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Biodiesel Production From Energy Tobacco

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Abstract:

The production of biodiesel from energy tobacco as an alternative fuel source is discussed herein. Biodiesel was obtained through the transesterification of tobacco seed oil using CH₃ONa as an alkaline catalyst. Full factorial design was used in these studies to determine the best operating conditions. The independent variables were oil/methanol molar ratio, temperature and catalyst loading. The tobacco oil was analyzed by gas chromatography, and the fatty acids present in the highest concentrations were linoleic, oleic and palmitic acids, which made up 74.16, 11.80 and 8.62% of the oil, respectively. Some physicochemical characteristics of this oil were determined: acid value 3.7 mg g⁻¹ KOH, iodine value 144.74 g 10⁻² g ⁻¹ I₂ and saponification index 190.66 mg g⁻¹ KOH. Subsequently, biodiesel was produced from this oil with a conversion > 97% using the optimized conditions, which were a temperature of 70 °C, an oil/methanol molar ratio of 1: 4 or 1: 8 and a catalyst loading of 2%.

Keywords: energy tobacco; factorial planning; fatty acids; biodiesel; catalyst; temperature

1. Introduction

Within the context of economic development, sustainability is one of the challenges of industry and agriculture as production processes must be greener. To be in line with the principals of green chemistry, ideally, the products of industry should be biodegradable, be produced from renewable sources, and require a low energy cost. In this sense, processing, transformation and evaluation of alternative vegetable oils is important to seek greater agro-industrial diversification and explore the possibility of producing biofuel from different raw materials. In this way, vegetable oils are an alternative to petroleum oil in compression ignition engines, which were evaluated in the 19th century [1]. The possibility of using agriculturally derived fuels in diesel engines is quite attractive considering the environmental aspects, as these

types of oils are a renewable source of energy, and their development allows a reduction in the dependence on petroleum [2].

The most produced vegetable oils in the world are those of soybean, canola, palm and sunflower. Among these, soybean stands out, and Brazil is one of the major producers of this oil. On the other hand, because the nature and quantity of fatty acids depend on the plant species and the edaphoclimatic conditions in which the plant was cultivated, the raw materials (vegetable oils) are rarely used in industrial processes.

In this sense, the search for regional alternatives that could potentially be used to produce biofuels or in other industrial applications and that do not affect the agricultural production of food is paramount. Tobacco (solanaceae) has enormous socioeconomic importance in this

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context, and even more so in the southern part of Brazil, which is responsible for 98% of the national tobacco production. According to the Association of Tobacco Growers of Brazil (Afubra), in the 2013/2014 harvests, tobacco production for cigarettes reached 735 thousand tons, and 52% was produced in Rio Grande do Sul, 29% in Santa Catarina, and 19% in Paraná. As a result, Brazil is the world's second largest producer of tobacco second only to China [3, 4].

Therefore, energy tobacco is considered a potential new raw material to produce biodiesel because the seeds contain approximately 36-41% oil and do not contain nicotine [5, 6]. The remaining parts of the plants may contain proteins, carbohydrates, raw fiber, and inorganic materials among other materials. The main triacylglycerols are glyceryl trilinoleate and glycerol palmito dioleinate (90%). The major fatty acids are linoleic acid (66-76%), oleic acid (17-27%), palmitic acid (7-10%) and stearic acid (3.1%) [7, 8].

One of the most important features of this strategy is that energy tobacco is not useful for human food consumption. That means that unlike the production of other oil plants such as soybean, sunflower, etc., the production of energy tobacco does not interfere with the price or availability of food. Similarly, energy tobacco can be planted in poor soils, leaving them in better condition for other foods thanks to their easy adaptability to different soils and climates. In addition, the chemical composition of biodiesel is directly related to fuel properties, such as freezing point, stability and viscosity [9]. Usta [10] found that the properties of tobacco biodiesel indicate that it can be used in proportions of 25% (B25) without requiring engine modifications and allow reduced emissions of CO and SO₂. According to Rozina et al. [6] tobacco seed oil is considered a suitable substitute for diesel in the future.

