

Evaluation of Mineral and Chemo-physical Compositions of Sandbox seed Oil

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Abstract: - Mineral and chemo-physical compositions of oil from sandbox seed were evaluated to establish their quality and utility as a base material for domestic and industrial applications. Mineral composition of the oil was obtained using atomic absorption spectroscopy while the fatty acid composition of the oil was analyzed using gas chromatography and flame ionization detector (GC-FID). Analyses of the mineral composition revealed that the oil is very rich in magnesium. The fatty acid composition of the oil revealed that oleic acid and stearic acid are the most abundant unsaturated and saturated fatty acids respectively. The moisture content was 0.39 %, density was 0.90 g/cm³, specific gravity was 0.93, saponification value was 210.00 mgKOH/g, iodine value was 177.66 gI₂/100g, acid value was 1.68 mgKOH/g, peroxide value was 0.87 mg/100g, fatty acid value was 8.42 mgKOH/g, viscosity at 28°C was 49.20 cSt, cloud point was 11°C, pour point was -3°C, flash point was 249°C while fire point was 252°C. The oil has a high saponification and iodine values with low acid and peroxide values. These show that the oil has great potential as promising oil and it will be a good source for a variety of industrial applications.

Key-Words: - Chemo-physical, Mineral, Non-edible, Oil, Sandbox, Underutilized

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1 Introduction

Sandbox (*Hura crepitans*) is an evergreen tree of the spurge family (Euphorbaiceae) usually planted in the cities and villages of Nigeria mostly for shade according to Oniya et al. (2017)[1]. It is also known as “Dynamite tree” due to the explosive noise of the ripe fruits when split into segments. The tree according to Adewuyi et al. (2014)[2] has large ovate leaves which grow to 2 ft wide with many dark and pointed spines, smooth brown bark and spreading branches. It produced pumpkin shaped seed pods which are usually green when fresh and brown when dried. The seed, as described by Obidiegwu et al. (2016) [3] and shown in Fig.1, is a large capsule with explosive dehiscence.

The seeds which have been reported to contain oil (about 53.61%) are enclosed in a hard protective coat which usually and suddenly splashes open and scatters when the seeds are ripped and well dried [4]. The relatively high oil content (53.61% (w/w) of the seed makes the seed a good source of oil, making it economically viable, though the seed is discarded as waste with no market value at present. This also

makes it suitable for industrial applications, as any oilseeds that can produce oil up to 30% are regarded as suitable. Sandbox seed oil is a highly neglected oil available at little cost of production. The oil, according to Giwa et al. (2016)[5], has no specific use and no commercial value in Nigeria presently, as the seeds are discarded as waste.



Fig. 1: Sandbox seeds

Adewuyi (2014)[2] reported sandbox oil as a promising alternative oil because it is environmentally friendly, renewable, cheap, and easily manageable. This work evaluated the mineral and chemo-physical compositions of oil from the sandbox (*Hura crepitans*) seeds. These compositions give a qualitative identification of oils and are very important in the selective application guide for the commercialization and utilization of oil products.

2 Materials and methods

2.1 Materials

Sandbox seeds (*Hura crepitans*) used for this work were collected from Wukari in Taraba State. The seeds were cleaned, manually dehulled and milled using a milling machine. The oil was extracted in a soxhlet extractor using n-hexane as extracting solvent. All the solvents and reagents used for the analyses were of analytical grade and were obtained from the Food Chemistry Laboratory of the Federal University Wukari. The reagents used in this research included: methanol, n-hexane, potassium hydroxide, sodium hydroxide, hydrochloric acid, glacial acetic, potassium iodide, Wij's solution, carbon-tetrachloride, phenolphthalein, isopropyl alcohol.

2.2 Methodology for determination of the mineral and chemo-physical compositions

2.2.1 Mineral composition

The mineral composition of the oil was determined in accordance with AOAC (2000)[6] method using line source PG-990 Atomic Absorption Spectrophotometer (AAS). The instrument is fully automated for flame or graphite furnace analysis. It uses two background correction system, self-reversal method and deuterium lamp method. The burner of the spectrophotometer was ignited by allowing air to mix with acetylene gas from the gas cylinder in good proportion. After the ignition, the spectrophotometer calibration was done immediately with distilled water and standard solution. The sample's aerosol was then aspirated through nebulizer into flame for analysis. Analysis was done for every element of interest at their specific wavelength using hollow cathode lamp of the element under investigation. The result was displayed on the computer and recorded.

