

CASTOR (RICINUS COMMUNIS L) SEED OIL AS AN ALTERNATIVE FEEDSTOCK FOR THE PRODUCTION OF BIODIESEL

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EXTENDED ABSTRACT

Biodiesel has high potential as a new and renewable energy source in the future, as a substitution fuel for petroleum-derived diesel and can be used in existing diesel engine without modification. Currently, more than 95% of the world biodiesel is produced from edible oil which is easily available on large scale from the agricultural industry. However, continuous and large-scale production of biodiesel from edible oil without proper planning may cause negative impact to the world, such as depletion of food supply leading to economic imbalance.

In this study, the profile of castor seed oil and its production to fatty acid methyl esters via a base-catalyzed transesterification were investigated. The advantage of castor oil over sunflower oil, soy bean oil and corn oil would lie in the oil price. The oil was extracted and was characterized by its physicochemical properties. The experimental results showed that the oil content of castor seeds was remarkably high (40.3%). The major constituent observed by GC analysis was ricinoleic acid (12-hydroxy-*cis*-octadec-9-enoic acid) in which the presence of a hydroxyl group at C-12 imparts several unique chemical and physical properties. Thus, castor oil and its derivatives are completely soluble in alcohols. Regarding the fuel-related properties, the high calorific value and the high cetane number are of advantage along with the low content of sulfur and carbon residue. The disadvantage is that castor oil has a significantly higher viscosity at 40°C (240.12 mm²/s), than other vegetable oils. Furthermore, the intent of this work was to investigate and to determine the influence of the chemical properties of the oil in the transesterification and to examine the quality parameters of the methyl ester according to the European standard EN 14214. The transesterification reaction was carried out by changing different parameters like catalyst amount (0.5, 1, 1.5 and 2%) and temperature (25 and 65°C). In all experiments, a methanol/castor oil molar ratio of 6:1 was used. Similar conversions were obtained when the reaction was carried at temperatures 25 and 65 °C and catalyst amount 1.5%; and more specifically at temperature 25°C the methyl ester concentration was 93.8 wt%, whereas at 65°C it was 94.2 wt %. The property analysis of prepared methyl ester samples showed that they does not fulfill either specific gravity or viscosity European specifications; as it turned out to have higher viscosity measurement, by six times above the specifications outlined as acceptable. However, the biggest viscosity of biodiesel can be corrected through the use of blends, which present diesel norm values. Moreover, it does not contain sulfur, has greater cetane number, which indicates a better quality of ignition, and it has more oxygen, making its combustion more complete.

KEYWORDS: Castor seed oil, Biodiesel, Alkaline transesterification, Fatty acid methyl esters, Biodiesel properties

1. INTRODUCTION

In the past few decades, fossil fuels mainly petroleum, natural gas and coal have been playing an important role as the major energy resources worldwide. However, these energy resources are non-renewable and are projected to be exhausted in the near future. The situation has worsened with the escalating energy consumption worldwide due to rapid population growth and economic development. This has caused the price of crude petroleum to hit a record high of USD (US dollar) 90 per barrel in October 2007 and still rising. Therefore, there is an urgent need to find a new energy resource that is renewable, clean, reliable and yet economically feasible as a substitution to the current fossil fuels. In this context, recently, biodiesel derived from vegetable oil has been shown to be a potential alternative replacing petroleum-derived diesel oil for diesel engine.

Biodiesel is mono alkyl ester derived from oils (plant or animal) which have characteristics similar to petroleum-derived diesel oil. Currently, about 84% the world biodiesel production is met by rapeseed oil [1]. The remaining portion is from sunflower oil (13%), palm oil (1%) and soybean oil and others (2%) [2]. Since more than 95% of the biodiesel is made from edible oil, there are many claims that a lot of problems may arise. By converting edible oils into biodiesel, food resources are actually being converted into automotive fuels. It is believed that large-scale production of biodiesel from edible oils may bring global imbalance to the food supply and demand market. Recently, environmentalists have started to debate on the negative impact of biodiesel production from edible oil on our planet especially deforestation and destruction of ecosystem [3]. They claimed that the expansion of oil crop plantations for biodiesel production on a large scale has caused deforestation in countries such as Malaysia, Indonesia and Brazil since more and more forest has been cleared for plantation purposes. Furthermore, the line between food and fuel economies is blurred as both of the fields are competing for the same oil resources. In other words, biodiesel is competing limited land availability with food industry for plantation of oil crop. Arable land that would otherwise have been used to grow food would instead be used to grow fuel [4]. In fact, this trend is already being observed in certain part of this world. There has been significant expansion in the plantation of oil crops for biodiesel in the past few years in order to fulfill the continuous increasing demand of biodiesel. Although there is continuous increase in the production of vegetable oil; however, the ending stocks of vegetable oils are continuously decreasing due to increasing production of biodiesel. Eventually, with the implementation of biodiesel as a substitute fuel for petroleum-derived diesel oil, this may lead to the depletion of edible-oil supply worldwide.