The environmental benefits of tobacco as a fuel should be exploited fully as it represents the potential for the healthy use and future agricultural markets of the crop. In this way, tobacco seeds, an agricultural waste product, could be a renewable and valuable raw material for the production of biodiesel. According to Poltronieri [11] the Solaris variety of tobacco is an important alternative for accessing the environmental benefits of this strategy because it has high seed production and low nicotine content, which can be exploited for the development of new biofuels, such as biokerosene for the aeronautics industry.

Therefore, considering the strength of the tobacco culture and the need for agro-industrial diversification, as well as the diversification of the national energy supply, the objective of this study was to characterize the oil and optimize the production of biodiesel from the seeds of energy tobacco (Solaris) produced as an experimental crop in southern Brazil.

2. Results and Discussion

The extraction and analysis of the oil, as well as the production of biodiesel, were performed using the classical quantitative techniques described by the AOCS, and the results can be observed in Table 1. The volume of biodiesel extracted from the oil was 30%, and then the produced fuel was centrifuged to remove the sludge and whole seeds that passed to the cylinder during the extraction.

2.1 Tobacco oil physico-chemical properties

shows the results of Table 1 the characterization of the tobacco oil. The acid value. which is lower than those reported by Veljkovic et al. [12] and Stanisavljević et al. [13], is an parameter indicative important of the conservation state of the oil. The saponification indices were similar to those previously reported, and they are related to the amount of free fatty acid in a sample, i.e., the lower the saponification index, the higher the molecular weight of the fatty acids.

The peroxide index was zero, meaning the oil did not undergo any oxidation. This is because the oil was crude (had not been processed) and was in very good condition, i.e., it had not been altered through oxidation, hydrolysis, etc.; therefore no toxic compounds such as peroxides, aldehydes, and trans fatty acids were formed [14]. The iodine indices are higher than that reported by Giannelos et al. [7], indicating that the oil of the energy tobacco seed presented a high degree of unsaturated fatty acids.

2.2 Characterization of the oil by gas

chromatography

The oil was characterized by gas chromatography, and the average fatty acid composition of the triglycerides is shown in Table 2.

Table 2 shows the results obtained from the composition of the fatty acids of tobacco oil, and

the values are similar to those reported by [13, 15]. The highest proportions correspond to 74.16% linoleic (18:2) and 11.80% oleic (18:1), which indicate these fatty acids are present in tobacco oil in high concentrations. This is consistent with the previously mentioned iodine index because these fatty acids are the most unsaturated.

Table 1. Physical-chemical indices of oil extracted from tobacco seed.

Indexes	Extracted oil	Ref 1	Ref 2	Ref 3	Rules AOCS
Acidity index (KOH mg g ⁻¹)	3.7	36.6	_	37	Cd 3d-63
Saponification index (KOH mg g ⁻¹)	190.66	192.5	193	193.9	Cd 3-25
Density (20°C) g mL ⁻¹	0.9230	0.9230	0.9175	0.924	To1a-64
lodine content (I ₂ g 1.10 ⁻² g ⁻¹)	144.74	130.2	135	130	Cd1b-87
Unsaponifiable matter (%)	2.2				Ca 6a-40
Peroxide index (meq kg ⁻¹)	0.0				Cd 8-53
Refractive index (25 °C)	1.478			1.4739	Tp 1a-64
Viscosity (25 °C) (mm ² s ⁻¹)	84.77	101.1		93.3	-

Source: Authors; and, ref1- Veljkovic et al. [12]; ref2- Giannelos et al. [7]; ref3-Stanisavljević et al. [13].

The third most prevalent acid was palmitic acid (8.62%) because other types of fatty acids, such as saturated fatty acids, are also components of the tobacco oil. These results agree with those reported by Usta et al. [16] who found that the fatty acids present in the highest proportions were linoleic acid (72.98%), oleic acid (12.14%), and palmitic acid (8.16%). In addition, the concentrations of saturated fatty acids were obtained, but they were present in smaller proportions, which agrees with the results of Ashraful et al. [17]. Recently, Math and Chandrashekhara [18] published a study where these same fatty acids were found to be present, although in different proportions, in tallow, pongamia, rice bran, palm, sunflower, coconut, polanga, jatropha, karanja, neem, colza and soybean, which are the main raw materials for the production of biodiesel.