2.2.2 Chemo-physical composition

Moisture content

The method of Okolie et al. (2012)[4] was adopted. The moisture content was obtained by using a known weight of the oil sample put into a clean previously weighed beaker and dried in an oven at 105°C for 4 hours. The sample was taken from the oven, cooled in a desiccator for about 30 minutes and then weighed. The procedure was repeated until a constant weight was obtained. The percentage moisture in the seed was then calculated.

Density and specific gravity

Oil of 5 ml was poured into a weighted beaker and weighed. The density was determined from the sample weight by taking the ratio of the weight of the oil to the known volume (5 ml) of the oil. Using the equation below:

$$\text{Density} = \frac{\text{sample mass}}{\text{sample volume}} \quad (1)$$

$$\text{Specific gravity} = \frac{\text{weight of a unit volume of the oil}}{\text{weight of equal volume of water}} \quad (2)$$

Refractive index

Refractive index of the oil was determined using ASTM D1218-01[7] method. Abbes' refractometer was used for the measurement of the refractive index of the oil. The oil was smeared on the lower position of the refractometer, after some adjustment the refractive index of the oil was read directly at room temperature (28°C).

Viscosity

The viscosity was measured in accordance with ASTM D445-15a [8]. This method covers the determination of viscosity using Smart series rotational viscometer TSM 21105. The viscosity was measured at room temperature (28°C). At a start a proper viscometer spindle (3) was chosen. The samples were transferred to a beaker large enough to hold the viscometer spindle. The beaker was placed on a heating mantle set to a desired temperature, while the temperature of the samples was raised. The viscosity was read at the desired temperature. The viscosities of the oils were determined using equation (3) reported by Mohammed (2015)[9]:

$$\text{Viscosity} = \text{reading obtained} \times \frac{\text{factor for the spindle}}{\text{speed}} \quad (3)$$

Saponification value

Saponification value was determined in accordance with ASTM D5558-95 [10] method by weighing 1 gram of the oil into a flask and adding 25 cm³ of 0.1

M alcoholic potassium hydroxide solution into the flask. A reflux condenser was attached and the flask was heated on a water bath for 1 hour with constant shaking. At the end of 1 hour the flask was removed from the water bath and 1cm³ of the 1 percent phenolphthalein indicator was added. It was then titrated with the standard 0.5 M hydrochloric acid.

$$\text{Saponification value} = \frac{(V_2 - V_1) \times N \times 56.1}{\text{weight of oil}} \quad (4)$$

Where: V_1 = sample titre value (cm³)

V_2 = blank titre value (cm³)

N = normality of HCl

56.1 = molecular weight of KOH

Acid value

Acid value of the oil was determined by mixing 25 cm³ diethyl-ether with 25 cm³ ethanol in a conical flask and 1 cm³ of 1 percent phenolphthalein indicator solution was added. The mixture was neutralized with 0.1 M potassium hydroxide solution then 1 gram of the oil was added to the neutralized solvent mixture in accordance with ASTM D 664-18 [11] method. This was then titrated with 0.1 M potassium hydroxide solution. It was then shaken constantly until a pink colour which persists for 115 seconds was obtained.

$$\text{Acid value} = \frac{V \times N \times 56.1}{\text{weight of sample used}} \quad (\text{mgKOH/g})$$

(5)

Where:

V = volume of standard KOH solution used (cm³)

N = normality of HCl

56.1 = molecular weight of KOH

Free fatty acids (FFA)

Free fatty acids were determined in accordance with AOAC (2000)[6] method by weighing 1 gram of the oil samples into a conical flask. This was followed by the adding 10 cm³ of neutralized 95 percent ethanol and Phenolphthalein. This was then titrated with 0.1 M NaOH, with constant shaking until a pink colour persisted for 30 seconds. The percentage free fatty acid was calculated from the equation (6):

$$\text{Free fatty acid (FFA)} = \frac{V \times M \times 2.82 \text{mg}}{\text{sample weight(g)}} \quad (6)$$

Where: V = Volume of NaOH,

M = Molarity of NaOH

2.82 = conversion factor of oleic acid.