In order to overcome this devastating phenomenon, research to produce biodiesel by using alternative or greener oil resources such as non-edible oils should be conducted. One such oil would be castor seed oil, which has merely less than 0.15% in the international oil seed trade market. For this reason Oil World, a well-known vegetable oil statistics, only note the production, trade and consumption data in the margins [5]. At present, about 1.3 Mt of castor oil seeds are produced in total per year corresponding to about 550 kt of oil [6]. Since the beginning of the 1970s, castor oil seed production has increased continuously but it is, in some cases, subject to yearly fluctuations of 20%, which are above all the result of storm damage in the main regions of cultivation.

About half of all the castor oil produced in the world is exported. India alone exports 80% of this and therefore largely dominates the market. More than 30 internationally operating wholesalers have joined together in the International Castor Oil Association Inc. (ICOA), which was founded in 1957. At the moment they produce over 90% of the entire castor oil world trade. As a result of fluctuations in production and speculation, the price of castor oil varies considerably. In the past, it was between US\$650 and 1500 t⁻¹ without tax ex Rotterdam. The average price in the past 10 years was about US\$900 t⁻¹ and therefore almost twice the price of rapeseed oil in Germany [7].

2. EXPERIMENTAL PROCEDURE

2.1. Castor seed oil extraction

The collected seeds were extracted in laboratory scale, and the procedure is as follows. The seeds were ground to a fine powder and then dried for 2 h at 100 °C. For the continuous extraction of the oil, the Soxhlet extraction apparatus was employed and hexane was used as solvent in the extraction process. The Soxhlet device temperature was kept at 65–70 °C and the overall process lasted 24 h. At the end of the process, the oil was separated from the organic solvent using a rotary vacuum evaporator, dried at 60 °C and weighed. Yield was calculated on dry weight basis.

2.2. Fatty acid composition of castor oil

Fatty acid composition of castor seed oil was determined using gas chromatography (GC Hewlett Packard 5890 series II, SP2340 column) with a FID detector. Helium was the carrier gas. The sample (castor seed oil, 0.5 µl) was injected into the system at 230 °C injector temperature. The oven temperature was kept at 175 °C for 30 min and then it was gradually increased at 3.0 °C/min up to 220 °C. The fatty acids of castor seed oil were identified and quantified using the external larodan standard 905518.

2.3. Physicochemical parameters of castor seed oil

The castor oil was examined in order to evaluate its use either as a blendstock in automotive or heating diesel fuel or as a suitable raw material for biodiesel production. All properties (density at 15 °C, viscosity at 40 °C, flash point, cloud point, pour point, net calorific value, cetane number, cetane index, sulfur content, carbon residue, ash content, phosphorous content, iodine number, acid value and water content) of the oil were evaluated in accordance with the standard test methods. However, as too low amounts of castor seed oil were available for determination of cetane number using the standard method a calculated cetane number was established according to Goering et al [8]. The average molecular weight of vegetable oil is calculated by $MW = 56.1 \times 1000 \times 3/(SV - AV)$, where AV (m_{KOH}/m_{oil} , mg/g) and SV is the saponification value (m_{KOH}/m_{oil} , mg/g) [9].

