

Alkali-catalysed Laboratory Production and Testing of Biodiesel Fuel from Nigerian Palm Kernel Oil

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ABSTRACT

Global concerns about the depletion of the world's non-renewable energy sources and the associated environmental impact of fossil fuel provided the incentives to seek alternatives to petroleum-based fuels. Nigeria is no exception in the fears for crude oil production going into extinction as recently stated by the Energy Commission of Nigeria. Alternative renewable fuel, found in vegetable oils such as Palm kernel oil (PKO), which abound in Nigerian forest, is characterized with high viscosities thus limiting their applications as fuel. The use of transesterified vegetable oils as fuel has been yielding successful results besides being a domestic, renewable resource that provides environmental benefits with lower emissions. In this work, laboratory scale quantities of ethyl ester of PKO (EEPKO) were produced and characterized as diesel fuel. KOH was selected to catalyze the transesterification process of PKO with ethanol using 100g PKO, 20.0g ethanol, 1.0% KOH at 60°C reaction temperature and 100 minutes reaction time. The process was triplicated and average results evaluated. The biodiesel produced was characterized as alternative diesel fuel through tests for specific gravity, viscosity, cloud point, and pour point following ASTM standard test procedures. The transesterification process yielded 92g PKO biodiesel. At 15.56°C, specific gravity is higher (1.033958 times) than that of commercial grade petroleum diesel (D2). At 40°C, EEPKO had 85.06% reduction of viscosity over its raw vegetable oil, thus enhancing its fluidity in diesel engine. Higher pour point (2°C) and cloud point (6°C) were obtained for EEPKO compared to -12°C, and -16°C respectively obtained for D2. Generally, results obtained were found to be in good agreement with previous works and within limits set by a number of International Standards for biodiesel. These findings will find useful applications in energy sector of the Nigeria economy.

Keywords: Energy, alkali catalyst, ethanol, palm kernel oil, biodiesel, renewable fuel, Nigeria

1. INTRODUCTION

The fuel and energy crises of the late 1970's and early 1980's as well as accompanying concerns about the depletion of the world's non-renewable resources provided the incentives to seek alternatives to conventional, petroleum-based fuels (Peterson, 1986; Al Widyan and Al Shyouk, 2002; Bernado *et al.*, 2003). Globally at present, the most widely used fuels are gasoline and diesel, both coming from fossil fuel. Besides the fear for depletion of fossil fuel, due largely to

its non-renewable status, use of gasoline is associated with environmental problems. Aside from being extremely flammable, it contributes to increase hazardous emissions (Munack *et al.*, 2000). On the other hand, diesel has higher carbon numbers and it is widely known that it contributes in emissions of high particulate matters, high sulphur dioxide and high poly aromatic hydrocarbons. The need to consider renewable sources of fuel, with acceptable environmentally friendliness, to meet the ever increasing global energy demands can therefore not be over emphasized.

In Nigeria, the Energy Commission (ECN) recently (2005) reported that Nigeria's fossil-led economy is under severe pressure. In decades to come, the sun will slowly but certainly set on crude oil production. Today, large hydropower plants are increasingly threatened by a shrinking River Niger, shaking the security of electricity supplies (ECN, 2005). Nigeria currently imports about 80% of its petroleum requirements and has been hit hard by rapidly increasing cost and uncertainty. Unfortunately, in Niger Delta region, the centre for oil extraction, severe environmental impacts have been ignored in the country's haste to develop the oil industry. This has generated militancy from the local people (Ijaw) making successful oil prospecting a nearly impossible task for the multinational companies in Nigeria. As a result, the cost of extracting the reserves will go on increasing in Nigeria. Thus there is an urgent need to find alternative renewable forms of energy before mineral oil supplies run dry. Hence, in the medium (2008-2015) and long term (2016-2025), Nigeria envisions an energy transition from crude oil to renewable energy (ECN, 2005).

Interestingly, Nigeria is endowed with significant renewable energy resources including large and small hydroelectric power resources, solar energy, biomass, wind and potentials for hydrogen utilization; and development of geothermal and ocean energy. Presented in Table 1 are the estimated renewable energy resources in Nigeria, excluding potential hydrogen, ocean and geothermal energy.