Fatty acid composition of the oil

Alabi et al. (2013)[12] method was adopted. 1 ml sodium methoxide (1.55 g NaOH in 50 ml of methanol) solution was added to 0.1 ml vegetable oil. The solution was stirred vigorously using a magnetic stirrer for 10 s. The solution was left for 10 mins to separate out, the clear solution of fatty acid methyl ester from cloudy aqueous layer. The upper layer was collected carefully which was injected into gas-chromatograph Model HP 6890 powered HP Chemstation Rev A 09.01 (1206) Software equipped with flame ionization detector and a capillary column HP INNOWax (30 m x 0.25 mm x 0.25 µm) to obtain individual peaks of fatty acid methyl esters. The inlet and detector temperatures were set at 250 °C and 320°C respectively. Gas flow was Nitrogen. The spin ratio was 20:1 with hydrogen pressure at 22 psi. The fatty acid methyl esters peaks were identified by retention times by means of comparing them with authentic standards analyzed under the same conditions.

Peroxide value

Peroxide value was determined using AOAC (2000)[6] method by weighing 1 gram of the oil into a clean dry boiling tube, 1 gram of powdered potassium iodide and adding 10cm³ of the solvent mixture. The mixture was allowed to boil vigorously for 30 seconds. The tube was washed twice with 25 cm³ portions of water and the washings were added to the titration flask. This was then titrated with 0.002 M Sodium thiosulphate using a starch indicator. The relation for peroxide value is given as:

$$\text{Peroxide value} = \frac{(V_2 - V_1) \text{cm}^3 \times \text{Molarity of titrant}}{\text{weight of oil}} \times 100 \quad (\text{meqKOH/g})$$

(7)

Where: V_1 = sample titre value,

V_2 = blank titre value

Iodine value

Iodine value was determined by weighing 0.2 gram of the oil into a 250 cm³ glass stopper flat, and adding 10 cm³ of carbon tetrachloride to the oil and dissolved. 20 cm³ Wijs' solution was equally added to the mixture and the content was corked with a stopper that initially moistened with potassium iodide solution in accordance with AOAC (2000)[6] method. The mixture was titrated with 0.1 M standard sodium thiosulphate solution using starch as an indicator just before the end point.

$$\text{Iodine value} = \frac{(V_2 - V_1) \times N \times 0.1269 \times 100}{\text{weight of oil (g)}} \text{ gI}_2 / 100\text{g}$$

(8)

Where V_1 = sample titre value (cm^3)

V_2 = blank titre value (cm^3)

N = normality of sodium thiosulphate

Pour point and cloud point

Pour point and cloud point were determined using ASTM D97-05 [13] method. The oil was poured into a test tube and placed into a refrigerator to solidify. The oil was removed after it solidified and a thermometer was used to read the temperature to which the solidified oil begins to melt and flow. The lowest temperature to which the movement is observed is the pour point. The cloud point is the temperature at which a cloud of wax crystals first appears when the oil is cooled.

Flash point and fire point

The flash point measurement was done in accordance with ASTM D93-02a [14] method. The oil was poured into a metal container and heated at 5°C interval with a flame being passed over the surface of the sample. The temperature at which an instantaneous flash occurs was taken immediately and recorded as a flash point. The fire point is that temperature at which the vapour of the oil burns constantly for 5 seconds when flame is brought near.

3 Results and discussion

3.1 Mineral composition of sandbox oil

It is good to note that pure sandbox oil contains magnesium, potassium, copper, iron and zinc among others as shown in Table 1. Potassium was the most abundant, followed in descending order by iron, sodium, zinc, copper, magnesium, manganese, nickel, lead and chromium. Lead and chromium were present in relatively low values. The values for these minerals are comparable with those reported by Abdulkadir et al. (2013)[15].

Table 1. Mineral composition of sandbox oil

Mineral	Value (ppm)
Magnesium	1.98 ± 0.02
Potassium	40.28 ± 0.03
Copper	3.02 ± 0.01
Iron	25.45 ± 0.02
Zinc	14.70 ± 0.01
Lead	0.89 ± 0.03

Sodium	15.20 ± 0.02
Manganese	1.86 ± 0.01
Chromium	0.50 ± 0.02
Nickel	1.61 ± 0.01

Values are means $\pm$ standard deviation of 3 replications.

