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Production and characterization of biodiesel from used edible cooking oil using bifunctional catalyst

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Abstract

Waste-to-wealth generation is a key technology attracting more attention from developing African countries. Used edible cooking oil was used in generating biodiesel using Bifunctional Acid Base derived catalyst through the process of transesterification in the presence of primary alcohol (Methanol), yielding to biodiesel and glycerol. Characterization of both stating materials (Used Edible Cooking Oil and the resulting biodiesel was conducted and the results were compared with standard of the American Society for Testing and Materials (ASTM) as well as existing literature. The physicochemical properties of used edible cooking oil showed a Dark brown Oil colour, higher moisture and Free fatty acids content 1.29 ±0.01%, 0.14±0.02mg/KOH, low acid and saponification value 0.269±0.001mg/kg, 196.6±0.1mg/KOH/g and peroxide value of 6.8±0.02mEq/kg. While the biodiesel fuel properties showed Density 0.86±0.02g/cm3, Viscosity @40 (^{0}C) 0.97±0.01 mm/s, Specific Gravity@60 0.87±0.04 kg/l, Acid Value 11.2±0.1 mgKOH/g, Pour point -11.4±0.1^{O}C, Cloud Point -2.7±0.1^{O}C and Flash point 156.7 ±0.05 ^{O}C respectively. The GC-MS qualitative analysis of the synthesized biodiesel, showed different fatty acid methyl esters produces at different retention time with the highest percentage area at 9-Octadecenoic acid, 12-hydroxy-, methyl ester, Hexadecanoic acid, methyl ester or palmitic acid methyl ester, Hexadecanoic acid, 9,12,15-Octadecatrienoic acid, methyl ester.

***Keywords:** Biodiesel, Catalyst, Transesterification, Used cooking oil, Desert date seed shell.*

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1. Introduction

It was postulated that energy consumption plays an important role in the economic development of every nation (Jabri and Yahyaoui, 2025). Energy is a key component of the global economy, attracting more attention from developing African countries. Petroleum (Hydrocarbon) based fuels are source of affordable and reliable energy for many decades, but as a result of population increase, insecurity, depletion of petroleum reserve, Vulnerability to unstable global oil and environmental damages, were linked to issues like global warming, high toxicity, and non-biodegradability (Ayhad *et al.,* 2015). The recent global economy, waste from agricultural produced is considered to be very important feedstock because of their carbon rich, low cost, abundance, renewability, also contains high concentration of volatiles and low ash content, the listed factors favor the production of highly porous structure within activated carbon matrix (Abdullahi *et al.,*2026). Agricultural waste is a general term for organic substance discarded by humans in the process of agricultural activities (Altenor 2009). The availability of biofuels-based energy feedstock's, and their potential as a long-term energy source, can reduce the excessive dependence on petroleum fuels by offering renewable alternative (Ben-Iwo *et al.,* 2016).

Biodiesel refers to a group of oxygenated fuels with ester bases made from renewable biological sources. Biodiesel as a term describing the monoalkyl esters of long chain fatty acids produced from renewable bio lipids, it is produced when vegetable (edible and non-edible oil) or animal fats react with a catalyst in the presence of alcohol (primary alcohol) to form ethyl or methyl esters, which are then converted into biodiesel and glycerin (Lee *et al.,* 2014). Biodiesel synthesis via transesterification can occur using either an acidic, a basic, or an enzymatic catalyst under heating conditions. Biodiesel is a well-recognized biofuel due to its higher lubricity, biodegradability, low carbon dioxide emission, lack of sulfur, and non-toxicity (Yaakob *et al.,* 2013; Mardhiah *et al.,* 2017).

For the production of biodiesel, however, high feedstock costs, soil degradation, price increase and hunger-related threats are factors that make many agencies, particularly food and agricultural organizations to view this option as unreliable for commercial production of biodiesel (Yan *et al.,* 2009; Na. *et al.,* 2015). Reliable feedstock's for the marketable production of biodiesel include Jatropha curcas, castor oil, algae oil, used vegetable oil, and seed oil from desert date, but most of the crops are perennial in nature, planting them could result in deforestation, which could change the environment in the affected area, making it both costly and time-consuming (Supamathanon *et al.,* 2011).