Table 1: Nigeria's renewable energy resources

Energy Source	Capacity
Hydropower, large scale	10,000MW
Hydropower, small scale	734MW
Fuel wood	13,071,464 hectares (forest land)
Animal waste	61 million tones/yr
Crop Residue	83 million tones/yr
Solar Radiation	3.5 – 7.0 kW/m ² -day
Wind	2-4 m/s (annual average)

Source: [ECN \(2005\)](#)

Vegetable oil as an alternative fuel has been under study in certain part of the world far back as 1979 (Peterson *et al.*, 1990). Since then researchers have considered the use of vegetable oil such as Canola, rapeseed, soybean oil, rapeseed oil as diesel fuel substitute. High viscosities of these oils have however been reported to have limited their applications. Silvio *et al.* (2002) were reported to have stated that high viscosities of pure vegetable oils reduces the fuel atomization and increase fuel spray penetration, which would be responsible for high engine deposits and

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thickening of lubricating oil. The use of chemically altered or transesterified vegetable oil does not require modification in engine or injection system or fuel lines and is directly possible in diesel engine (Chitra *et al.*, 2005). Biodiesel is a name applied to fuels manufactured by the transesterification of renewable oils, fats and fatty acids. It has gained importance in the last years in more than 21 countries leading to commercial projects in Austria, the Czech Republic, France, Germany, Italy, Malaysia, Nicaragua, Sweden and the USA (Best, 2006).

The use of biodiesel as fuel continues to attract attention because of the successful results obtained in its applications and the intent to utilize a domestic, renewable resource that provides environmental benefits with lower emissions compared to the conventional petroleum-based fuel. Oil palm (*Elaeis guineensis*) is an oil-bearing crop common in Nigeria. It has been found to be an appropriate renewable alternative source of biodiesel (Abigor *et al.*, 2000). The kernel of this crop is used to extract oil with about 50% palm kernel oil recovery. It contains 3.8 mt oil, found mostly in Malaysia, Indonesia and Africa (Jekayinfa, 2006). While considerable works have appeared in print on biodiesel production from vegetable oils, limited studies were found for vegetable oils common in Nigeria. Abigor *et al.* (2000) produced fatty acids esters from two Nigerian lauric oils, palm kernel oil and coconut oil, by transesterification of the oils with different alcohols using PS30 lipase as a catalyst. In their lipase-catalysed conversion of PKO to alkyl esters (biodiesel), ethanol gave the highest conversion of 72%.

This work therefore seeks to produce test quantities of ethyl ester of palm kernel oil (EEPKO) via transesterification of the PKO with ethanol using alkali-catalyst, KOH. The choice of potassium hydroxide amongst other alkali catalyst was made as a result of possible generation of potash fertilizer as a by-product alongside glycerine. This work further seeks to characterize the EEPKO biodiesel produced as alternative diesel fuel through tests for specific gravity, viscosity, cloud point, pour point and flash point. The results are expected to contribute to baseline data needed for future replacement of conventional diesel with renewable biodiesel. Specifically, these findings will find useful applications in energy sector of the Nigeria economy.

2. MATERIALS AND METHODS

Palm kernel oil like any other vegetable oils and animal fats are triglycerides, containing glycerine. The biodiesel process turns the oils into esters, separating out the glycerine. The glycerine sinks to the bottom and the biodiesel floats on top and can be syphoned off. The process is called transesterification, which substitutes alcohol for the glycerine in a chemical reaction, using a catalyst (Fig.1).