3.2 Chemo-physical composition of the oil

The result of the chemo-physical composition of the sandbox oil is presented in Table 2.

Table 2. Chemo-physical properties of sandbox oil

Properties	Value
Moisture content	0.40 ± 0.02 (%)
Density	0.90 ± 0.04 (g/cm^3)
Specific gravity	0.93 ± 0.02
Refractive index	1.4679 ± 0.01
Viscosity at 28°C	49.20 ± 0.02 (cSt)
Saponification value	210.00 ± 0.05 (mgKOH/g)
Acid value	1.68 ± 0.02 (mgKOH/g)
Free fatty acid value	8.42 ± 0.03 (mgKOH/g)
Peroxide value	0.87 ± 0.04 (mg/100g)
Iodine value	177.66 ± 0.01 ($\text{gI}_2/100\text{g}$)
Pour point	-3.00 ± 0.03 ($^\circ\text{C}$)
Cloud point	11.00 ± 0.02 ($^\circ\text{C}$)
Flash point	249 ± 0.03 ($^\circ\text{C}$)
Fire point	252 ± 0.01 ($^\circ\text{C}$)

Values are means $\pm$ standard deviation of 3 replications.

Moisture content

The moisture content of the sandbox oil as shown in Table 2 was 0.40 ± 0.02 %, which is within the allowable limit of 0.1% - 0.5% for vegetable oils and is the same as that reported by Okolie et al. (2012)[4]. This low moisture content according to Nkafamiya et al. (2010)[16] shows that the oils will have a longer shelf-stability as it will not easily give in to hydrolysis and ease of attack by oxygen. The lower the water content, the better the quality of the oil, this is because the water in the oil can trigger a hydrolysis reaction which causes a decrease in the quality of the oil due to an increase in free fatty acid levels [17].

Density and specific gravity

The density and specific gravity of the sandbox oil at 28°C were 0.90 ± 0.04 g/cm^3 and 0.93 ± 0.02 respectively which fall within the standard range of common oils as approved by the Standard Organization of Nigeria [18]. The obtained values are in line with the values reported by Mohammed (2015)[9] and Bilal et al. (2013)[19] for jatropha oil and 0.915 g/cm^3 reported by Trajano et al. (2014)[20] for soybean and sunflower oils. The

density of oil, according to Fakhri and Qadir (2011)[21], is dependent on their fatty acid composition, minor component and temperature, while the specific gravity is influenced by the chemical composition of the oils. The density and specific gravity values of the oil showed that the oil is less dense than water. Hence, in case of contamination with water, water will settle below the oil and will be subsequently drained off.

Refractive index

Refractive index indicates the level of saturation of the oil and is widely used in quality control to check for the purity of materials, to follow and control hydrogenation and isomerization processes [22]. The refractive index of the oil was 1.4679 ± 0.01 as shown in Table 2, which was within the acceptable range of 1.4677 to 1.4707 for virgin, refined and refined-pomace oils reported by Aremu et al. (2015)[23]. The values indicated that the oil is less thick when compared with most oils whose refractive indices were between 1.480 and 1.490 reported by Olatidoye et al. (2010)[24]. This value is satisfactory as it lies within the standard range found in common oils reported by Aji et al. (2012)[25] and Akintayo et al. (2002)[26].

Viscosity

Viscosity, which is defined as a measure of fluid's resistance to flow, is one of the critical and important properties of oil. The viscosity of sandbox oil at room temperature (28°C) was 49.20 ± 0.02 cSt as shown in Table 2. This value agreed with the viscosity values of some common oils reported by Olatidoye et al. (2010)[24]. Sripada (2012)[27] reported viscosities of 48.50 cSt for canola oil, Mohammed (2015)[9] also reported viscosities of 47.57 cSt for jatropha oil, 50.73 cSt for moringa oil and 46.46 cSt for cotton oil at 28°C. The viscosity of oils, according to Kim et al. (2010)[28], depends on molecular structure and decreases with the unsaturation of fatty acids. Oils are mixtures of triglycerides and their viscosity depends on the nature of the triglycerides present in the oil. Oils with low viscosity values are light and probably highly unsaturated. The more viscous oil is, the better its use for industrial purposes according to Aremu et al. (2015)[23].