Used cooking oil (UCO) /Waste cooking oil (WCO) is produced when food is cooked or fried in locally sourced oils (such as animal fats, oil from fish, palm kernel, castor, coconut, linseed, and soy bean (Rashtizadeh *et al.,* 2014). As a result of Humans' daily cooking and frying activities, approximately 15 million tons of used cooking oil is disposed up each year, while majority is drained into water ways systems and resulted in to water pollution making it unfit for the aquatic animal and difficult for waste management agencies to utilized (Lee *et al.*, 2014). Utilization of the used cooking oil as a feedstock for biodiesel production would lower costs, enhance food security associated with use food-grade oil, and minimize water contamination.

2. Materials and Methods

2.1. Sample Collection and Pre-treatment

About 5L of used cooking oil was collected at Dogondaji district tambuwal local government of Sokoto state Nigeria, after several used in frying Awara a local Hausa food, the sample was filtered to remove the residual Awara using Muslim cloth and then filter paper the clean and clear oil was stored at room temperature for further analysis.

2.2. Physicochemical Analysis of Used Cooking Oil

1. Colour of Oil

The colour of used cooking oil was determined according to the method of Ogala *et al*., (2018).

2. Moisture Content

The moisture content was determined according to the official method of the Association of Official Analytical Chemists AOAC (2008): 2g of sample was weighed and transferred into a pre-weighed beaker and then placed into an oven at 105^{O}C until a constant weight is achieved, after drying, the sample was removed and transferred to a desiccator and cooled at room temperature before reweighing. This was repeated in triplicate and the percentage moisture content was calculated

$$\text{Moisture content (\%)} = \frac{WI-W2}{WI} X\ 100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots . (1)$$

Where;

W_1= Sample weight before drying

W_1 =Sample weight after drying

3. Acid Value of the Oil

The method of AOAC, 2008 was used. 2g of the oil sample was weighed into a conical flask, and then 50cm^3 petroleum ether was added and gently mixed. Flowed by 50cm^3 ethanol then mix, the mixture was titrated with 0.1 M KOH to pink colour. The Acid value was calculated using the below equation

$$\text{Acid value (mg KOH/g)} = \frac{\text{Titre value} \times \text{Normality} \times 56.1}{\text{Weight of sample}} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots . (2)$$

Where

1cm^3 of 1 M KOH is equivalent to 56.1 mg of KOH

4. Peroxide Value of the Oil

The peroxide value (PV) of used cooking oil was determined according to the method reported by Wail *et al.,* (1995) and adopted by Mohammed (2018) : 1 g of the oil sample was weighed into 250ml conical flask, then 30 ml of a glacial acetic acid/chloroform solution (ratio 3:2) was added, and gently shaken until the sample is dissolved, 0.5ml of saturated potassium iodide was added and gently shake for 1 minute, and 30 ml of distilled water was added followed by 0.5ml of 1% starch solution. The content of the flask was titrated with 0.1 N sodium thiosulphate with constant and vigorous shaking until the blue colour just disappeared. A blank test was also carried out in a similar method. The volume of the 0.1N sodium thiosulphate required would be recorded as peroxide value using the below equation:

$$\mathrm{PV} = \frac{(\mathrm{Va} - \mathrm{Vb})\ \mathrm{N\ X\ 100}}{W} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots.(3)$$

Where:

Va = Volume of sodium thiosulphate solution used in the titration

Vb = Volume of sodium thiosulphate solution used in the blank test

W = Weight of the sample in grams

N = Normality of sodium thiosulphate.

