

RESEARCH ARTICLE

Effect of extraction methods on food and biodiesel properties of shea-nut oil (*Vitellaria paradoxa*)

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ABSTRACT

Shea nut oil is one of the most abundantly available vegetable oils produced in the sub Sahara region. It is mostly used for food and medicine. The need to use it for biodiesel to solve the energy need of the region has been a major concern for African. Three extraction methods, namely: Traditional, solvent and mechanical method was used to extract shea nut oil. Some food and biodiesel properties of the oil were tested and compared between each other. Properties investigated were; colour, moisture content, protein, free fatty acid, saponification, solidification, viscosity, melting point, specific gravity, flash point, cloud point, refractive index, iodine value. The results showed that extracting methods of Shea nut oil have a significant effect ($p < 0.05$) on moisture content, protein, free fatty acid, saponification, viscosity, melting point and iodine but not on solidification, specific gravity, flash point, cloud point and refractive index. Among the three extraction methods compared, mechanical method had less effect on food and bio-diesel properties of shea nut oil, followed by the traditional method and then the solvent method.

Keywords: Traditional methods; solvent method; mechanical method; Shea nut oil, food properties

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INTRODUCTION

In Nigeria, there are abundant vegetable oils, namely; palm oil, coconut oil, groundnut oil, rubber seed oil, cotton seed oil, soya bean oil, etc. (Dawodu, 2009). Vegetable oils are extracted from fruits, seeds kernel and nuts either by mechanical press or by solvents (Akpabio et al., 2011). The shea tree (*Vitellaria paradoxa*) grows either naturally or as a cultivated tree crop in the dry Savanna belt of West Africa. The West Africa sub-species *Vitellaria paradoxa* and *Vitellaria nilotica* occur in 19 countries across the African continent. It is a perennial tropical tree, which grows to the height range of 15-20 m, the fruits fall when fully ripened. The Shea-butter nuts obtained from the fruit shell tree also known as Chamen, Kadanya/Mankade, Osisi/Okwuma, and Emi/Orioyo among the Tiv, Hausa, Igbo and Yoruba people of Nigeria respectively.

The Shea fruit has a kernel (from which Shea oil is extracted), a green pericarp, a fleshy mesocarp (pulp) and a shell cover (endocarp). The kernel, according to Axtell et al. (1993) contains about 60% edible fat. The kernels can be dried, cracked and crushed to obtain Shea oil, which forms a solid, fatty, butter-like substance called 'Shea butter' (Coulibaly et al., 2009), the kernels can be processed using; Traditional extraction method (Jatto et al., 2010), Solvent extraction method (Asuquo et al., 2010; Kar and Mital, 1999) and Mechanical extraction method.

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Shea butter is a vegetable fat extracted from the kernel of the fruit of the Shea tree (*Vitellaria paradoxa*), a tree belonging to the family of sapotaceae. The tree is the main indigenous oil producing wild plant spontaneously growing in Africa (Honfo et al., 2012). Hee (2011) reported that the Shea tree begins to bear fruit of commercial quantities after approximately 20 to 50 years. In comparison to other trees grown as plantation crops, Shea tree takes longer time to reach maturity, which discouraged its commercial plantation. Alander (2004) reported that the trees do not reach maturity until 45 years and can continuously produce Shea nuts for up to 200 years in commercial quantities. The tree grows wild across a 500 km wide belt of savanna (Masters et al., 2004), including West African countries like Senegal, Mali, Code D'Ivoire, Burkina Faso, Togo, Ghana, Benin, Nigeria, Niger, Cameroon, and in East Africa such as Uganda, Sudan and Ethiopia (Hee, 2011). Four of these countries account for about 600,000 MT (approx. 80%) of world Shea nut production, which includes, Nigeria (370,000 MT), Mali (85,000 MT), Burkina Faso (70,000 MT) and Ghana (61,000 MT) (Karen, 2005). Among these countries, Ghana and Burkina Faso are the main Shea nut exporters (Hee, 2011). Nigeria produces about 50% of global Shea nut production, but tends to consume most of its Shea nuts locally (Karen, 2005).