2.4. Transesterification process

The transesterification reaction of castor oil was carried out in a 500ml spherical flask, with anhydrous methanol, using potassium hydroxide (KOH) as catalyst. The reaction was carried out by changing different parameters like catalyst amount (0.5, 1, 1.5 and 2%) and temperature (25 and 65°C). In all experiments, a methanol/castor oil molar ratio of 6:1 was used. The procedure followed is described next. The reactor was preheated to 75 °C, to eliminate moisture, and then castor seed oil was added. When the reactor reached the temperature established for the reaction, the methanol and the catalyst were added, in the amounts established for each experiment, and the stirring system was connected, taking this moment as time zero of the reaction. Each mixture was vigorously stirred and refluxed for the required reaction time. When the reaction reached the preset reaction time, heating and stirring were stopped. The products of reaction were allowed to settle overnight producing two distinct liquid phases: crude ester phase at the top and glycerol phase at the bottom. The crude ester phase separated from the bottom glycerol phase was then washed by cold or warm deionized water several times until the washed water became clear. The excess methanol and water in ester phase were then removed (or recycled) by evaporation under vacuum.

The FAMES obtained from the transesterification reaction were analyzed by gas chromatography using a Varian 3400 CX instrument, equipped with a capillary injection

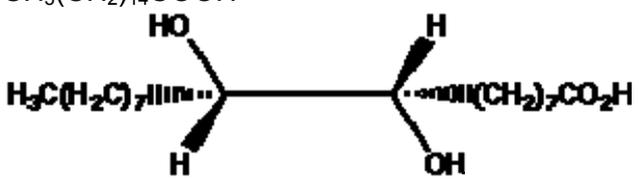
system operating at 240 °C, with a split ratio of 100:1 and sample size of 1 μ L. The capillary apolar column, with 2.2 m in length, 0.32 mm of internal diameter, and 0.1 mm film thickness, was employed, and the column temperature program was initial temperature of 50 °C (1 min), 15 °C/min to 180 °C, 7 °C/min to 230 °C, and 30 °C/min to 245 °C. The detection system was equipped with a flame ionization detector (FID) operating at 250 °C. The carrier gas was high-purity hydrogen. Biodiesel yield, described as the amount of FAMES formed from the transesterification reaction, was quantified in the presence of tricaprylin as an internal standard. Approximately 0.15 g of the products, obtained according with the procedure described in the transesterification experiments section, was weighted in a vial. An amount of 1 mL of tricaprylin solution (0.01 g/100 mL of hexane) was added. This solution was injected in a chromatographic apparatus, and the peak areas of the compounds were integrated.

Each experiment was evaluated twice, and each sample was evaluated as an average of two injections. The biodiesel yield (percent FAMES) was calculated as

$$\text{percent FAMES} = \frac{m_{\text{tricaprylin}} \times A_B \times f_{\text{tricaprylin}}}{A_{\text{tricaprylin}} \times m_s}$$

where $m_{\text{tricaprylin}}$ weight of the internal standard, A_B peak area of FAMES, $f_{\text{tricaprylin}}$ response factor, $A_{\text{tricaprylin}}$ peak area of the internal standard, and m_s weight of the sample.

Table 1: Fatty acid composition of castor seed oil.

Fatty acid	Chemical structure	Wt. %
Ricinoleic	$\text{CH}_3(\text{CH}_2)_5\text{CH}(\text{OH})\text{CH}_2\text{CH}=\text{CH}(\text{CH}_2)_7\text{COOH}$	89.3
Linoleic	$\text{CH}_3(\text{CH}_2)_3(\text{CH}_2\text{CH}=\text{CH})_2(\text{CH}_2)_7\text{COOH}$	4.20
Oleic	$\text{CH}_3(\text{CH}_2)_7\text{CH}=\text{CH}(\text{CH}_2)_7\text{COOH}$	2.30
Stearic	$\text{CH}_3(\text{CH}_2)_{16}\text{COOH}$	1.00
Palmitic	$\text{CH}_3(\text{CH}_2)_{14}\text{COOH}$	1.00
Dihydroxystearic		0.70
Linolenic	$\text{CH}_3(\text{CH}_2\text{CH}=\text{CH})_3(\text{CH}_2)_7\text{COOH}$	0.30
Densipolic	$\text{CH}_3\text{CH}_2\text{CH}=\text{CH}(\text{CH}_2)_2\text{CH}(\text{OH})\text{CH}_2\text{CH}=\text{CH}(\text{CH}_2)_7\text{COOH}$	0.93
Eicosanoic	$\text{CH}_3(\text{CH}_2)_{18}\text{COOH}$	0.30

3. RESULTS AND DISCUSSION

3.1. Castor oil characterization

Table 1, presents the fatty acid profile of the castor oil. Based on the GC analysis 9 types of fatty acids were identified and quantified. These fatty acids vary in carbon chain length and in the number of unsaturated (double) bonds present. The total saturated and unsaturated fatty acid composition of castor oil is 2.68% and 97.44%, accordingly, and the most abundant fatty acid is Ricinoleic acid (89.27 %). This acid has 18 carbon atoms with one hydroxyl in carbon 12, therefore castor oil contains more oxygen than other oils and, therefore, it is more soluble in alcohols during the transesterification reactions.

