A Dissertation thesis entitled

"Preparation of Biodiesel from Waste Cooking Oil Catalyzed by Sodium Hydroxide"

Submitted in partial fulfilment of the requirements For the award of the degree of

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IN

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Submitted By

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Dedicated to

My Beloved Family

Without their love, support and constant encouragement, this would not have been possible

DECLARATION

We undersigned, hereby declare that the work assimilated in the dissertation thesis entitled **"Preparation of Biodiesel from Waste Cooking Oil Catalyzed by Sodium Hydroxide"** has been carried out by us at Faculty of Science, Department of Industrial Chemistry, Atmiya University, Rajkot, Gujarat, India, under the supervision and Guidance of **Er. Dhaval A. Tank, Assistant Professor, Faculty of Science, Department of Industrial Chemistry, Atmiya University, Rajkot, Gujarat, India.**

To the best of our knowledge and belief, the work included in this thesis isquite original and has not submitted to any other Institution or University for the award of any degree either in this or any other form.

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ABSTRACT

One alternative energy source that required to be developed in India is biodiesel. Used cooking oil is raw material for making biodiesel. The biodiesel was made from used cooking oil and methanol using the transesterification method with NaOH catalyst. The production of biodiesel from used cooking oil Start with mixing raw materials of used cooking oil collected into one. The mixture is then precipitated for 24 hours. The transesterification process was carried out by mixing NaOH (2% of oil weight) with methanol (ratio of methanol: oil 6: 1) at a temperature of 65 $^{\circ}$ C. After the temperature is reached, the methanol and NaOH solution is added slowly while stirring, with a variation of time 30, 45, 60 ,75 and 90 minutes. Biodiesel purification is done by washing using hot water (temperature 70 °C) washing. Characteristics of biodiesel based on the best conditions for density 0.9104 g / mL, viscosity 0.589 cm²/s, acid value 6.1 mg NaOH and flash point 165 °C.

Keywords: Biodiesel, NaOH catalyst, Transesterification.

1.0 INTRODUCTION

Biofuels have become more attractive in recent years as an alternative fuel because of its renewability and positive environmental benefits. It burns up to 75% cleaner than fossil fuels. It substantially reduces unburned hydrocarbons, carbon monoxide and particulate matter in exhaust fumes which are responsible for acid rain, global warming, and ozone layer depletion. The biodiesel synthesis from used cooking oil have been carried out. The synthesized biodiesel from used cooking oil with the trans-esterification process. The biodiesel processing process that uses two stages, first esterification and transesterification require double consumption of methanol. The addition of catalyst can increase conversion percentage of biodiesel produced. In this biodiesel was made from used cooking oil and methanol using the transesterification method using NaOH catalyst. As for the selection of used cooking oil as a raw material for making biodiesel, in addition to being easy to obtain and the price is low also to utilize used cooking oil which is usually disposed of to be a useful product [1].

Rakopoulos, Faupel and Kurki (2002); Mohammed et al. (2002) gave some of negative aspects encountered while the use of biodiesel in compression ignition engine. They found that biodiesel fuels have better viscosity [2], better pour point, lower heating price and volatility than petroleum diesel gasoline. Also, the oxidation balance of biodiesel fuels becomes discovered to be lower and hygroscopic. As solvents they reason corrosion of component, attacking a few plastic substances used for seals, hoses, paints and coatings.

They confirmed expanded dilution and polymerization of engine sump oil, for that reason requiring greater common oil changes. Further more, using vegetable oil for gasoline reasons opposition with using vegetable oil for meals and it has better in keeping with gallon fee than petroleum diesel with inside the present-day market. Ahouissoussi (1995) has referred to that despite the fact that biodiesel have better overall expenses than diesel gasoline, they have got the capability to compete with compressed natural gas and methanol as fuels for city transits buses. Also, it's far noteworthy that Krawczyk (1996) diagnosed biodiesel as a likely alternative to fossil's fuels because the world's number one power source. Biodiesel will be acquired from an array of agricultural produce which includes soybean, groundnut [3].