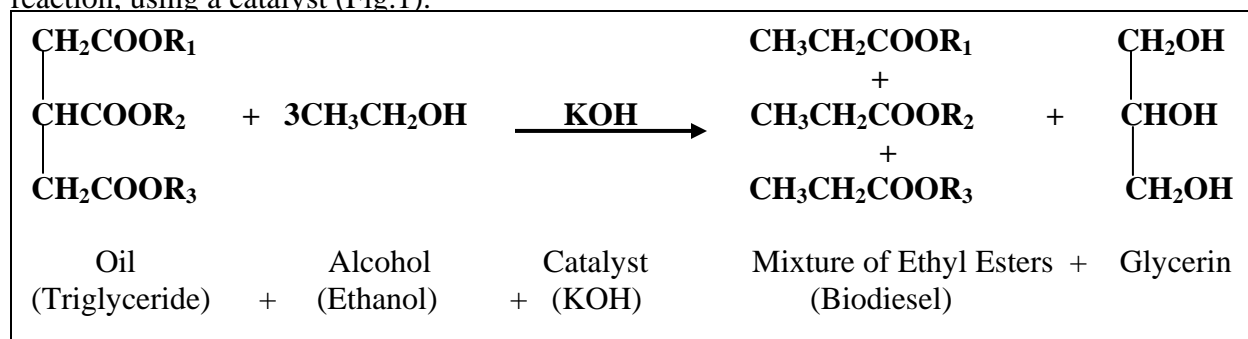


Fig. 1: Transesterification chemistry for ethyl ester (biodiesel) production

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2.1 Experimental Materials

The major feedstock used in this work is Palm kernel oil, locally produced in Nigeria. It was purchased at the local market in Ogiyo, Ogun State, Nigeria. By the stoichiometric equation of the process, 1 mol of PKO is required to react with 3 moles of ethanol to produce 3 moles of the biodiesel and 1 mole of glycerol (Kavitha, 2003). 100g PKO was used for the transesterification process. Reaction temperature for the process must be below the boiling point of alcohol used (Van Gerpen, 2004). The Ethanol used, manufactured by Aldrich Chemicals Co. Ltd, England has a boiling point of 78^oC; therefore, a reaction temperature of 60^oC was selected. Different researchers have reported different reaction times for transesterification process as well as the entire biodiesel production process. The reported reaction time ranges from less than 30 minutes to more than 2 hours (Chitra *et al.*, 2005). Reaction time of 100 minutes was therefore selected. Most researchers have used 0.1 to 1.2 % (by weight of oil) of catalyst for biodiesel production (Alacantara *et al.*, 2000; Ma *et al.*, 1999, Chitra *et al.*, 2005). 1.0% **KOH** (by weight of PKO) concentration was therefore selected while 20% ethanol was used. KOH used was manufactured by Aldrich Chemicals Co. Ltd, England.

The blender used was a Dry and Wet mill Blender 462; product of Nakai, Japan. It has a clear glass 1,250 capacity containers with stainless steel cutting blades. Other materials used include scales, measuring beakers, translucent white plastic container with bung and screw-on cap, funnels, 2-litre PET bottles, duct tape and thermometer.

2.2 Experimental Procedures

2.2.1 Potassium Ethoxide Production

20.0g of ethanol was measured and poured into a plastic container a funnel and the lid of the ethanol container was tightly replaced. 1.0g of KOH was carefully added to the plastic container via a second funnel. The bung and the screw on the cap were replaced tightly. The container was shook a few (about six) times by swirling round thoroughly for about 2 minutes until the KOH completely dissolved in the ethanol, forming potassium ethoxide.

2.2.2 Transesterification Reaction

100.0g of PKO was measured out in a beaker, pre-heated to the required temperature (60^oC) and poured into the blender. With the blender still switched off, the prepared potassium ethoxide from the plastic container was carefully poured into the PKO. The blender lid was secured tightly and switch on. The mixture was left to blend for the 100minutes at moderate speed before the blender was switched off.

2.2.3 Settling

The mixture was poured from the blender into a 2-litre PET bottle for settling and the lid was screwed on tightly. The reaction mixture was allowed to stand overnight while phase separation occurred by gravity settling into (an expected) clear, golden / pale liquid biodiesel on the top with

the light brown glycerol at the bottom of the bottle. The next day, the PKO biodiesel/ester at the top was carefully decanted into a PET bottle leaving the glycerol at the base.

2.2.4 Washing

For washing, two 2-litre PET bottles were used in succession, with half a litre of tap water added for each of the four washes carried out. A small 2mm hole was pierced in the bottom corner of each of the two bottles and the hole covered securely with duct tape. The biodiesel was poured into one of the wash bottles. Half-litre of fresh water was added and the cap screwed on tightly. The bottle was turned on its side and rolled about until oil and water were well mixed and homogenous. After washing and settling, the water was drained off from the bottom of the bottle by removing the duct tape from the hole. The hole was blocked again when it reached the biodiesel. The biodiesel was transferred to the second wash bottle; fresh water was added and washed again. The first bottle was cleaned and the duct tape replaced. The process was repeated four times.