5. Saponification Value of the Oil

Determination of the Saponification value of the oil was carried out according to the AOAC (2008): 1 g of the oil sample was measured and transferred into 200ml conical flask, 25ml of 0.1N alcoholic KOH solution was added. The flask is then boiled under reflux for one hour with frequent agitation, 1ml of phenolphthalein indicator was added while the solution is still hot, and the excess alkali was titrated with 0.5N HCl, and the volume of HCl required to complete the titration was recorded. The same procedure was repeated for the blank and the volume of HCl required to complete the titration was also recorded. Saponification value was calculated using the equation

$$\text{Saponification value} = \frac{(\mathrm{b} - \mathrm{a}) \times 0.02805}{S} \times 1000 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots.4$$

Where;

a = Volume of HCl used to titrate the sample

b = Volume of HCl used to titrate the blank

S = Weight of oil in gram.

6. Free Fatty Acid (FFA) of the Oil

The method of Afolabi, (2008) adopted by Ogala *et al*., (2018) was employed, 2g of the oil was weighed in to 250mL conical flask and warmed, 2.5mL of methanol was added with constant stirring, followed by 3 drops of phenolphthalein indicator, and this is then titrated against 0.14 M potassium hydroxide solution with vigorous shaking until a permanent light pink color was observed, which persisted for 1 min. The end point was recorded, and the free fatty acid value was calculated using equation bellow:

$$\% \text{ Free fatty acid} = \frac{\text{Titre V} \times 28.2 \times \text{M}}{\text{Wight of sample}} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(5)$$

Where;

V = Volume of potassium hydroxide used

M = Molarity of potassium hydroxide used

W = Weight of the Sample.

2.3. Trans-esterification (Biodiesel Production)

The method of Mansir *et al*., (2017) was used for the transesterification. This was carried out in a 500 ml conical flask equipped with a reflux condenser and a magnetic stirrer. The sample was measured and transferred in to the reaction flask heated at 50°C while stirring at 850 rpm. Methanol / catalyst were added at molar ratio of methanol: oil was 6:1, reaction temperature 65°C and reaction time 4hours. After the complete reaction, the solid catalyst was filtered out using a filter paper. The filtrate was transferred to a separating funnel and left overnight. The top layer was collected and washed with hot distilled water until clear and the FAME was dried at 110°C, and the percentage yield was calculated using the following equation:

$$\text{FAME Yield } (\%) = \frac{\text{Weight of FAME produced}}{\text{Weight of oil used}} X\ 100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots .6$$

2.4. Biodiesel Analysis

1. Determination of Flash Point

The ASTM D93 method was used as adopted by Ravikumar *et al.,* (2011). The biodiesel test sample was poured into the test cup until it reached the filling point. The assembly was put into the apparatus after the test cover was put on the test cup. The IP 15^{0}C thermometer was placed inside the holder. The lit in the test flame was ignited. After turning on the electric heater, the temperature was raised to 5 to 6 degrees Celsius every minute. The sample was agitated at a speed of 100-120 rpm. The test flame was applied at an interval of 2 ^{0}C raise in temperature after reaching the temperature of the sample to 90^{0}C. The test flame was applied by lowering the test flame using the shutter operator on the test cover. The temperature at which the test flame, when lowered into the vapor space, caused a blue halo and then a distinct flash in the interior of the test cup was recorded as the flash point.

2. Determination of Cloud Point and Pour Point

The cloud and pour points were determined using the method of Ferdous *et al.,* (2013) the Haeckel Caton Changsha cloud and pour point analyzer tool; KD R1072 following ASTM D4539 method. For cloud point as reported by Ferdous *et al.,* (2013): 50 cm^3 sample was placed in the sample holder fitted with a thermometer. As the machine is set on the temperature begins to drop. The temperature at which the first crystal is formed was noted as the cloud point. For the pour point, the temperature of the machine starts to increase and the temperature at which the sample cannot move is noted as the pour point.

3. Determination of Kinematic Viscosity

Calibrated Cannon -Fenske viscometer No.100 with nominal constant 0.015.as per ASTM D44 adopted by Kumar *et al*., (2013).