2.3 Characterization of the oil by infrared spectroscopy

FT-IR was used to identify the major functional groups and bands corresponding to stretches and vibrations in the energy tobacco oil samples. The results of FT-IR analysis of the oil are presented

in Figure 1 and Table 3. A very intense band at 3005 cm^{-1} , characteristic of the asymmetric vibration of a =CH group, and two characteristic bands for the esters derived from the carbonyl group (C=O) at 1748 cm⁻¹ and the CO group at 1240 and 1098 cm⁻¹ can be seen. In addition, a very weak band is present at 1659 cm⁻¹, which corresponds to the presence of the oil starting material.

Table 2. Mean ratio of fatty acids found in tobacco oil samples.

Fatty acids	(%)
Palmitic 16:0	8.62
Stearic 18:0	3.06
Oleic 18:1	11.80
Isomer 18:1	0.70
Linoleic 18:2	74.16
Isomer 18:2	0.03
Isomer 18:3	0.07
Linoleic 18:3	0.74
Arachidonic 20:0	0.19
Others	0.66

Source: Authors



Figure 1. FT-IR spectrum of the tobacco oil. Source: Authors

The spectra of vegetable oils such as soybean were compared to the tobacco oil spectrum, and there were some similarities in the bands in the spectral region. Signals at 1740 cm⁻¹ (ν -C=O), 2851 cm⁻¹ (ν -CH) and at 1027 cm⁻¹ (ν -CO sim, ester) were common to both sets of spectra according to Castilho-Almeida et al. [19].

Table 3. Bands in the infrared region of tobacco oil.

Specification	Signal (cm ⁻¹)
υ=CH asym	3005 F
υ-CH2 sym	2870 F
υ-C-H	2825 F
υ-C=O	1748 F
υ-C=C	1659 d
δ-CH₂	1469 m
δ-CH₃	1360 d
υ-C-O asym (Ester)	1240 d
υ-C-O sym (Ester)	1098 m
<u>δ-CH₂ (Rock)</u>	730 m
Source: Authors	

2.4 Production of biodiesel

Experimental planning for biodiesel production was 2³ factorial, and monitoring with the probe allowed results to be obtained throughout the reaction (Figure 2). The spectra of each measurement (240 measurements) were used to evaluate the behavior of the reaction. Transesterification is a very rapid reaction using basic catalysts. The conversion of methyl esters requires a reaction time of approximately 5-10 min, and then the reaction stabilizes after at most 20-30 min. At equilibrium, di- and monoglycerides can also be found in concentrations of 2 and 4% respectively according to Encinar et al. [20].

Therefore, in situ monitoring with an infrared probe is an important factor in the optimization of biodiesel production because the observations in the optimization of the conversion are faster. According to de Lima et al. [21] *in situ* spectroscopic techniques have been selected for monitoring because they are faster, nondestructive and easy to use in reactions such as transesterification.

In Figure 2, it can be seen that the reaction times were short. Following addition of the catalyst after 7 min, the triglycerides were rapidly converted to methyl esters. The conditions that afforded the best conversions were conditions 7 and 8, which are a temperature, molar ratio and catalyst loading of 70 °C, 1: 4 and 2% and 70 °C, 1: 8 and 2%, respectively.



Figure 2. Behavior of biodiesel during the reaction as determined by the infrared probe in situ measuring the absorbance between 2800 and 650 cm⁻¹. Source: Authors

It was also found that conditions 7 were more efficient for conversion than conditions 8 because in this reaction, the conversion of the triglycerides to methyl esters peaked and then the reaction reached equilibrium after 14 min. This result showed that when the catalyst loading was increased, it was easier to break the triglyceride bonds, thus facilitating the formation of the esters (biodiesel). Another important factor is minimizing the composition of free fatty acids and the water content of the biodiesel, which also favors the formation of the methyl esters.

The results obtained by TLC showed the formation of the products, confirming the efficiency of the total conversion of the triacylglycerols to methyl esters. This technique confirmed the content of free fatty acids was low, and this was verified by the acidity index, which was also low. Although TLC is effective, it is a very simple technique and can only be used qualitatively. Chromate plates showed no indications of excess oil in the experiments conducted under the conditions that gave the highest conversions as shown in Figure 3. M10 to M12 and M20 to M24 correspond to experiments 4, 7 and 8, and the results of those experiments all show excellent conversions. Experiments in which oil residues are observed at the end of the reaction also show high conversions to methyl esters.