2.3 Testing for some Physical Properties of the EEPKO

The pure biodiesel obtained through the above procedure gave the ester yield, measured on weight basis. The experiment was replicated three times and average experimental parameters recorded. ASTM standard fuel characterization was subsequently carried out on the EEPKO biodiesel. Specific gravity (relative density) and viscosity measurements of the ethyl ester produced were made following ASTM standards D1298 and D445 respectively. The biodiesel was analyzed for pour, cloud and flash points following ASTM standards D97, D25100-8 and D-56 respectively. Detailed procedures for these tests have been presented elsewhere (Alamu, 2007).

To enable comparison of the biodiesel produced with petroleum based (fossil) diesel, similar fuel characterization tests were conducted for Philips low sulphur diesel reference fuel (No. 2 Diesel) purchased at TOTAL [fuel station](#), Ifo, Ogun State, Nigeria.

3. RESULTS AND DISCUSSION

For the alkali-catalysed transesterification experiment conducted using the stated reaction parameters, the experiment was triplicated and average experimental results evaluated. Results obtained are as presented in Table 2.

The ethyl ester of palm kernel oil (EEPKO) biodiesel prepared using 100g PKO, 20.0g ethanol, 1.0% [KOH](#) (by weight of PKO) at 60°C and 100 minutes reaction time yielded 92g PKO biodiesel and 26.26 glycerol, while 2.8g of the total reacting masses could not be accounted for. These losses are expected to be some un-reacted alcohol, residual catalyst and emulsion removed during the washing stage of the production process. The results stated are averages of three different experimental runs. Detailed results for each of the experimental runs are as presented (Table 2).

Table 2: PKO-ethanol-KOH transesterification results

Experimental Conditions	1 st Run	2 nd Run	3 rd Run	Average
Reaction temperature (°C)	60	60	60	60
Reaction time (min.)	100	100	100	100
Palm kernel oil (PKO) quantity (g)	100	100	100	100
Ethanol quantity (g)	20.00	20.00	20.00	20.00
KOH (catalyst) concentration* (g)	1.00	1.00	1.00	1.00
PKO biodiesel obtained (g)	90.30	91.90	93.80	92.00
Glycerol obtained (g)	26.50	26.00	26.10	26.20
Losses (g)	4.20	3.10	1.10	2.80
PKO biodiesel yield (%)	90.30	91.90	93.80	92.00

*by weight of 100g PKO

With a biodiesel yield of 92%, it also shows that the combinations of experimental parameters used are within reasonable ranges around which the parameters could be varied to achieve an optimum combination of operating conditions for the PKO biodiesel production.

In characterizing the PKO biodiesel produced as alternative diesel fuel, The PKO biodiesel and the petroleum diesel, used as control, were analyzed for specific gravity at 60°F, viscosity at 40°C, pour point, cloud point and flash point. Results obtained are presented in Table 3.

Table 3: Measured fuel properties

Fuel characteristics (Properties / Parameters)	Values	
	(PKO biodiesel)	(Petroleum diesel)
Viscosity (at 40°C) (mm ² /s)	4.839	2.847
Specific gravity (at 60°F/60°F)	0.883	0.853
Pour point (°C)	2	-16
Cloud point (°C)	6	-12
Flash point (°C)	167	74

3.1 Specific Gravity

Specific gravity has been described as one of the most basic and most important properties of fuel because some important performance indicators such as cetane number and heating values are correlated with it (Tat and Van Gerpen, 2000; Ajav and Akingbehin, 2002). It has also been reported to be connected with fuel storage and transportation (Yuan *et al.*, 2004).

Results presented in Table 3 revealed that the specific gravity recorded for the PKO biodiesel is higher than the values obtained for the petroleum diesel. This is in agreement with earlier observations made by several authors (Peterson *et al.*, 1990; Graboski and McCormic, 1998; Yuan *et al.*, 2004). From 10°C to 60°C, specific gravity values of PKO biodiesel have been reported to be 1.033413 to 1.035419 times that of fossil diesel (Alamu, 2007). At the reference

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temperature ($60^{\circ}\text{F} = 15.56^{\circ}\text{C}$), where the specific gravity value obtained for PKO biodiesel and petroleum diesel are respectively 0.883 and 0.854 (Table 3), the specific gravity of PKO biodiesel is 1.033958 times that of petroleum diesel.