In Nigeria, the Shea tree grows in Niger, Kwara, Kebbi, Kaduna and Oyo State. Shea-butter oil extracted from Shea nut is botanically called *Butyrospermumparkii*. It is a soft paste of melted fat with a milky color in solid form and brownish when melted with a characteristic odor (Eka, 1997). It is an ancient African commodity, which plays an important role in village life (Honfo et al., 2012). This native source of edible oil or fat is traditionally used for frying, adding to sauces, as a skin pomade, for medicinal applications, to make soap, oil for lanterns and for cultural purposes at ceremonies, such as births, weddings and funerals. It can also serve as a cocoa butter equivalent in the manufacture of chocolate as well as an ingredient in cosmetics (Alander, 2004).

This exceptionally rich vegetable extract contains fatty acids, phytosterol and unsaponifiable matter, which stimulate the skin's natural renewable process. Asuquo et al. (2010) reported that unrefined Shea butter oil is superior to refined Shea butter oil in that it retains all its natural vitamins, especially vitamins A and E. It also has natural anti-oxidant properties due to its tocopherol content. Shea nut contains 37-55% of fats; it is composed mainly of two fatty acids, stearic and oleic, which together account for 85-90% of the fatty acids (Ferris et al., 2001). Maranz et al. (2004) reported that Shea butter oil contains 2-6% palmitic acid (C16), 15-25% linoleic acid. Asuquo et al. (2010) in their effort to fill the gap existing in literature on the Nigeria Shea butter reported the extraction of Shea butter from the Shea nut obtain from Kano, a satellite town in Abuja. The author reported that there are usually variation in the Physico-chemical composition of vegetable oils depending on environmental factors such as available rainfall, soil fertility, maturation period, agronomic practices and genetic substitution. Sonau et al. (2006) added that the composition of the Shea nut product depends on a number of criteria, particularly the geographical occurrence, its botanical origin, handling of the seeds and processing e.g. drying time and ripening.

Shea butter is mainly used in the cosmetics industry for skin- and hair-related products (lip gloss, skin moisturizer creams and emulsions, and hair conditioners for dry and brittle hair). It is also used by soap makers, typically in small amounts (5-7% of the oils in the recipe), because it has plenty of unsaponifiables. In some African countries such as Benin, Shea butter is used for cooking oil, as a waterproofing wax, for hairdressing, for candle-making, and as an ingredient in medicinal ointments. It is used by makers of traditional African percussion instruments to increase the durability of wood (such as carved djembe shells), dried calabash gourds, and leather tuning straps. Shea butter can be an ingredient of organic broth (National Research Council, 2006).

Enweremadu and Alamu (2010) studied Shea nut butter extraction from cold press method and investigated it as feedstock for the production of biodiesel. Biodiesel yield was used to verify the optimization, while density and viscosity were

chosen to serve as an indicator for the effectiveness and completeness of the ester conversion process. Based on the amount of Shea butter used, the final product yield obtained was 94.55% mass and the percentage conversion of FFA in Shea butter to biodiesel was 92.3% using a methanol/oil ratio of 6:1 and 1.0% mass KOH at 60 min and 55 °C, respectively. The important properties of the biodiesel (density, kinematic viscosity, cloud point, pour point, cetane number, neutralization number, iodine value, methyl ester content and higher heating value) were compared to those of ASTM and EN standards for biodiesel. The comparison shows that the shea butter methyl ester could be used as an alternative to diesel.

Eziekel (2008) reported the production of biodiesel from Shea butter oil using methanol as solvent. He concluded that the density was 0.876g/ml at 15 °C, specific gravity was 0.887, viscosity was 4.65cst, flash point was 94 °C, ash content was 0.007%, smoke point was 5.7cm and PH value was 2.15. Okafor et al. (2015) studied the effect of process variables on the production of biodiesel by the non-catalytic supercritical trans-esterification of Sheanut oil. Physico-chemical properties of Shea-nut oil extracted using mechanical and solvent methods were compared with that of Shea butter. It is relevant to advance the study of previous researchers to include all extraction methods and other properties of shea nut not mentioned in their works. The objective of this paper was to report food and bio-diesel properties of shea nut oil and discuss the effect of three extraction methods, namely traditional, mechanical, and solvent method on these properties.

