sectors. In addition to originating initiatives for the use of waste that strengthen the circular economy.

MATERIALS AND METHODS

Area of study

The research was carried out at the Escuela Superior Politécnica Agropecuaria de Manabí, Manuel Félix López (ESPAM), in the Environmental Chemistry Laboratory, located in the province of Manabí, Bolívar canton, Calceta parish, El Limón site (Figure 1).

Factors under study

Table 1 represents the study factors in the research, which consist of the amount of extract obtained from the orange peel (epicarp of the fruit) and the dose of betaine that acts as a foaming agent finally the mixture was complemented with glycerin that works as a wetting agent referenced in the methodology of Quiroz [2009].

Experimental design

The methodology of Gutiérrez and De la Vara [2008] was applied, using the additives used in this research for the formulation of the natural degreaser, also, as evidenced in Table 2, a factorial design was used to manipulate the components of the experimentation, determining the different reactions between factors A and B (A: doses of orange extract and B: betaine), which were the research treatments. The design is given based on the application of the three levels of the essential extract and the two levels of betaine.

Experimental units

For the experimental units, samples of essential extract were taken at three different levels and combined with two different doses of betaine, and three repetitions were performed per treatment,

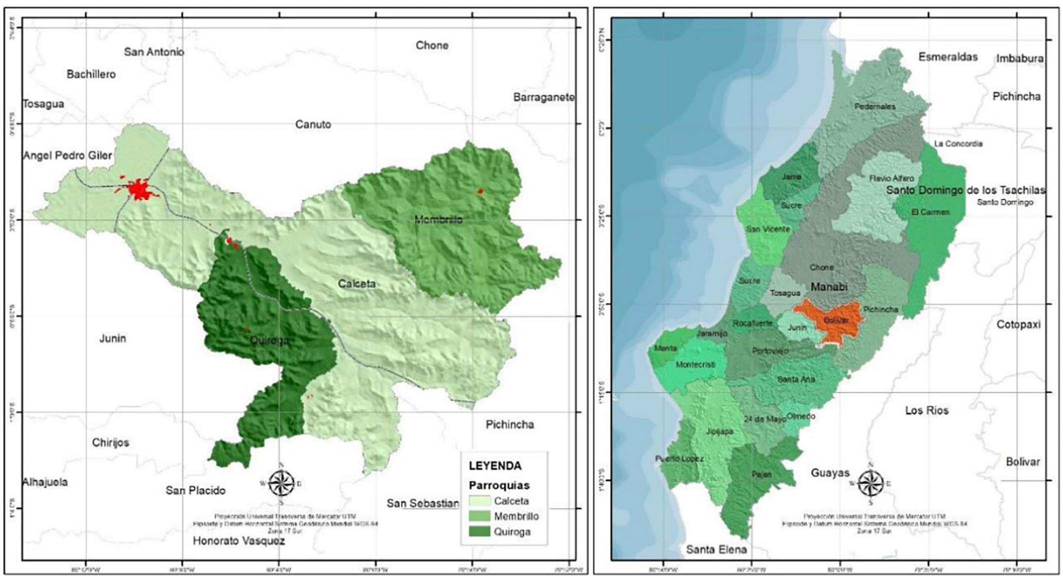

Figure 1. Maps

Table 1. Factors and levels used in bifactorial DCA

Extract from the orange peel (A)	Dose of betaine (B)
A_0 = 100 ml	B_0 = 11%
A_1 = 125 ml	B_1 = 15%
A_2 = 150 ml	

consequently, glycerin was added to complement the experimental unit (Table 3).

Statistical analysis

Once the assumptions were met, analysis of variance (ANOVA) was applied. The normality of the data (modified Shapiro-Wilks) and the homogeneity of the variance residuals (Levenne test) were checked. Comparison of means for variables with statistical significance was performed with Tukey's test at 5% significance. Analyses were performed with the Minitab statistical package.

Diagnosis of the generation of citrus waste

To verify the processes and activities carried out by the company, an interview was conducted to obtain information about the production, processing of fruits, the months of highest and lowest production, types of waste, the amount they generate, and the existence of treatments in their final disposal.