Saponification value

Saponification value gives an indication of the molecular weight of the fatty acid contained in the oil and the purity status of the oil or whether the oil is adulterated [29]. It enhances the quality of the oil, according to Aremu et al., (2015)[23], as it shows the

presence of lower molecular weight components in 1 gram of the oil which will yield more energy on combustion. The saponification value of the oil was 210.00 ± 0.05 mgKOH/g, which is significantly high and suggest that the oil contained a high proportion of free fatty acids. Olatidoye et al. (2010)[24] reported that oils with high saponification values contain a high proportion of free fatty acids. Yerima et al. (2018)[30] also reported that the higher the free fatty acids, the higher the saponification value and vice versa.

The presence of high saponification values which indicate the presence of a high percentage of free fatty acids might lead to foam formation. Foaming is a desired characteristic of good surfactants with applications in preparation of emulsions, soaps and detergent formulation according to Kudirat and Otutu (2016)[31]. This suggests that the oil can be used in production of soaps, shampoos, lather shaving creams, paints and bio-lubricant [29]. There could also be an extension to its application into stable foam by incorporation of other ingredients. Therefore, the oil may be used in many other industrial applications. The obtained value is comparable to 198.76 mg KOH/g reported by Mohammed (2015)[9] for crude jatropha oil, 193.04 mg KOH/g for moringa oil, 196.35 mg KOH/g for castor oil and 194.75 mg KOH/g for cotton oil. The value also aligns with 198.76 mg KOH/g reported by Bilal et al. (2013)[19] for crude jatropha oil.

Acid value

Acid value, according to Maliki et al. (2020)[29], measures the amount of free fatty acids which have been liberated by hydrolysis from the glycerides due to the action of moisture, temperature and/or lypolytic enzyme lipase. It is also a relative measure of rancidity as free fatty acids are normally formed during decomposition of oil glycerides. It is an important indicator of vegetable oil quality. Acid value gives an indication of the deterioration, rancidity, or edibility of the oil. It is used as an indicator for edibility of an oil and its suitability for use in industrial applications [23].

The lower the acid value of the oil, the higher the quality. High acid levels imply that the oil will require an excess polyol for its polycondensation reaction. The acid value of the oil was 1.68 ± 0.02 mgKOH/g, which falls within the range of 0.34 – 68.88 reported by Aremu et al. (2015)[23] for common oils. The acid value of the oil was low which indicates that the oil was in a good non-degraded state of the lipid and was within limits for

industrially useful oils [32]. This is very good because the lower the acid value of the oil, the higher the quality and oils with low acid values are useful industrially in the manufacture of lubricant, paint and varnish [4]. Low acid values in oils indicate that the oils will be stable over a long period of time and protect against rancidity and peroxidation.

Free fatty acid

Free fatty acid value is the value of specified fatty acid in oil and it is an index for determining the quality of oils. Oils with substantial amounts of unsaturated fatty acids are susceptible to oxidation. Aremu et al. (2015)[23] reported that the quantity of free fatty acid in oil is an indicator of its overall quality; high quality oils are low in free fatty acids. Li et al. (2007)[33] also reported that oil with low free fatty acid has lesser susceptibility to rancidity. An excessive amount of free fatty acids lowers the flash point of oil and will cause ‘popping’ of the oil during heating. The free fatty acid value of the oil was 8.42 ± 0.03 mgKOH/g as shown in Table 2, which was around the free fatty acid values of common oils reported by Aremu et al. (2015)[23].

Fatty acid composition

The oil contains high content of unsaturated fatty acids such as oleic (33.12%), linoleic (1.01%), and linolenic (2.30%) acids as well as stearic (4.32%) and palmitic (3.17%) which are saturated. This revealed that oleic acid (33.12%) and stearic acid (4.32%) are the most abundant unsaturated and saturated fatty acids respectively. These values are in close agreement with those reported by Abdulkadir et al. (2013)[15].