MATERIALS AND METHODS

Sample preparation

Shea butter seed was bought from the Kasuwan Magani market in Kaduna, Nigeria. It was clean by winnowing using a sieve called (matankadi). After the cleaning, it was sorted to remove the bad and rotten ones. The seeds, were manually shelled, fried, pounded, and divided into three parts of 20kg in accordance to the extraction methods (traditional, solvent and mechanical methods of extractions). Two portions were grounded into pastes for the traditional and solvent extraction.

Traditional methods

For the traditional methods, water was warmed up to 85 °C while the warming was going on, the paste was prepared and poured into a big clay pot, the warm water was added while stirring the paste, the surface of the paste turned oily and it was stirred harder. At a point in time, cold water was added to almost the brim of the clay pot and it was observed that oily substance mixed with fine grains of the past were suspended on the surface of the water. The oily substance was skimmed using a tablespoon and heated to evaporate the trapped molecules of water. On cooling, it was decanted into jars and at solidification; it turns a hard gray substance called shea butter oil.

Solvent methods

The process for the solvent extraction has many things in common with the traditional method, from cleaning to stirring, except for the fact that a known volume of n-hexane was introduced. In this case, 20 cl of the substance was added while stirring was going on. Warm water was added while stirring. When the paste turned oily, cold water was filled to the brim of the clay pot. The oil was skimmed, mixed with fine grains of ground materials and water as in the traditional method it was heated to evaporate water molecules and decantation was done with jars. On cooling a gray hard substance called shea butter oil was formed.

Mechanical press method

The pounded sample was put in a funnel-like container, which has a rod that has round blades that is connected to the shaft. The blades as they turn press the sample down to the turning shaft, which presses the sample there by pressing out the oil and the press is powered by electricity. The machine has pulleys that turn the shaft. As the pressing continues, water is added from time to time, possibly to moisten it for easier flow. The machine presses out the oil into a collecting tray that has a sieve underneath sieving the oil from the dregs. The oil is collected into 1/3 drum while cake is collected on the other side of the machine in a basin. The collected sieved oil was heated to remove water molecules trapped in it. It was allowed to stand for some time, it was decanted into a jar on cooling and a hard gray substance called shea-butter oil.

Determination of moisture content, protein, colour

The moisture content was determined using the ASTM D1744 standard. Protein content was determined using IS: 7219 – 1973 standard. Colour was determined using ASTM D1981-11 (2015) standard.

Determination of specific gravity

The prepared sample placed in a dry pycnometer to prevent the entrapment of bubbles after removing the cap of the side arm. The pycnometer immersed in a water bath of 30 °C and held for 30 minutes. The oil coming out of the capillary opening was wiped off. The bottle removed from both cleaned and dried thoroughly. The cap of side arm removed and weighed to ensure that the temperature did not fall below 30 °C. From the relation, specific gravity at 30 °C = $(A - B) / (C - B)$. Where: A, B and C, represent weight in gm of specific gravity of bottle with oil, bottle and bottle with water all at 30 °C.

Determination of melting point

Dry sample melted and filtered using a filter paper in order to remove any impurities and last traces of moisture, the sample then mixed thoroughly. A capillary tube introduced into the molten sample so that a column of the sample of about 10mm long soaked into the tube. The tube containing the sample immediately chilled by touching the tube against a piece of ice until the fat solidified. The tube placed in a small beaker and held for 1 hr in water at a temperature of 10 °C. The tube removed and attached with a rubber band to the thermometer at the same level. The thermometer with the capillary tube containing the sample of oil immersed in water at 10 °C in the "Thiele" tube. The temperature gradually increased by heating the side tube of the thiele at the rate of 2 °C per minute until the temperature reaches 25 °C then reduced thereafter at the rate of 0.5 °C per minutes. The temperature of the water was noted when the sample column began to rise in the capillary tube. The average of the two separate determinations reported as the melting point if the reading did not differ by more than 0.5 °C.

Determination of saponification

The dry sample melted and filtered using a filter paper to remove impurities and last traces of moistures. The dry sample mixed thoroughly and weighted at 1.5g into a 25ml Erlenmeyer flask. 25ml pipette of alcoholic potassium hydroxide solution introduced into the flask. A blank determination was conducted along with the sample. The sample flask and blank flask with air condensers were connected and kept in the water bath, it boiled gently and steadily until saponification completed as indicated by the absence of any oily matter and appearance of clear solution. Clarity achieved within 1hr of boiling. After the flask and condenser had cooled, the inside of the condenser washed with 10ml of hot ethyl alcohol. Titrate the excess potassium hydroxide with 0.5N hydrochloric acid, using 1.0ml phenolphthalein indicator.