Regarding the fuel-related properties, the high calorific value (9440 cal/g) and the high cetane number (42) are of advantage along with the low content of sulfur (0.3 ppm) and carbon residue (0.22 %). With an iodine number of less than 90, castor oil is a non-drying oil. Long storage times are unproblematic under airtight conditions. The disadvantage is

that castor oil has a significantly higher viscosity at 40 °C (240.12 mm²/s), than other vegetable oils. The high viscosity of the oil reduces the fuel atomization and increases fuel spray penetration. The bigger fuel spray penetration is considered to be partially responsible for the difficulties with deposits in the engine and thickening of the oil. However, these effects can be eliminated through the transesterification of the oil to form esters.

The two major quality parameters that influence the production process of biodiesel are the FFAs and water content. Several studies showed that the raw oil acid value should be less than 1 mg KOH/g and that all raw materials should be anhydrous (water content <0.3%). If the above requirements are not met it is still possible to produce biodiesel, but the overall yield of the reaction is significantly reduced due to the deactivation of the catalyst and the formation of soaps. In this study, the amount of FFAs measured in the oil was 0.35% while the water content of the oil was rather low (1830 ppm).

Table 2: Physicochemical properties of castor seed oil

Property	Castor seed oil	Test method
Kinematic viscosity (cSt, 40 °C)	240.12	EN ISO 3104
Density (kg/m ³ , 15 °C)	967.3	EN ISO 12185
Water content (mg/kg)	1830	EN ISO 12937
Ash content (% m/m)	0.02	EN ISO 6245
Sulfur content (mg/kg)	<0,04	EN ISO 20846
Flash point (°C)	>260	EN 22719
Cloud point (°C)	+1	EN 23015
Pour point (°C)	-14	ISO 3016
Cetane number	42	-
Gross calorific value (MJ/kg)	9440	ISO 1928
Acid value (mg KOH/g)	0.7	EN 14104
FFA (%)	0.35	-
Saponification value (mg KOH/g)	181.4	AOAC CD3-25
Iodine number (cg I/g oil)	84.4	EN 14111
Average molecular weight (g/mol)	857	-

3.2. Preparation of castor oil biodiesel

Figure 1 and 2 present the influence of catalyst concentration on the conversion of castor oil. Similar conversions were obtained when the reaction was carried at temperatures 25 and 65 °C and catalyst amount 1.5%; and more specifically at temperature 25°C the methyl ester concentration was 93.8 wt%, whereas at 65°C it was 94.2 wt %. In both of these cases, the yield of the castor oil methyl esters turned out to be within the acceptable limit of 97.5 wt %.

The low yields of FAME obtained following base-catalyzed conversion of castor oil may be the result of two factors. First, the high acid value exhibited by castor oil may lead to the neutralization of part of the catalyst present, thus reducing the formation of ethoxides and producing soaps within the reaction medium. Soap formation would not only reduce mass transfer during the reaction but also exacerbate the problem of phase separation at the stage of product recovery (8-10). Second, the hydroxyl group at C-12 of ricinoleic acid may be converted, in basic medium, to an alkoxide. The generation of this anionic species may compete with the formation of methoxide species and consequently reduce the yield of ester. It is important to mention that this alkoxide probably is not effective for the transesterification reaction, using conventional catalysts. (11).

Based on the results presented, one may conclude that the production of biodiesel by ethanolysis of castor oil may be improved through further development and optimization of appropriate catalytic systems and processes. Strategies involving acid catalysis might

provide a promising solution to this problem since it has recently been demonstrated that on an industrial scale acid-catalyzed production of biodiesel can compete economically with base-catalyzed processes [12]. Moreover, acid-catalyzed transesterification reactions exhibit an important advantage in that the performance of the acid catalyst is not strongly affected by the presence of FFA in the oil. In fact, acid catalysts can simultaneously catalyze both esterification and transesterification [13,14].