Peroxide value

Peroxide value is used to determine the extent to which oil can go rancid as a result of storage, heating or oxidation. It is the most common indicator of lipid oxidation and gives an indication of the quality and stability of oil. High peroxide values, according to Aremu et al. (2015)[23], indicate high levels of oxidative rancidity of the oils and also suggest absence or low levels of antioxidants. Rancid oils have peroxide values of 10 – 20 meqKOH/g oil. The peroxide value of the oil was found to be 0.87 ± 0.04 mg/100g which is low and falls within the standard range reported by Saeed and Shola (2015)[34]. The low peroxide value recorded for the oil showed the oil is less prone to rancidity. This indicates that the oil is stable to oxidative rancidity and can stand oxidative degradation.

Iodine value

Iodine value is a measure of degree of unsaturation of fats and oils and could be used to quantify the amount of double bond present in the oil which reflects the susceptibility of the oil to oxidation. High iodine value means high degree of unsaturation fats and oils. Saturated oils and fats have zero iodine value because they cannot take up any iodine [29]. The higher the iodine value, the less stable, softer, more reactive and susceptible to oxidation the oil will be. According to Aremu et al. (2015)[23], the lower the iodine value the lesser the number of unsaturated bonds; thus the lower the susceptibility of such oil to oxidative rancidity. The iodine value of the oil was 177.66 ± 0.01 gI₂/100g as shown in Table 2. This value is comparable to the iodine value of 174.9 gI₂/100g for jatropha oil and 185.6 gI₂/100g for moringa oil reported by Mohammed (2015)[9] in a previous research work. The iodine value of the oil was high, owing to the fact that the oil contains unsaturated glycerides, which have the ability to absorb a definite amount of iodine and can be used for the production of lubricant, paint and alkyd resin. Oil whose iodine value is high according to Wara et al. (2011)[35], possesses the property of absorbing oxygen on exposure to the atmosphere, become thickened and remain sticky but do not form a hard dry film and they can be used in the production of margarine and soap.

Pour and cloud points

Pour point is defined as the lowest temperature at which movement was observed, while cloud point is the temperature at which a cloud of crystals first appears when the lubricant is cooled. Viscosity, according to Ekwierhoma and Ekop (2016)[22], influences the pour point of oils; the higher the viscosity the lower the pour point. Pour point of the oil was -3.00 ± 0.03 °C; while cloud point of was 11.00 ± 0.02 °C as shown in Table 2. These values agreed with the results reported by some researchers. Menkiti et al. (2017)[36] reported the pour point of -7 °C for crude jatropha oil, Bilal et al. (2013)[19] reported pour point of -5 °C for jatropha oil, Aji et al. (2015)[25] reported pour point of 1.30 °C for neem oil. Low temperature fluidity according to Sripada (2012)[27] is the most essential property for oils to perform in environments that are extremely cold.

Flash and fire points

The flash point is the temperature at which an instantaneous flash occurs when flame is brought near the oil while the temperature at which the

vapour of the oil burns constantly for 5 seconds is the fire point. Fire point is always flash point plus 5°C up to 400°C. The flash point of the oil was 249 ± 0.03 °C, while the fire point was 252 ± 0.01 °C as presented in Table 2. This value is similar to the value reported by Ettefaghi et al. (2013)[37]. Ahmed et al. (2014)[38] reported flash point of 256°C for soybean oil, Aji et al. (2015)[25] reported a flash point of 258°C for neem oil and 254°C for jatropha oil. It is clear from the results obtained that the oil has very good flash and fire points as it can be used in both humid and temperate regions and transported safely with minimum risks of explosion.

4 Conclusion

The mineral composition of the oil showed that the oil contains some metallic elements. The presence of these elements in the oil will help in friction reduction in the engine and as cleaner and neutralizer when used as lubricant. These metallic elements will form protective film by chemical or physical absorption on the contacting metal surface, leading to the sliding friction reduction. The chemo-physical properties of the sandbox oil revealed that the oil has many industrial attributes. The low viscosity of the oil would make it useful in the production of bio-fuels. Its high saponification value would make it to be used in the manufacture of soaps, body creams, cosmetics, plasticizers and lubricants. The low peroxide value indicates that the oil is stable to oxidative rancidity and can stand oxidative degradation. The overall results suggest some potential industrial applications of the sandbox oil for bio-lubricant, paint, cosmetics, shampoos, lather shaving creams, and soap productions.

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Conflict of Interest

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