Mathematically, saponification Value=56.1 (B-S) /NW

where B and S represents; volume in ml of standard hydrochloric acid required for blank and sample while N and W is for normality of standard hydrochloric acid and weight in gm of the oil taken for the test.

Determination of solidification

Solidification was determined using ASTM D5565-95 (2011) standard.

Determination of iodine value

An appropriate quantity of dry oil was accurately weighed into a 500 ml conical flask with a glass stopper to which 25 ml of carbon tetrachloride was added. The contents were properly mixed. The weight of the sample was such that there was an excess of 50% of Wij's solution. Pipette 25 ml of Wij's solution and replace, the glass stopper after wetting with the potassium iodine solution. Mixed properly, the flask was kept in the dark for an hour for drying oils. A blank was simultaneous, 15 ml was added after standing, 15 ml of potassium iodine solution added, followed by 100 ml that was recently boiled and cooled in water, the stopper was also rinsed. Titrated liberated iodine with a standardized sodium phosphate solution, a starch indicator was used at the end until the blue colour formed disappeared after a thorough shaking with the stopper on. Blank determinations were conducted in the same manner as test sample, but without the oil. Slight variations in temperature appreciably affected titer of iodine solution as chloroform had a high coefficient of expansion. Thus, it was necessary that blanks and determination were made at the same time.

Mathematically: iodine value = 12.69 (B-S) /NW

where B=volume in ml of standard sodium thiosulphate solution required for the blank. S=volume in ml of standard sodium thiosulphate solution required for sample. N=normality of the standard sodium thiosulphate solution. W=weight in g of the sample.

Determination of flash point procedure

The sample was heated in a test cup at a slow and constant rate and stirred continuously. A small test flame was directed into the cup at regular intervals with simultaneous interrupted stirring. The flash point was taken at the lowest temperature at which the application of the test flame caused the vapor above the sample to ignite momentarily.

Determination of free fatty acid

10 ml of oil sample was weighed into a conical flask. Three drops of phenol phthalin indicator and titrated against 0.1N sodium hydroxide solution to a pink end point. Mathematically; % FFA= (Titra value) / (sample x 5.61)

Determination of cloud point

10 ml of oil sample was placed into a 15 ml conical flask and transferred into a refrigerator at 10 °C and observed every 30 minutes until a cloud was observed in the temperature at which the cloud was observed was taken as the cloud point.

Determination of viscosity

259 ml of the oil sample was weighed into 250 ml conical flask. The sample was transferred to the viscometer at a speed of 60 rpm and spindle number for the readings obtained was recorded and the viscosity (cp) was calculated.

Determination of refractive index

Refractive index was determined using ASTM D1747-09 (2014) standard.

Statistical analysis

The statistical analysis was done using a software program called SPSS. The mean separation was done using Duncan Multiple Range Test at 95% confidence level ($P < 0.05$).

RESULTS AND DISCUSSION

Table 1 shows the effect of processing methods on some food and biodiesel properties of shea nut oil while table 2 is the ANOVA result on effect of processing methods on properties of Shea nut oil.

Color

Table 1 shows that the colors of the shea nut oil obtained for the different processing methods were different from each other. The colour obtained from the traditional methods was brown. This could be due to the fact that the oil produced reacts with clay properties of the pot it was produced in. The colour for the mechanical method was cream yellow which could also be because the cake particles and the oil were completely separated. Result of solvent method was black, this could be because the oil extracted reacted with the chemical used to extract it.

Moisture content

Table 2 shows that processing methods had a significant effect ($p < 0.05$) on moisture content of shea nut oil when extracted. The moisture content was found to be in the range of 0.05% to 0.086% for traditional method to solvent method. Mean separation shows that moisture found in the extracted oil differed significantly from each extraction method. Similar results were obtained by Okafor et al. (2014).