Obtain the essential extract

To obtain the essential extract from the orange peel, the direct reflux extraction method

Table 2. Experimental design

Tukey's test	DCA bifactorial		Levels	
	Numbers of treatments	Repetition numbers	Essential extract	Betaine
95%	6	3	A_0 = 100 ml	B_0 = 11%
			A_1 = 125 ml	B_1 = 15%
			A_2 = 150 ml	

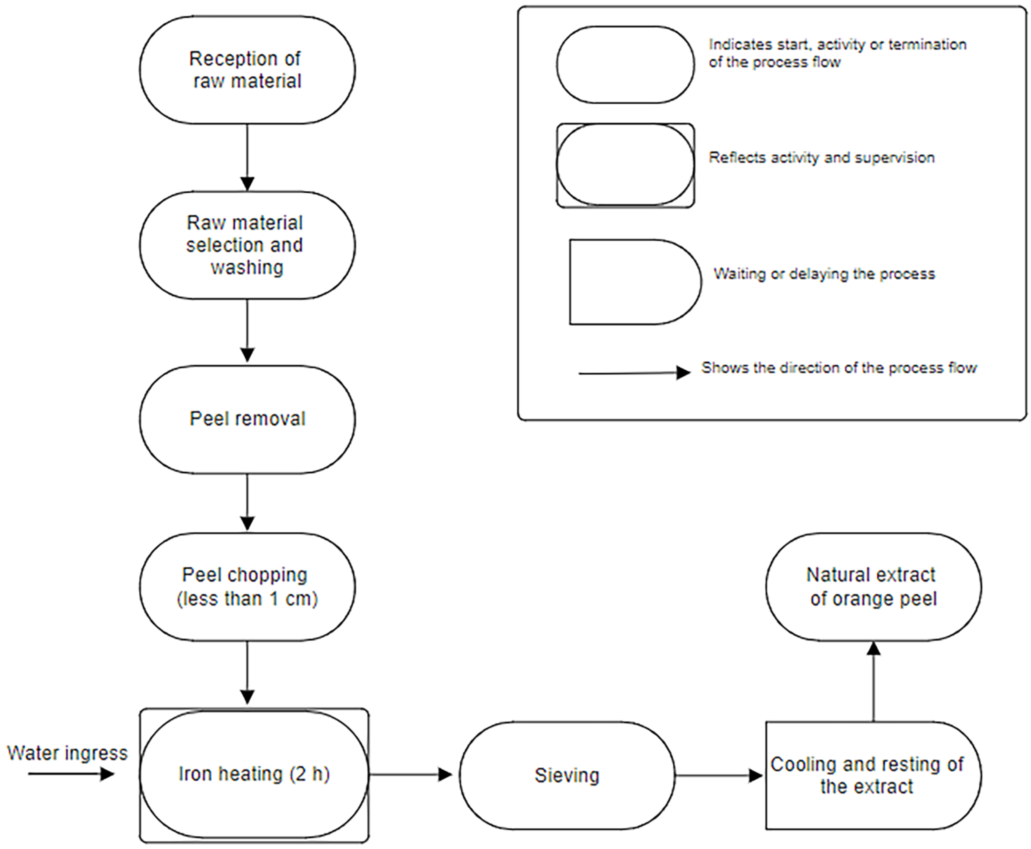

Figure 2. Process diagram for obtaining the essential extract

Tabla 3. Experimental units

Treatments	Amount of extract	Dose of betaine (11% and 15%)	Dose of glycerin	Repetitions
T_1	A_0 = 100 ml	B_0 = 11%	22%	3
T_2	A_0 = 100 ml	B_1 = 15%		
T_3	A_1 = 125 ml	B_0 = 11%		
T_4	A_1 = 125 ml	B_1 = 15%		
T_5	A_2 = 150 ml	B_0 = 11%		
T_6	A_2 = 150 ml	B_1 = 15%		

was used (Figure 3), according to the methodology proposed by Barrantes et al. [2019].

Parameters evaluated to the essential extract and the natural degreaser

Table 4 represents the parameters of the evaluation of the essential extract and the natural degreaser obtained. The quality of the extract was referred to according to previous work by Ochoa et al. [2013], on the other hand, the efficiency of the natural degreaser was carried out by applying the washing technique as exposed by the World Health Organization [2017]. A representative water sample was collected for further analysis, applying the methodology of Altmajer [2004], in which the effectiveness of cleaning by spectrophotometry (NTU) was verified, this being the method that allowed measuring the absorbance of the representative substance. The turbidimeter used was "Milwaukee Mi 415" brand. The procedures of the measured parameters for the quality of the essential extract are detailed below:

- determination of limonene – 5 ml of sample was taken where 0.5 g of potassium permanganate was added and according to the color the presence of limonene was detected, being positive the brown color.
- total solids – 5 ml of sample was placed in a clean porcelain capsule; it was evaporated at 250 °C until the residue dried, then it was passed to an oven with a temperature of 105 °C for 2 hours. The capsule was removed from the stove and placed in the desiccator for 30 minutes until it reached room temperature and proceeded to weigh it. Finally, Equation 1 was applied to obtain results from the total solids:

$$\%ST = \frac{FSW - ECW}{SW} \times 100 \quad (1)$$

where: FSW – final sample weight, ECW – empty capsule weight, SW – sample weights.