High conversion was also accompanied by high yields (Table 4). There was no significant difference (p> 0.05) among the performance results, indicating that performance was independent of the reaction conditions. The yields are influenced by temperature, alcohol content and catalyst loading. It is also understood that during the transfer of the phases and the rotary evaporation of the residual solvent, there were losses that resulted in lower yields.



Figure 3. Analysis of biodiesel using thin-layer chromatography where M1-M9, M10-M18 and M19-27 correspond to the triplicate reactions of the samples of all the biodiesel production reactions; A = energy tobacco oil and M = samples of energy tobacco biodiesel. Source: Authors

The formation of soaps can also contribute to diminished yields according to Verma and Sharma [22]; however, the low free fatty acid contents, according to the curves obtained with monitoring with the infrared probe, and the rapid separation of the phases (biodiesel-glycerol) do not indicate that this was a reason for reduced performance in these experiments.

Low water content was also an important factor in achieving high conversions and yields and indicated which reaction medium was suitable for the formation of soaps or hydrolysis [23, 24]. In the biodiesel samples analyzed, the water contents were found to be very low.

The oils had very low contents of free fatty acids, which facilitated the production of methyl esters and made it faster and more efficient. If the oils had higher contents of free fatty acids, it would have been a major problem since it complicates the separation of the biodiesel from glycerin and reduces yield.

חו	Temp. (°C)	Catalyst (%)	Ratio oil:methanol	Performanc	e (%)	Acidity index	Density
				Average	SD	(mg g⁻¹ KOH)	(g mL⁻¹)
1	60	1	01:04	88.9	4.2	0.11	0.8894
2	60	1	01:08	94.1	7.6	0.07	0.8857
3	60	2	01:04	94.3	2	0.07	0.8845
4	60	2	01:08	83.1	4.1	0.11	0.8847
5	70	1	01:04	85.7	6.7	0.03	0.8891
6	70	1	01:08	84.6	5.8	0.07	0.8859
7	70	2	01:04	85	16.7	0.05	0.8842
8	70	2	01:08	86.6	12.1	0.09	0.8850
9	65	1,5	01:06	86.2	13.6	0.11	0.8844

Table 4. Factorial Planning and Results of Biodiesel Production Experiments.

Source: Authors

These experiments were monitored by infrared spectroscopy; however, it was observed that the reactions with higher conversions were not always the ones with the highest yields. From the chromatograms, it was possible to obtain the total peak areas of the components of each experiment conducted in triplicate because they started from the same amounts of reactants and were subjected to the same dilution and chromatographic analysis conditions.

In Figures 4 and 5, the impact of the independent variables determined in the experiment design for the production of tobacco biodiesel, as well as the interaction between the variables, is interpreted. The results obtained did not indicate significant differences (p> 0.05) when the averages of triplicate experiments were considered. However, considering the different planning experiments, there was a significant difference in in yield based on changes in the catalyst loading, the ratio of methanol to oil and temperature. The Pareto diagram (Figure 4) shows the significance of the effects.



Figure 4. Pareto diagram of the independent variables in biodiesel production from our experimental design considering catalyst concentration (1 and 2%), oil:methanol ratio (1:4 and 1:8) and temperature (60 and 70 °C), centered on 1.5% catalyst, 65 °C and 1:6 oil:methanol. Source: Authors

Significant positive effects were also identified in the interaction between the catalyst loading and the temperature according to Ezekannagha et al. [25]. Increasing the temperature and catalyst loading will not effectively increase the conversions (X1 * X2). It is possible that this effect would have been observed if the temperatures evaluated were lower. The temperature studied were near each other, but the increment of some degrees are responsible by viscosity decreasing and resistances to mass transfer lower [26].

Similar results were observed bv Muthukumaran et al. [27] using temperatures of 40 to 70 °C for the transesterification of the oil of Madhuca indica. These authors did not find a significant interaction effect of the catalyst loading and the proportion of methanol like the effect observed in the present work (X2 * X3). It is also observed that the oil-methanol ratio had a insignificant effect in negative and the experiments performed and the usual is 1:6 oil methanol molar proportion to several oils [28-30]. Sometimes major methanol content is important in heterogeneous catalysis or with high acid feedstock [31, 32].