Protein

Methods of shea nut extraction had a significant effect ($p < 0.05$) on the protein content of the oil. It ranged from 3.763 mg/ml in solvent extraction to 4.776 mg/ml in traditional. Mean separation shows that protein content for a traditional and mechanical method had no significant difference, but were significantly different from solvent method. This difference could be because some of the chemicals used in the solvent process react with the protein content to form different compounds. In the food and pharmaceutical industry protein content is very important and it is best to use traditional or mechanical methods.

Free fatty acid

ANOVA (table 2) shows that extraction methods had a significant effect at 95% confidence level on free fatty acid

content of shea nut oil. The values ranged from 10.783 in the mechanical method to 13.940 in the solvent method. The mean separation shows that the values obtained from traditional and mechanical methods were statistically the same, but were statistically different from the solvent method. The increase in free fatty acid could be due to the chemical used in the solvent method. Julius et al. (2013) obtained similar results.

Table 1: Effect of processing methods on some fuel properties of Shea butter oil

| Method | Colour | Moisture (%) | Protein (mg/ml) | FFA | Saponification (mg/g) | Solidification | Viscosity (mm ² /s) | Melting Point (°C) | Specific Gravity (g/cm ³) | Flash Point (°C) | Cloud Point (°C) | Reflective Index | Iodine (mg/g) |
|-------------|--------------|--------------------------------|--------------------------------|--------------------------------|---------------------------------|--------------------------------|--------------------------------|--------------------------------|---------------------------------------|-----------------------------------|--------------------------------|-------------------------------|--------------------------------|
| Traditional | Brown | 0.0533 ^a (0.006) | 4.7767 ^a (0.375) | 10.953 ^a (0.666) | 174.950 ^a (0.624) | 10.334 ^a (0.577) | 2.743 ^a (0.136) | 60.667 ^a (1.155) | 0.907 ^a (0.012) | 155.000 ^a (32.924) | 19.000 ^a (0.000) | 1.471 ^a (0.012) | 15.210 ^a (0.017) |
| Mechanical | Cream yellow | 0.733 ^b (0.006) | 4.387 ^a (0.258) | 10.783 ^a (0.252) | 175.433 ^a (0.465) | 10.667 ^a (0.577) | 2.850 ^a (0.139) | 62.667 ^b (0.577) | 0.913 ^a (0.006) | 746.333 ^a (980.918) | 18.667 ^a (0.577) | 1.468 ^a (0.025) | 15.420 ^b (0.026) |
| Solvent | Black | 0.0867 ^c (0.006) | 3.763 ^b (0.235) | 13.940 ^b (0.164) | 143.033 ^a (1.903) | 11.000 ^a (1.000) | 3.450 ^b (0.500) | 62.667 ^b (0.577) | 0.927 ^a (0.006) | 180.667 ^a (1.155) | 17.333 ^a (0.577) | 1.469 ^a (0.012) | 16.113 ^c (0.058) |

Different letters along the column shows different means according to Duncan new multiple range test ($p < 0.05$) and numbers in parentheses are standard deviation

Table 2: ANOVA on effect of processing methods on some fuel properties of Shea butter oil

| Source of Variance | Sum of Square | df | Mean Square | F | Significant |
|--------------------|---------------|----|-------------|----------|----------------------|
| Moisture content | 0.002 | 2 | 0.001 | 25.333 | 0.001* |
| Protein | 1.567 | 2 | 0.784 | 8.947 | 0.016* |
| Free fatty acid | 18.914 | 2 | 9.457 | 890.286 | 0.000* |
| Saponification | 2068.667 | 2 | 1034.334 | 733.784 | 0.000* |
| Solidification | 0.667 | 2 | 0.333 | 0.600 | 0.579 ^{N.S} |
| Viscosity | 0.871 | 2 | 0.435 | 32.464 | 0.001* |
| Melting point | 6.889 | 2 | 3.444 | 5.167 | 0.050* |
| Specific gravity | 0.001 | 2 | 0.000 | 4.667 | 0.060 ^{N.S} |
| Flash point | 670312.667 | 2 | 335156.333 | 1.044 | 0.408 ^{N.S} |
| Cloud point | 4.667 | 2 | 2.333 | 10.500 | 0.11 ^{N.S} |
| Iodine | 1.341 | 2 | 0.670 | 1946.355 | 0.000* |
| Refractive index | 0.000 | 2 | 0.000 | 2.593 | 0.94 ^{N.S} |