- relative density – the weight of the beaker to be used was considered as (m_1). Subsequently, the precipitation vessel was weighed again once washed and distilled water (m_2) was placed, finally the precipitation vessel was weighed with the sample obtained (m_3) and to

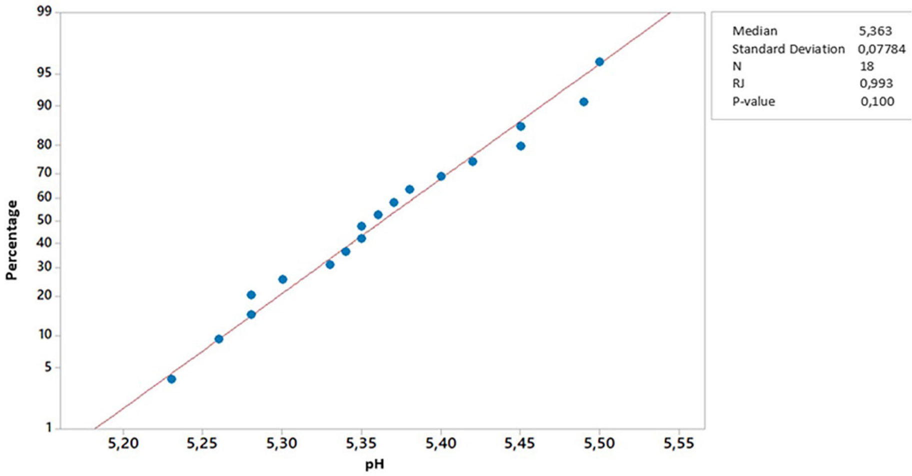

Figure 3. Normality test of the pH of the amount of extract/dose of betaine

Table 4. Representation of experimental units

Essential extract		The natural degreaser	
Parameters	Units	Parameters	Units
Limonene	–	pH	Units of pH
Total solids	mg/l	Turbidity	NTU
Relative density	kg / m³		
pH	Units of pH		
% acidity	%		

determine the relative density the following equation was applied Equation 2:

$$d = \frac{m_3 - m_1}{m_2 - m_1} \quad (2)$$

- pH – it was measured by a potentiometer "Milwaukee EC 60" brand, which was placed inside a precipitation vessel containing 80 ml of previously obtained sample.
- % acidity – it was calculated by titration, placing 5 ml of sample in a beaker, then 5 drops of 1% phenolphthalein were added and finally proceeded to titrate with sodium hydroxide, where the consumption value of this was obtained to determine the acidity the following equation was applied Equation 3:

$$\%A = \frac{ConsumptionHS.normality.mq.100}{vol.sample} \quad (3)$$

RESULTS

Determining the amount of waste generated in an industry

The interview was conducted in the company "Jugos Sunset", located in the province of Guayas, city of Guayaquil. Approximately 300 kg of waste generated from fruit is obtained, which results in the development of environmental alternatives (Table 5).

Obtaining and characterization of the essential extract for natural degreaser

The extraction process was carried out for 3 days, using the method of direct reflux extraction, obtaining 2800 mL of the essential extract from 700 g of orange peel. Then we proceeded to perform the physicochemical characterization of the natural extract obtained (Table 6), and the qualitative determination of limonene, which was positive for the verification of its active ingredient.

Formulation and characterization of the natural degreaser

The parameters evaluated for the natural degreaser were pH and turbidity (Table 7).