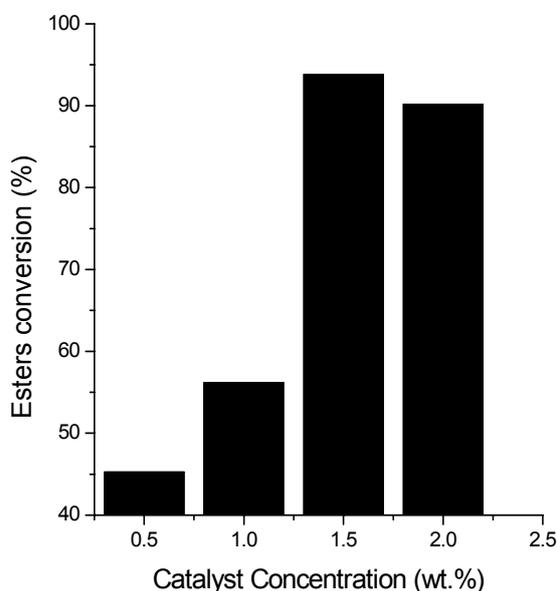


Figure 1. Influence of the mass ratio of KOH to oil on methyl esters yield. Methanol/oil molar ratio, 6:1; reaction temperature, 25 °C; reaction time 2 h.

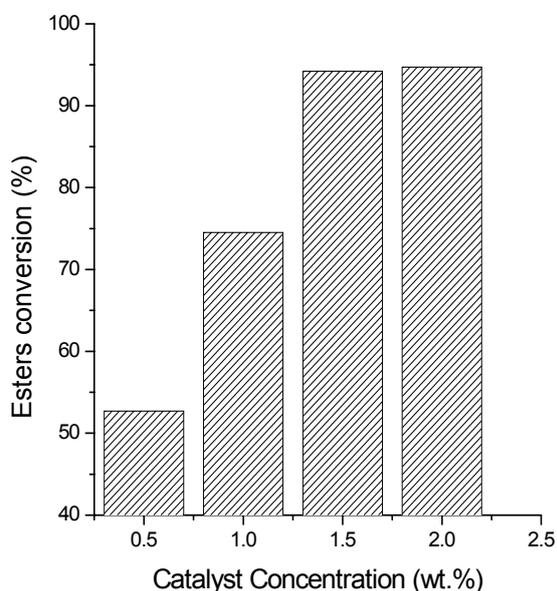


Figure 2. Influence of the mass ratio of KOH to oil on methyl esters yield. Methanol/oil molar ratio, 6:1; reaction temperature, 65 °C; reaction time 1 h.

3.3. Castor oil biodiesel characterization

Property analysis of prepared methyl ester samples was done, in order to examine their quality parameters according to the European standard EN 14214. The fuel properties of castor oil methyl esters, along with the test methods adopted for the fuel property analysis, are given in Table 2.

The standard for biodiesel states that the fuel should have a density between 860 and 900 kgm⁻¹. Density is an important parameter for diesel fuel injection systems. The results obtained showed that the produced methyl esters do not fulfill specific gravity European specifications.

The two samples of biodiesel obtained from the castor oil present a lower viscosity than castor oil owing to the transesterification process, but they still present a high viscosity when compared with the type D mineral diesel oil; this drawback can be corrected by means of blends of biodiesel/mineral diesel at different proportions. The mineral diesel oil presented a Newtonian behavior, a linear relationship between shear stress and shear rate, leading to a viscosity independent of the shear stress. The castor oil biodiesel samples present viscosity higher than mineral diesel oil due to the presence amounts of triglycerides in small amounts.

The iodine number is an index of the number of double bonds in biodiesel, and therefore is a parameter that quantifies the degree of unsaturation of biodiesel. This property greatly influences oxidation stability and the polymerization of glycerides. This can lead to the formation of deposits formed in diesel engines injectors. The iodine number is directly correlated to biodiesel viscosity, cetane number and cold flow characteristics (cold filter plugging point) [15]. At a high saturation, the cetane number is improved while poor low-

temperature qualities may be observed. In this work, the iodine number of the produced methyl esters was 84, below the specification maximum limit.