4. Determination of Density

The biodiesel density was determined by pycnometry method of Kumar *et al.*, (2013): empty weight of pycnometer (W1g) was recorded then filled with the distilled water and the weight (W2 g) was recorded. The pycnometer was emptied and dried, and filled with the sample and the weight (W3 g) was determined and the final density was calculated as follows

$$\text{Density (g)} = \frac{W3-W1}{W2-W1} \times 100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots..(7)$$

Where;
W1 = Empty weight of pycnometer
W2 = Empty weight of pycnometer +Water
W3 = Empty weight of pycnometer+ Sample

2.5. GC-MS Analysis of Biodiesel (FAME)

Fatty acid methyl esters (FAME) were analyzed using SHIMADZU Gas chromatography Mass Spectrophotometer (GC-MS). A column with dimensions of 30 m x 0.32 mm and a 0.2 µm film thickness was used. The auto-injector was operated in the split less mode at 250°C, helium gas was used as a carrier gas at pressure of 1633kPa and a flow rate of 9.5 mL/min. 1µL of sample biodiesel was injected using the auto injector, the oven temperature is held at 100°C for 1 min and then increased to 250°C at a rate of 10°C/min then increased to 270°C at 8°C/min according to the method of Whetstine Courtney et al. (2020).

3. Result and discussion

3.1. Percentage Biodiesel Yield

$$\text{Biodiesel yield \%} = \frac{\text{Weight of biodiesel produced}}{\text{Weight of oil used!}} \times 100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots.(8)$$

$$\text{Biodiesel yield (\%)} = \frac{100}{123} \times 100 = 82$$

3.2. Physicochemical properties of used cooking oil

The physicochemical properties of used cooking oil was determined and presented in table 1, below

Table 1. Physico-chemical Analysis of Used Cooking Oil

S/N	Parameters	Result	ASTM Standard
1	Colour	Brown	
2	Net content (g)	331.38±0.02	
3	Matter Volatile (%)	0.40±0.03	
4	Acid Value mg/kg	0.269±0.001	4
5	Peroxide Value mg/kg	6.80±0.02	10 mEq/g
6	FFA (%)	0.14±0.01	5.78 – 7.28
7	Saponification Value g/KOH	196.6 ±0.1	195-205
8	Moisture Content (%)	1.29±0.01	<0.05%

Physicochemical analysis of used cooking oil was presented in **Table 1,** showing that, colour is Brown liquid. The low acid value of 0.269mg/kg was generated as shown in table 1: is an indication of low saponification value and less or no soap formation during process of biodiesel production. Warra *et al.,* (2011) reported an Acid value of 2.39 ± 0.065 mgKOH/g for Jatropha oil, 0.81 ± 0.01 mgKOH for cotton seed and 2.39 ± 0.065 for castor seed oil which are higher in comparisons with the used cooking oil from this analysis. Mazumdar *et al.,* (2012) also reported an acid value of 0.26mg/KOH for Jatropha curcas which was in line with the reported one from this research, all aim at the production of biodiesel.

The chemical molecules formed in used cooking oil as a result of change in colour during titrimetric experiment are known as free fatty acids. The maximum value required for biodiesel production according to the ASTM was 0.05 mgKOH, higher free fatty acid content will lead to increase viscosity and saponification resulting to low biodiesel yield (Yaakob *et al.,* 2013). Alias *et al.,* (2018) reported series of free fatty acids analyzed from different wastes cooking oils/used cooking oils ranging from 1.05- 9.05 mgKOH in which the initial free fatty acid content of 1.05 mgKOH has enormous influence in transesterification reaction of catalysts because when the concentration is high, the production of biodiesel is reduced resulting in the formation of soap. The higher the free fatty acid the higher the soap formation, conclusively the result of this work indicated low free fatty acid content of 0.14mg/KOH thus expecting higher yield of biodiesel without undergoing saponification reaction. Saponification value occurs because of the presence of free fatty acids, the analysis showed saponification valued of 196mg/KOH/g which is lower than the values reported by Alias *et al.,* (2018) from different waste cooking oils from different catering, indicating that free fatty acid level is higher as reported by Ullah *et al.,* (2014) and higher saponification value affect the production of biodiesel. The time taking for the cooking process, the more the accumulation of volatile and non-volatile compounds in the oil and thus the darker the oil colour the higher the FFA content and acid value content (Wahab *et al.,* 2015)