As the use of biodiesel increases, it is important to meet the standards for biodiesel as a fuel. Therefore, the nature of the feedstock in the production of biodiesel needs to be clearly identified, and the complete characterization of the produced biodiesel has been an important aspect of biodiesel research. Waste cooking oil is a promising alternative for producing biodiesel because it is a cheaper raw material that also avoids the cost of waste product disposaland treatment. Besides, it reduces the need to use land for biodiesel-producing crops. But these used frying oils have different properties from those of refined and crude vegetable oils. The presence of heat and water accelerates the hydrolysis of triglycerides and increases the content of free fatty acids (FFA) in the oil. The optimum conditions for biodiesel production (methanol/oils ratio and concentration of catalyst) are inconsistent. They strongly depend on the properties of WCO. The most common way to produce biodiesel is by transesterification which refers to a catalyzed chemical reaction involving vegetable oil and an alcohol to yield fatty acid alkyl biodiesel and glycerol.

Figure.1 Reaction of FAME (Bio-diesel)

2.0 LITERATURE REVIEW

2.0.1 BIODIESEL

Biofuels are alcohols, esters and different natural compounds created from biomasswhich includes herbaceous and woody plants, residues from agriculture and forestry, and a few agro- industrial wastes which includes waste from the meals industry.

The term biomass refers to any substance which may be received via way of means of photosynthesis. Most plant life make use of sun strength to create sugars, beginning from easy materials along with water and carbon dioxide. This strength saved in molecules of glucose, starch, oil, etc. Biofuels may want to consist of ethanol, biodiesel, and bio methanol among others. Most advanced and used are bioethanol and biodiesel. The term biomass refers to any substance which may be received via way of means of photosynthesis. Most plant life make use of sun strength to create sugars, beginning from easy materials along with water and carbon dioxide. This strength is saved in molecules of glucose, starch, oil, etc. Biofuels may want to consist of ethanol, biodiesel, and bio methanol among others. Most advanced and used are bioethanol and biodiesel [4].

2.0.2 ADVANTAGES AND DISADVANTAGES IN THE USE OF BIODIESEL FUEL:

Advantages:

- Biodiesel is a renewable resource, biodegradable and nontoxic.
- Lower emissions of carbon monoxide, unburnt hydrocarbons, and carbonparticles, contributing to the reduction of greenhouse gases.
- It may be used without delay in direct injection engines without uniqueadaptations.
- The engine overall performance improves while the usage of biodiesel asopposed to fossil diesel, because of its high lubricity.
- It is safer, as it has a high flash point.
- The gases from combustion of biodiesel do now no longer include sulphurdioxide, essential purpose of acid rain.
- Promotion of nearby and regional development.

Disadvantages:

- Increased emissions of NO_x and aldehydes.
- Because it is a great solvent, it is able to dissolve sediments withinside the engine gasoline device and purpose filter plugging at some point of its first use in engines generally running with fossil diesel. So it's far recommended to clean the gasoline device while switching from diesel to biodiesel.
- \bullet Below 0 °C, the biodiesel has a tendency to freeze causing engine deposits.
- Biodiesel has approximately 8% much less power consistent with liter thandiesel. The energy and gasoline consumption of the engine is decreased.
- The feed stocks for biodiesel require massive regions for cultivation and willhave an effect on the food prices.
- Biodiesel has a tendency to oxidize greater rapidly than diesel.
- High prices of production.

2.0.3 TRANSESTERIFICATION:

Biodiesel is commonly received through transesterification of oils or fat through reacting a shortchain alcohol, like methanol, withinside the presence of a heterogeneous catalyst. The maximum common raw substances for manufacturing of biodiesel are vegetable oils from sunflower, soybean, palm oil, groundnut oil and other [5].