Table 3: Properties of cotton seed oil methyl esters.

Property	Castor oil biodiesel at 25 °C	Castor oil biodiesel at 65 °C	EN 14214 limits	Test method
Density (kg/m ³ . 15 °C)	914.3	924.4	860-900	EN ISO 3104
Kinematic viscosity (cSt. 40 °C)	13.87	13.34	3.50–5.00	EN ISO 3104
Flash point (°C)	165	167	120 min	EN 22719
Sulfur content (mg/kg)	0.3	0.3	10 max	EN ISO 20846
Cloud point (°C)	-7	-5	-	EN 23015
Pour point (°C)	-27	-30	-	ISO 3016
CFPP (°C)	-13	-16	+5 max	EN 116
Oxid. stability at 110 °C (h)	15.7	19.4	6 h min	EN 14112
Iodine value (g I ₂ /100 g)	84.4	84.3	120 max	EN 14111
Ash content (% m/m)	0.008	0.013	0.02 max	EN ISO 6245
Water content (mg/kg)	154	120	500 max	EN ISO 12937
Carbon residue (% m/m)	0.0164	<0.001	0.3 max	EN ISO 10370
Gross calorific value (MJ/kg)	39.5	39.4	-	ISO 1928
Acid value (mg KOH/g)	0.15	0.13	0.5 max	EN 14104
Phosphorus content (mg/kg)	0.4	0.4	10 max	EN 14107
Potassium content (mg/kg)	0.07	0.09	5 max	EN 14109

Fuel contaminated with water can cause engine corrosion or may cause a reversion of fatty acid methyl esters to fatty acids, which can lead to filter plugging [16]. Therefore, EN 14214 imposed a maximum content of 500 mg/kg of water in biodiesel.

In this study, the produced methyl esters were routinely dried over anhydrous sodium sulfate. This technique was proved efficient enough and the water content of castor oil methyl esters was below the specification limits.

Sulfur and phosphorus content in biodiesel is sourced mainly from the crops and animal fats used as feedstock. Most virgin or used vegetable oil and animal fat-based biodiesel have very low levels of sulfur content. However, methyl esters obtained from used frying oils have detected high levels of sulfur, which is suspected to originate from food cooked in the oil. Combustion of fuel containing sulfur causes emissions of sulfur oxides, particulate matter and can also lead to poisoning of post-treatment devices. On the other hand, the phosphorus content in biodiesel is generally low. However, specifying this parameter is important for engine operability. The present results agree well with the specification limits of sulfur and phosphorus content [17,18].

Alkaline metals (Na+K) result from the metal catalyst used in the transesterification. Because the presence of high amounts of alkaline metals in biodiesel may be linked to ash formation in the combustion engine, EN 14214 specifies a maximum value of 5 ppm. High content of metals in the ester phase indicates insufficient washing and purification during the production process. The potassium content of the castor oil methyl ester produced at 25°C was 0.07 mg/kg, whereas the biodiesel sample produced at 65°C was 0.09 mg/kg. These two results indicate that the washing procedure used was efficient in catalyst removal.

5. CONCLUSIONS

The aim of this study was to evaluate the castor seed oil as a blendstock in automotive or heating diesel fuel or as a suitable raw material for biodiesel production. The castor oil was extracted and chemically converted via an alkaline transesterification reaction to fatty acid methyl ester. The experimental results are described as follows:

- Castor seeds were found to be rich in oil, with an average yield of approximately 40.3%.
- Castor oil has peculiar characteristics, such as its fatty acid composition (90% of ricinoleic acid). This acid has 18 carbon atoms with one hydroxyl, therefore it contains more oxygen than other oils
- Similar conversions were obtained when the transesterification reaction of castor seed oil was carried at temperatures 25 and 65 °C and the catalyst amount was at 1.5%. In both cases, the yield of the castor oil methyl esters turned out to be within the acceptable limit of 96.5 wt %.
- The produced methyl esters do not fulfill either specific gravity or viscosity European specifications; as they turned out to have higher viscosity measurement, by six times above the specifications outlined as acceptable. However, the biggest viscosity of biodiesel can be corrected through the use of blends, which present diesel norm values.

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