Statistical analysis

Analysis of variance of pH

Table 8 shows the analysis of variance. Due to the p-value of less than 0.05, there is a significant difference in the pH of the experimental units in relation to the amount of extract; however, with regard to the doses of betaine, it is determined that there is no significant difference since the p-value of the test is greater than 0.05. Likewise, it is expressed that, as for the interaction's quantity of extract/dosage of betaine, there is no significant difference either, given that the p-value is greater. This statistical

Table 6. Physicochemical analysis of the essential extract

Parameters	Units	Results
pH	–	4.7
% acidity	%	0.11
Relative density	g/ml	1.009
Total solids	mg/l	2.03

Table 5. Amount of waste generated from industrialized fruits

Fruit	Fruit purchased (kg)	Extracted pulp (kg)	Efficiency (%)	Waste generated (kg)	Frequency
Orange	600	300	50	300	Weekly
Naranjilla	20	168	80	4	Weekly
Lemon	200	50	25	150	Weekly
Passion fruit	50	15	30	35	Weekly
Pineapple	20	17	85	3	Weekly
Strawberry	50	42	85	8	Weekly

Table 7. Representation of the analysis of the natural degreaser

Treatments	Amount of extract	Dose of betaine	Dose of glycerin	pH	Turbidity (NTU)		
					Before	After	Fat shedding
T_1	100	11 %	22%	5.29	67.00	1 590	1 523
T_2	100	15%	22%	5.29	68.00	1 163	1 095
T_3	125	11%	22%	5.34	52.54	2 103	2 051
T_4	125	15%	22%	5.36	50.78	2 250	2 199
T_5	150	11%	22%	5.44	76.67	2 227	2 150
T_6	150	15%	22%	5.46	59.09	2 530	2 471

Table 8. Analysis of variance of pH

F.V.	SC	gl	CM	F	p-valor
Amount of extract	0.08	2	0.04	24.17	0.0001
Dose of betaína	5.6E-04	1	5.6E-04	0.33	0.5769
Amount of extract* dose of betaína	5.4E-04	2	2.7E-04	0.16	0.8529
Error	0.02	12	1.7E-03		
Total	0.10	17			

value agrees with research by Uc and Delgado [2012], who point out that, in the formulation of the degreaser, no greater significance of pH will be obtained among the experimental units, because this parameter maintains a relationship with the percentage of acidity, which makes a synergistic reaction to the lubricating grease. According to Tukey's test applied to pH, concerning the amount of extract, it was obtained that, if there were two different groups of the experimental units, the treatment containing 150 ml of extract obtained the best results (Table 9). Regarding the pH in the betaine dose, Table 10 indicates that, by Tukey's test, only one group was found, and their means were not significant to each other.

Table 11 shows the statistical significance of the pH means by combining the amount of betaine extract/dose, showing that the highest pH was reported in T6 with an average of 5.46. Based on the results obtained in Table 12, the normality and homoscedasticity test was applied. According to Balluerka et al. [2002], the Shapiro-Wilk test should be applied in the case of handling data less than 50, for which these results should be converted, using logarithm for their respective correlation.

Díaz [2009] states that, if the significance is greater than 0.05, the data will maintain a normal distribution, which is reflected in a linear model detailed in Figure 3. Regarding the homoscedasticity test, Galindo [2020] mentions that the Levenne test should be applied, in which, if the significance is greater than 0.05, the null hypothesis (H_0) is accepted, that is, that there is homoscedasticity between their variances, which agrees with what was developed in this research, giving a p-value of 0.111, as shown in Table 13. The Figure 3 also shows the graphic of the results the normal test of pH of the amount of extract/dose of betaine.

Table 10. Tukey's pH test of betaine doses

Betaine	N	Stocking	Grouping
11	9	5.35778	A
15	9	5.36889	A

Table 9. Tukey's test of the pH of the extract amounts

Extract	N	Stocking	Grouping	
100	6	5.28833		B
125	6	5.35000		B
150	6	5.45167	A	

Table 11. Tukey's test of the amount of betaine extract/dose

Extract* betaine	N	Stocking	Grouping		
100 11	3	5.29000			C
100 15	3	5.28667			C
125 11	3	5.34333		B	C
125 15	3	5.35667	A	B	C
150 11	3	5.44000	A	B	
150 15	3	5.46333	A		

Table 12. Test of normality of the pH of the amount of extract/dose of betaine

Parameter	Kolmogorov-Smirnov(a)			Shapiro-Wilk		
	Statistical	gl	Sig.	Sstatistical	gl	Sig.
pH	0.089	18	0.200(*)	0.974	18	0.869

Table 13. Homoscedasticity test of the pH of the amount of betaine extract/dose

Test of homoscedasticity of variances			
pH			
Statistical level	df1	df2	Sig.
2.291	5	12	0.111

Analysis of variance of turbidity

The analysis of variance of the experimental units applied shows that, in turbidity, there is no difference in terms of the amount of extract, betaine dose and the interaction amount of extract/betaine dose, since the p-values were greater than 0.05, as shown in Table 14. Therefore, Acosta [2017] attributes that this parameter will not determine greater significance, since all experimental units are a function of the release of lubricating grease, referring to the presence of particles in suspension. Regarding the other variables taken for the study, it was evident that both the analysis of variance of the experimental units applied shows that, in the turbidity before and after and the detachment of fat, there is no difference in terms of the amount of extract, dose of betaine and the interaction amount of extract/dose of betaine, since the p-values were greater than 0.05.