The value of specific gravity obtained for PKO biodiesel (0.883) fall within the limit specified for biodiesel fuels (0.87 – 0.90) by the BIS Standard (Table 4). Specific gravity value for the control fuel sample (0.854) also fall within the range specified for diesel fuel (0.81 – 0.86) by the same Standard.

Table 4: BIS standards for biodiesel and diesel fuel

Properties	BIS Standard	
	Diesel fuel	Biodiesel fuel
Specific gravity	0.81 – 0.86	0.87 – 0.90
Viscosity	2.0 – 5.0	3.5 – 5.0

source: Chitra *et al.* (2005)

At 15.56°C , the specific gravity obtained for the PKO biodiesel falls within the limit specified by various international standards. Specific gravity ranges specified by EN14214 (Europe), ONC1191 (Austria), CSN656507 (Czech Republic), Journal Officiel (France), DINV51606 (Germany), UNI10635 (Italy) and SS155436 (Sweden) standards are (0.86-0.9), (0.85-0.89), (0.87-0.89), (0.87–0.90), (0.875-0.9), (0.86-0.90) and (0.87-0.90) respectively for biodiesel fuel (Knothe and Steidly, 2005; Chitra *et al.*, 2005) From Table 3, the specific gravity of 0.884 at 15.56°C obtained for the PKO biodiesel is in very good agreement with all the above standards as the value falls within the different ranges recommended.

3.2 Viscosity

Viscosity, the measurement of the internal flow resistance of a liquid, constitutes an intrinsic property of vegetable oils. It is of remarkable influence in the mechanism of atomization of the fuel spray. Results obtained for viscosity measurement fall within the range of values acceptable for alternative diesel fuel. These results also agree closely with a number of earlier works on other oil crops as well as series of Alcohol-Diesel blends.

From the result presented in Table 3, PKO biodiesel has higher viscosity than conventional diesel fuel, in agreement with observations of Peterson *et al.* (1995), Graboski and McCormic (1998), Yuan *et al.* (2004), Knothe and Steidley (2005), Lebedevas and Vaicekauskas (2006) amongst several other authors. From 10°C to 60°C , viscosities of PKO biodiesel are 1.684 to 1.712 times that of petroleum diesel (Alamu, 2007). Similar conclusion was drawn by Peterson *et al.* (1995) for eight biodiesel fuels from vegetable oil including rapeseed methyl ester, rapeseed ethyl ester, canola methyl ester, canola ethyl ester, beef tallow methyl ester, soybean methyl ester, soybean ethyl ester and midwest biofuel methyl soyate. Viscosities for these biodiesel fuels were reported to be 1.3 to 2.1 that of low sulphur grade conventional diesel (No.2 Diesel).

The 40°C, used by Van Gerpen *et al.* (2004) as reference point for comparison of values of kinematic viscosity is the parameter required by biodiesel and petroleum diesel standards (Knothe and Seidley, 2005; Lebedevas and Vaicekauskas, 2006). At this temperature, the PKO biodiesel has viscosity of 4.839 mm²/s, which almost doubles the 2.847 mm²/s obtained for the reference conventional petroleum diesel used (Table 3). This is in close agreement with the findings of Peterson *et al.* (1995), Van Gerpen *et al.* (2004) and more recently, Lebedevas and Vaicekauskas (2006).

From the results obtained it can be seen that the esterification of PKO (a vegetable oil) produced a marked decrease in values of viscosity from the reported range of 30 to 50 mm²/s for vegetable oils at 40°C (Brevad biodiesel.org, visited 2007) to 4.839 for PKO biodiesel at the same temperature. Abigor *et al.* (2000) specifically indicated that Nigerian palm kernel oil has a viscosity of 32.40 mm²/s. With viscosity value of 4.839 mm²/s obtained for the PKO biodiesel produced in this work, it implies that a reduction of 85.06% viscosity has been achieved in the PKO fuel. Also, the reduction in viscosity achieved in this study is 15.06% greater than that achieved by Abigor *et al.* (70%), where PKO was transesterified with ethanol using lipase catalyst. This appreciable reduction in viscosity, no doubt, will enhance the fluidity of this alkali catalysed ethyl ester alternative fuel in diesel engine.