Table 1 showed a moisture content values of 1.29% which is higher than the ASTM D6751 standard for biodiesel production 0.05% as reported by Glycerintraders (2007). Majority of samples from literature showed values greater than ASTM. Presence of water in the oils is known as hydrolysis and lesser the production of biodiesel while resulting to higher catalyst consumption, soap generation and larger viscosity (Yaakob *et al.,* 2013). In addition, gel and foam formation prevent separation of the biodiesel and glycerol (Lupea, 2004). The peroxide value of 6.80mEq/kg was recorded from this research. Popa *et al.,* (2017) reported the value of the peroxide parameter exceeding 10meq/kg only after 45 days for the daylight exposed sunflower oil. The sunflower oil examined in his study showed large variations in Peroxide Value between exposure on daylight and darkness after 60 days up to 7.09meq/kg, which was within the range of the current studies. Generally, the oxidation rate increase with the temperature. While the temperature is increasing, the changes of the partial pressure of the oxygen had less influence on the reaction rate of oxidation, because the oxygen is becoming less soluble in lipids and water. The Peroxide Value is applicable for monitoring the formation of peroxides in the early stages of oxidation (Abramovic *et al.,* 2005).

3.3. Biodiesel Properties

The fuel Properties of the biodiesel were analyzed, and the generated results are presented in Table 2. The fuel Properties of biodiesel generated from the produced biodiesel showed a specific gravity of 0.86 g/cm^3 which is in conformance with the ASTM D6751 standard (0.86 – 0.90). Onyezeka *et al.,* (2020) reported 0.862 ±0.077 – 0.876 ±0.084 range from various Trans' esterified waste cooking oils. As reported by Tat and Van Gerpen (2000), the composition of fatty acid composites determines the specific gravity of biodiesel, and biodiesel density varies with the purity and composition of fatty acid compounds. It's a vital fuel property because injection systems, pumps, and injectors must supply a precisely regulated amount of gasoline to ensure optimum combustion (Dzida & Prusakiewicz, 2008). The specific gravity values for ideal air-to-fuel ratios must be maintained within acceptable limits for complete combustion.

Table 2. Biodiesel Properties

S/N	Properties	Biodiesel values	Standard Biodiesel ASTM D6751
1	Density (g/cm3)	0.86±0.02	0.88
2	Viscosity @40 (^{0}C) mm/s	0.97±0.01	1.9 to 8
3	Specific Gravity@60 kg/l	0.87±0.04	0.88
4	Acid Value (mgKOH/g)	11.2±0.1	0.80
5	Pour point (^{0}C)	-11.4±0.1	-15 to 10
6	Cloud Point (^{0}C)	-2.7±0.1	-3 to 12
7	Flash point (^{0}C)	156.7±0.05	130-170

Flash point is the measure of flammability of the fuel, and the values of flash point gotten from this research is 156 ^{0}C as indicated in Table 2, and fall within the range of (range; 131 – 207°C) reported by Onyezeka *et al.,* (2020) from different waste-praying oil and showed conformance to the ASTM D6751 of (130°C - 170°C). Based on this study, the flash point of the produced biodiesel is within the ASTM D6751 standard and above the minimum limit of 130 °C, and therefore poses no risk of fire outbreaks in the event of an accident.

The cloud point is the temperature of the fuel at which small, solid crystals can be witnessed as the fuel cools. the produced biodiesel has a cloud point of -2.8^{0}C and pour point of -11.5^{0}C this is less than the reported values by Tunio *et al.,* (2016) who reported a pour point of -12 and a cloud point -10 from Castor Seeds oil respectively, the result also is below the standard of ASTM D-97 -15 to +5^{0}C and ASTM D-2500 -3 to -12^{0}C of biodiesel standard

Kinematic Viscosity, which is a numerical value, indicates how much a liquid material resists flowing or how much it resists being sheared, indicating that thicker, harder-to-flow liquid materials are produced by higher viscosities. More importantly, kinematic viscosity is a vital parameter that can influence fuel spray atomization, flow capacity, and spray penetration. In this research th,e recorded viscosity of the produced biodiesel is 0.96, which was within the range of 0.79 and 1.38 reported by Abati *et al.,* (2024). The finding shows a less viscous biodiesel compared with 4.7mm^2 reported by Usman and Rufai *et al.,* (2020). The viscosity of the FAME was lower that of the ASTM standard.