Also, in the Tukey tests performed, it was evidenced that there is a single homogeneous group in each variable, evidencing that, the experimental units have the same effectiveness in all treatments, therefore, any of the amounts of extract can be used as well as any of the doses of betaine.

Table 15 details that all experimental units can release lubricating grease, however, it can be shown that the combination A_2B_1 corresponding to the T_6 treatment (150 ml of extract and 15% betaine) obtained a greater detachment of fat with

Table 15. Analysis of the best treatment

Amount of extract	Dose of Betaine	NTU
A_0 100 ml	B_0 11%	1 095.33
A_0 100 ml	B_1 15%	1 523.00
A_1 125 ml	B_0 11%	2 050.54
A_1 125 ml	B_1 15%	2 150.00
A_2 150 ml	B_0 11%	2 199.22
A_2 150 ml	B_1 15%	2 470.91

Table 14. Analysis of variance of turbidity

F.V.	SC	gl	CM	F	p-valor
Amount of extract	3404813.01	2	1702406.51	2.40	0.1331
Dose of betaína	879.06	1	879.06	1.2E-03	0.9725
Amount of extract* dose of betaíne	461107.26	2	230553.63	0.32	0.7290
Error	8524224.82	12	710352.07		
Total	12391024.15	17			

a value of 2470.91 NTU which was verified by physicochemical analysis in a spectrophotometer.

DISCUSSION

Peñaranda et al. [2017] mention that, in industries that work with raw material from agriculture, there is approximately generation of 120 million t/year of waste between husks and seeds, of which 40%, that is, approximately 20 million tons are reused for other activities favorable to the environment. Vásquez [2019] states that, within industrial processes, there are different percentages, for example, in terms of soft fruits such as strawberry, blackberry, and guava, a lower amount is generated compared to fruit residues with stronger rinds such as passion fruit, pineapple, orange, soursop, and lemon.

According to the characteristics of the extract, the pH was 4.7, which agrees with what was reported by Cerón and Cardona [2011], who states that the proximal value of the extract obtained from the orange peel for use should be higher than 4.5. However, the pH varies depending on the harvest seasons, however, this does not influence its active ingredient. On the other hand, the relative density was 1.009 g/ml, considered similar data to that exposed by the authors Martínez et al. [2006], who mention that this parameter should be of an approximate value of 0.99598 g/ml.

According to Durán et al. [2012], the % acidity should be less than 0.58, which is related to what was found in this study. Also, Arévalo [2013], points out that the % of acidity and total solids are related, this is due to the maturity of the fruit, the longer it has, these parameters will increase, and it is advisable to use these fruit residues before their ripening stage.

The pH in the treatments evaluated remained between 5.23 and 5.5. Conde [2020], mentions that the pH of a cleaner or degreaser should range between 5 to 9, depending on the use.

The turbidity was analyzed before and after the detachment of the grease according to the hand-washing technique exposed by the WHO [2017], resulting in the T_6 releasing more lubricating grease (2471 NTU). Quiroz [2009] states that this action is because the amount of betaine or any type of surfactant will act more effectively when mixed with another active component of greater quantity; In this case 150 ml of extract and a combination of glycerin at 22% constant.

CONCLUSIONS

The company chosen for the diagnosis of waste generation uses as raw material 6 varieties of fruits, among which the orange stands out, from which 50% of its pulp is used. The frequency of production of the company is weekly and only the orange has a production that lasts approximately 6 months. However, the company does not have treatments for the waste they generate or a correct final disposal. In the formulation of the natural degreaser, it was found that all experimental units practiced can release lubricating grease, however, the treatment with greater effectiveness of detachment with a value of 2 470.91 NTU and a pH of 5.46 was the T_6 comprised by the fusion of additives with 150 ml of extract and 15% betaine. This was confirmed by physicochemical analysis in a spectrophotometer. The parameters of turbidity before and after and fat shedding were not different in terms of the amount of extract, dose of betaine, and the interaction amount of extract/dose of betaine.

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