It is also observed that the viscosity of PKO biodiesel (4.839 mm²/s) fall within the range prescribed by ASTM D6751 standard (1.9 – 6.0) mm²/s for biodiesel. Also, the viscosity of Petroleum diesel (2.847 mm²/s) fall within the range prescribed by ASTM D975 (1.9-4.1) mm²/s for conventional diesel fuel (Knothe and Steidly, 2005; Chitra *et al.*, 2005). Similar agreement is observed with the BIS standard in Table 4. This implies that the PKO biodiesel satisfies the fluidity requirement of an alternative biodiesel.

3.3 Pour Point

Pour point is the temperature at which wax becomes visible when the fuel is cooled. From Table 3, it is observed that the pour point obtained for the PKO biodiesel (2°C) is higher than that (-16°C) obtained for the conventional petroleum diesel. Aside the general report that biodiesel has higher pour point than petroleum diesel (Alberta Research Council, 2006; Graboski and McCormic, 1998), this result is consistent with earlier findings on such biodiesel fuel like ethyl and methyl esters of rapeseed, canola, beef tallow, soybean and midwest biofuels methyl soyate (Peterson *et al.*, 1990); ethyl ester of frying oil (Graboski and McCormic, 1998); methyl esters of palm kernel oil and coconut oil (Abigor *et al.*, 2000) as well as butyl ester of soybean (Schwab, 1987) amongst others.

The PKO biodiesel Pour point obtained in this work is also 6°C less than that obtained by Abigor *et al.* (2000) for similar feedstock (PKO) but which was transesterified with lipase catalyst.

Pour point has been described as an important parameter for low temperature operation of a fuel. It is the temperature at which the amount of wax out of solution is sufficient to gel the fuel, thus it is the lowest temperature at which the fuel can flow.

3.4 Cloud Point

The cloud point is another important parameter for low temperature operation of a fuel. It is the temperature at which solidification of heavier components of the PKO biodiesel resulting in a cloud of crystals within the body of the biodiesel first appeared. The value obtained is within a comparable range with previous results.

It is observed from Table 3, that the cloud point obtained for the PKO biodiesel (6°C) is higher than that (-12°C) obtained for the conventional petroleum diesel. This agrees perfectly with the general report that biodiesel has higher cloud point than petroleum diesel (Alberta Research Council, 2006; Graboski and McCormic, 1998). The cloud point obtained here is also consistent with earlier findings on such biodiesel fuel like ethyl and methyl esters of rapeseed, canola, beef tallow, soybean and midwest biofuels methyl soyate (Peterson *et al.*, 1990); ethyl ester of frying oil (Graboski and McCormic, 1998); butyl ester of soybean (Schwab, 1987) as well as methyl esters of palm kernel oil and coconut oil (Abigor *et al.*, 2000).

From Table 3, the cloud point obtained for the PKO biodiesel is 18°C higher than the cloud point for the petroleum diesel fuel.

4. CONCLUSIONS

From the laboratory scale production and characterization of ethyl ester of PKO (biodiesel) studied, the following conclusions can be drawn:

- The transesterification process carried out using 100g PKO, 20.0g ethanol, 1.0% KOH (by weight of PKO) at 60°C reaction temperature and 100 minutes reaction time yielded 92g PKO biodiesel.
- At 60°F or 15.56°C reference temperature, specific gravity of PKO biodiesel is higher (1.033958 times) than that of fossil diesel.
- At 40°C (104°F) reference point, the PKO biodiesel had 85.06% reduction of viscosity over its raw vegetable oil, thus enhancing its fluidity in diesel engine.
- Higher pour point (2°C) was obtained for PKO biodiesel compared to conventional petroleum based diesel (-16°C).
- Cloud point obtained for PKO biodiesel (6°C) is higher than that (-12°C) obtained for petroleum diesel.
- The limited fuel characterization carried out demonstrated that the ethyl ester of PKO biodiesel produced can successfully fuel a diesel engine, through the level of agreement between the results obtained and previous works.

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