N.S = Non significant ($p > 0.05$) and * = significant ($p < 0.05$)

Saponification

Values for saponification ranged from 143.033mg/g in the solvent method to 175.433mg/g in the mechanical method. ANOVA shows that extraction method had a significant effect ($p < 0.05$) on saponification content of the oil. The mean separation showed that the values obtained for traditional and mechanical methods were statistically same, but were statistically different from the solvent method. The low saponification value found in the solvent method was due to the high fat content of the oil. Oil that is used for fuel are recommended to be extracted using either the traditional method or the mechanical method. Similar range was obtained by Okafor et al. (2014).

Solidification

Solidification values ranged from 10.334 for traditional method to 11.0 for solvent method. But statistical analysis

showed no significant difference ($p < 0.05$) for the values of solidification for different extraction method. Julius et al. (2013) obtained the similar result.

Viscosity

Value of viscosity was found to range from 2.74 for traditional method to 3.450 mm² /s for solvent method. ANOVA shows that extraction methods had a significant effect ($p < 0.05$) on the viscosity of the oil. Mean separation showed no statistical difference between the traditional and the mechanical method, but different with the solvent method. This could be due to the presence of the chemical used in the solvent methods. Similar results were obtained by Enweremadu and Alamu (2010) and Eziekel (2008)

Melting point

Extraction methods had a significant effect on the melting point of shea nut oil at 95% confidence level. Values obtained in the research ranged from 60.667 for traditional to 62.667 °C for both mechanical and solvent methods. Mean separation showed that melting point of mechanical and solvent method were the same, but differed from that of traditional methods. Julius et al. (2013) obtained similar results

Specific Gravity

ANOVA shows that extraction methods had no significant effect at 95% confidence level on specific gravity of Shea nut oil. The result obtained was found to range from 0.907 in the traditional method to 0.927g/cm³ in the solvent method. Julius et al. (2013), Enweremadu and Alamu (2010) and Eziekel (2008) also obtained similar results

Flash point

Values for flash point ranged from 155.0 in the traditional method to 180.667°C in the solvent method. ANOVA shows that extraction method had no significant effect ($p < 0.05$) on the flash point value of the oil.

Cloud point

Values for cloud point ranged from 17.333 in the solvent method to 19.0 °C in the traditional method. ANOVA shows that extraction method had no significant effect ($p < 0.05$) on the cloud point value of the oil. Enweremadu and Alamu (2010) and Okafor et al. (2014) also obtained similar results

Refractive index

Values of refractive index ranged from 1.471 in the traditional method to 1.469 in the solvent method. ANOVA shows that extraction method had no significant effect ($p < 0.05$) on the refractive index value of the oil. This is also in agreement with results obtained by Julius et al. (2013) and Okafor et al. (2014).

Iodine

Methods of shea nut extraction had a significant effect ($p < 0.05$) on the iodine content of the oil. Its value ranged from 15.210 in traditional extraction to 16.113 mg/g in solvent method. Mean separation showed that the iodine content for a

traditional, mechanical and solvent method were significantly different from each other. Julius et al. (2013) and Enweremadu and Alamu (2010) also obtained similar results

CONCLUSION

The values of moisture content, protein, free fatty acid, saponification, solidification, viscosity, melting point, specific gravity, flash point, cloud point, refractive index and iodine found to vary from 0.05- 0.086% , 3.763-4.776 mg/ml, 10.783-13.940 , 143.033-175.433 mg/g , 10.334-11.0, 2.74-3.450 mm²/s, 60.667-62.667°C, 0.907-0.927 g/cm³ , 155.0-180.667°C , 17.333-19.0 °C , 1.471-1.469, 15.210 and 16.113 mg/g, respectively. Colour of oil obtained was brown, cream yellow and black for traditional, mechanical and solvent methods, respectively.

Extraction method of shea nut oil was found to have a significant effect ($p < 0.05$) on moisture content, protein, free fatty acid, saponification, viscosity, melting point and iodine, but not on solidification, specific gravity, flash point, cloud point and refractive index. Among the three extraction methods, mechanical method had less effect on food and bio-diesel properties of shea nut oil, followed by the traditional method and then the solvent method.

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