According to Math and Chandrashekhara [18], in the production of biodiesel from safflower oil, the effect of the interaction between the amount of methanol and the concentration of alkaline catalyst, in this case sodium hydroxide, was not significant. However, they observed the yield increased when there was an increase in the proportion of methanol and rotation speed, and increasing the catalyst loading decreased the yield due to the formation of soap. Using blends of oils for biodiesel production, Gupta et al. [33] observed a positive interaction effect between the alcohol ratio and the reaction temperature; however, in that study, this effect was limited to 50 °C and 1:6 mol (oil-methanol). We did not observe this effect probably because we used higher temperatures.

The 3D representations of the conversions calculated by comparing the total peak area of the esters obtained with the total peak area of the derivatized starting oil are presented in Figure 5. Based on this analysis, it was possible to identify the most suitable conditions for the highest conversion of tobacco oil to biodiesel. The results were found from the means of the areas of the chromatograms obtained from gas chromatography. For the production of biodiesel from tobacco oil, >97% conversion is generally obtained with 2% catalyst. The ratio of methanol to oil in combination with higher temperatures and catalyst loadings improved conversion.



Figure 5. 3D graphs of the conversion of tobacco oil into biodiesel, considering the variables of catalyst loading ((A) 1%; (B) 1.5% and (C) 2%), oil:methanol ratio (1:4 and 1:8) and temperature (60 and 70 °C), centered at 1.5%, 65 °C and 1:6 oil:methanol. Source: Authors

The conditions studied in this article are based on studies carried out by other authors. Temperatures near 60 °C are usually used for the production of biodiesel. According to Verma and Sharma [34], the temperatures that lead to optimum yields are between 50 and 60 °C, however several studies achieved high yields with temperatures close to 80 °C. According to the results of infrared monitoring and thin-layer chromatography, the conversions were high.

2.5 Experimental cost of production

The experimental biodiesel production costs of the extracted tobacco oil in the pilot process are presented in Table 5. The purification of the oil and the inputs for the cleaning of the equipment were not considered in the calculation. Nor was the use of the glycerin since this biodiesel was produced from crude oil. Industrial scale studies are more effective for calculating costs related to various market factors. The feasibility and competitiveness of production chains are related to productivity, technology, the costs of inputs, and other factors [35]. However, on a laboratory scale, it is already possible to observe where the cost is most concentrated. For other crops, the largest fractions of the production cost are mainly related to the raw material and energy consumption [36, 37].

Process for 100 L of biodiesel	Cost (US\$)
Seed production	25.16
Extraction of tobacco oil	15.71
Transesterification	12.20
Total	53.07
Source: Authors	

Source: Authors

3. Material and Methods

3.1 Extraction of oil

To perform the various oil extractions, an extraction plant with a filtration system (PE60-Standard) was used. The system consisted of an extractor (ERT60-Standard) and a rotary dryer (SMR600-Standard).

3.2 Physico-chemical properties of tobacco oil

To determine the physicochemical properties of Solaris tobacco oil and the main quality control parameters required by the ANP, the oil was analyzed according to procedures described by the American Oil Chemists' Society [38]. The acidity index (AOCS Cd 3d-63), saponification index (AOCS Cd 3-25), unsaponifiable matter (AOCS Ca 6a-40), peroxide index (AOCS Cd 8-53), iodine index (AOCS Cd1b- 87), refractive index (AOCS Tp 1a-64) and density (AOCS To1a-64) were determined.

3.3 Production of biodiesel

The production of biodiesel from energy tobacco was studied on a laboratory scale to observe the kinetic parameters of the reaction. Therefore, the reaction was carried out using an infrared probe (Mettler Toledo, ReactIR 15, probe 2800 to 650 cm⁻¹), which allowed monitoring of the reaction at all times under different temperatures, alcohol contents and catalyst loadings. By using the probe, it can be determined if the presence of free fatty acids led to the formation of by-products.