This could, however, be attributed to the presence of highly volatile components

3.4 GC-MS Analysis of Biodiesel

For primary identification and confirmation Fatty Acid Methyl Esters present in the produced biodiesel was analyzed using GC-MS and the result was presented in **Table 3** below

Table 3. GC-MS Result for biodiesel produced from used cooking oil

S/N	Retention time/Min	Area%	Synthetic name	Common name	Molecular formula	M/W
1	18.569	1.21	Hexadecanoic acid, methyl ester or palmitic acid methyl ester	Methyl palmitate	$C_{17}H_{34}O_2$	270
2	19.001	5.39	Hexadecanoic acid		$C_{16}H_{32}O_2$	256
3	20.798	3.26	9,12-Octadecadienoic acid (Z,Z)-, methyl ester	Linoleic acid methyl ester	$C_{19}H_{34}O_2$	294
4	20.850	0.24	9,12,15-Octadecatrienoic acid, methyl ester,	Methyl linoleate	$C_{19}H_{32}O_2$	292
5	20.917	2.99	9-Octadecenoic acid, methyl ester,	Methyl oleate	$C_{19}H_{36}O_2$	296
6	21.295	2.13	Methyl stearate	Methyl stearate	$C_{19}H_{38}O_2$	298
7	23.227	75.97	9-Octadecenoic acid, 12-hydroxy-, methyl ester	Methyl ricinoleate	$C_{19}H_{36}O_3$	312
8	25.527	0.35	9-Hexadecenoic acid, methyl ester	Methyl palmitelaidate	$C_{17}H_{32}O_2$	268

GC-MS analysis produced the chromatogram, where the mass spectrometer detects compounds that appeared in the form of peaks as shown in Table 3, The results presented different methyl ester peaks as displaced in table 3 with 9-Octadecenoic acid, 12-hydroxy-, methyl ester as the maximum with higher peak percentage area of 75.97% at retention time of 20.917 this is an indication of its more availability than any other detected compounds in the produced biodiesel. The result was in line with the findings of Chellappan & Nair, (2018), who reported a series of fatty acid methyl ester from esterification – transesterification of *Pongamia pinnatta* oil (PPO) from sawdust Biochar-based catalyst. His findings are consistent with current research on cooking oil. Whetstine, (2020) reported 9,12-Octadecadienoic acid (Z, Z)-, methyl ester commonly known as Linoleic acid methyl ester, Hexadecanoic acid, methyl ester or palmitic acid methyl ester also known as Methyl palmitate from transestericationof cooked and un cooked vegetable oils, his finding was in line with the current analysis using Used cooking oil also *Maulidiyah et al.,* (2021) reported Hexadecanoic acid, 9,12-

Octadecadienoic acid (Z,Z)-, methyl ester/ Linoleic acid methyl ester from two step Esterification-transesterification of crude palm oil using sulfuric acid and K_2O Catalysts which was in conformity of the present work. The result is also within the ASTM D6751 standard; the FAME consists of both saturated and unsaturated esters ranging from C14 to C24, typically used for GC analysis to verify purity and conformity. Key FAME components included palmitic, stearic, oleic, linoleic, methyl stearate, and linolenic methyl esters, among others.

Conclusion

The analysis of both raw material (Used cooking oil) and the produced biodiesel were in conformity with the ASTM standard and some established research conducted using different waste cooking oils from different source. The percentage biodiesel yield is an indication of higher quantity while the analysis prove high quality of the biodiesel. Utilization of discarded used cooking oil will be a promising coast effective alternative for the production of biodiesel in commercial quantity there by limiting the environmental pollution and enhance waste to wealth generation

Conflict of Interest

The authors declared no conflict of interest

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