3.3.1 Reaction

The sodium methoxide solution (CH₃ONa) 30% in methanol Rodhia (Brazil) was added to the preheated oil already in the reactor. The reaction proceeded for 1 hour under constant stirring at the desired temperature (60, 65 and 70 °C). After completion of the reaction, the product was left to stand for approximately 20 min, allowing complete separation of the glycerol and biodiesel. The glycerol was removed and would be available for purification. The excess alcohol was removed from the biodiesel by rotating it for approximately 15 min at a temperature of 65°C under constant agitation.

3.3.2 Experimental design

A complete factorial planning was performed with three independent variables using the Chemoface program; temperature, molar ratio of oil to methanol and catalyst loading were analyzed. The dependent variable evaluated was the yield of biodiesel. According to the proposed experimental design, the experiments were performed in triplicate, generating a total of 27 trials.

The intervals evaluated for each variable were 60, 65 and 70°C for the temperature; 1:4, 1:6 and 1:8 for the molar ratio of oil to methanol; and 1.0, 1.5 and 2.0% catalyst loading. These study

intervals were considered to be the most appropriate. In each test, 50 g of the oil was maintained under constant stirring, and the temperature and catalyst loading were selected according to the proposed experimental design. The tested alcohol contents were greater than a stoichiometric ratio to increase the conversions and yields. Which variables were the most important factors for the production of biodiesel from energy tobacco was determined by the statistical test of significance (ANOVA - analysis of variance, with significance level of 0.05).

3.4 Biodiesel and oil characterization

3.4.1 Infrared spectroscopy

The spectra of the tobacco oil were obtained using a Fourier transform infrared spectrometer (Perkin-Elmer, FT-IR/FT-NIR Spectrometer) over the 650-4000 cm⁻¹ range.

3.4.2 Chromatography

A gas chromatograph coupled to a mass spectrometer (GC/MS) (Shimadzu, QP 2010 PLUS) fitted with a ZB5wax column (30 m x 0.25 mm x 0.25 μ m) was used. The triacylglycerols (tobacco oil) were derivatized using BF₃/methanol as the catalyst [39, 40]. The initial column temperature was 70 °C. The column was then heated at 4 °C/min to 240 °C (5 min) and then heated at 5°C/min to 250°C; the injector was at 260 °C; the detector and interface were at 250 and 260 °C, respectively; tests were conducted in 1:10 split injection mode.

Thin-layer chromatography (TLC) analysis using silica gel chromatoplates (Macherey Nagel[®]) using hexane solution (Nuclear[®]): ethyl acetate (Vetec[®]) (9: 1 v/v) as the mobile phase was carried out.

3.5 Cost of production

The cost was calculated based on the production of biodiesel on a pilot scale according to Kaercher et al. [41] for 100 L of transesterification product. The data collection performed in accordance with the was experimental evaluation selected for the laboratory scale tests. The costs of the experimental agricultural production phase of the seeds are calculated for the production of one hectare of crop. The production of 100 L of oil is considered to require 272 kg of seed, which is the harvest from 0.0453 ha, considering 3 harvests per field. We sought to limit consumption of inputs and energy (considering no August of 2017 in the state of Rio Grande do Sul - Brazil with red tariffs, level 1), and we excluded the costs of equipment investments and the associated disadvantages, labor cost for operation of the transesterification plant, and overhead rates.

4. Conclusions

The oil synthesized herein presented characteristics similar to those of other oils that are used in the production of this biofuel, and in the evaluated experimental conditions of the biodiesel production process, the conditions that presented the highest conversion were 70 °C, an oil-methanol molar ratio of 1:4 to 1:8 and 2% catalyst loading. The conversion results were observed by chromatographic and spectroscopic methods, and infrared monitoring showed that transesterification reached its equilibrium in approximately 14 min. It was also possible to conclude that the main variables leading to higher conversions of the oil to biodiesel were temperature and catalyst loading.

Finally, the economic importance of the production of energy tobacco and the possibility of producing biodiesel from this crop is highlighted. Energy tobacco production can enhance agro-industrial diversity in southern Brazil and in other countries that produce tobacco for the manufacture of cigarettes. Tobacco oil does not compete with feed oils and is a suitable option to replace diesel, making it an important alternative feedstock for oleochemistry. Therefore, the production of energy tobacco can bring economic growth.

In this way, the biodiesel industry, which is well established in Brazil and has tax incentives because it is linked to family agriculture, may be the way to convert experimental crops into commercial crops.

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