The transesterfication reaction specifically depends on the character of the feedstock, catalyst concentration, the molar ratio alcohol-oil, temperature, agitationrate, pressure and reaction time in addition to moisture content material and quantity of unfastened fatty acids.

In the transesterfication reaction, one mole of triglyceride is reacted with 3 moles of alcohol to shape one mole of glycerol and 3 moles of the respective fatty acid alkyl ester in a chain of 3 reversible reactions in which the triglycerides are transformed to diglycerides after which to monoglycerides. Glycerol is produced asbyproduct. After the reaction the combination forms two phases: the glycerin– rich–segment and ethyl esters–rich–segment. The phases may be separated via wayof means of decantation or centrifuging [6].

There are numerous kinds of catalysts for transesterfication. The maximum normally used catalyst for manufacturing of biodiesel is the homogeneous catalysis. The homogeneous catalysis used for biodiesel manufacturing may be divided particularly in alkaline and acidic catalysts. Heterogeneous catalysts (solid phase) is likewise used. Another exciting alternative can be to use lipases as enzymatic catalysts.

figure 2. NaOH Transesterification **figure 3.** KOH Transestrification

2.0.4 ACID CATALYSIS:

Acid catalysts consist of sulphuric, phosphoric and hydrochloric acid. The acid catalysts are encouraged for oils with a excessive content material of free fatty acids which includes waste oil. The acid catalysis is also used to pre-deal with a few oils as a step previous to the fundamental transesterfication. In this example the free fatty acids are esterified tillthe quantity of FFA is decrease than 0.5 %. Then, extra methanol and26 base catalysts are brought to complete the response. However, the response is slow and requires higher temperatures and pressures, and also needs higher quantities of alcohol.

2.0.5 HETEROGENEOUS CATALYSIS:

Heterogeneous catalysts act in a unique phase than the reactants. They do now no longer dissolve in alcohol, oil or biodiesel. They are in solid state and are without difficulty recoverable through decantation or filtration at the stop of the reaction. Transesterfication with homogeneous catalysis has offers issues such as formation of soaps and want for purification and disposal of byproducts of the reaction, the remedy of waste effluents and withinside the case of catalysts issues associated with corrosion [7].

Using heterogeneous catalysis most effective the evaporation of methanol is needed and no waste effluents are produced, further it's also received naturalglycerol. However, their high price because of the need of increasedtemperature and pressure conditions as well as massive volumes of alcohol, isn't always still permitting an industrial manufacturing of biodiesel the use of heterogeneous catalyst.

2.0.6 PRESENCE OF FREE FATTY ACIDS (FFA):

A fatty acid is an natural molecule such as an extended aliphatic chain of an even range of carbon atoms. At the end of the chain there is a carboxyl group. A fatty acid is a carboxylic acid of excessive molecular weight.

The content material of free fatty acids and moisture are key parameters that decide the feasibility of the system of transesterfication. The content material of free fatty acids determines the catalytic path to be applied for the transesterfication. For a fundamental catalyst the quantity of FFA within side the oil must be much less than3% due to the fact the high acidity of the oil reasons a low biodiesel yield. When oil has greater than 5% FFA, it can't be transesterified through alkaline catalysis, because of the formation of soaps which inhibit the separation ofglycerol and biodiesel [8]. Also, formation of emulsions for the duration of washing takes place. The presence of FFA causes formation of cleaning soap and hampers the separation of the biodiesel and glycerol. Besides, a high quantity of catalyst is consumed and the catalytic performance is significantly reduced.

One trouble of this technique is the presence of water. The accumulation of water produced for the duration of the esterification can stop the reaction earlier than it's far complete.

2.0.7 ALCOHOLS:

The alcohols which can be historically used in the transesterification are of short- chain, specially methanol and ethanol. Other alcohols may be used consisting of propanol, isopropanol, butanol and pentanol [9], however those alcohols are a lot greater sensitive to water contamination. They also are greater high-priced and large molar ratios and higher temperatures are required. Methanol has a lower priceand provides some chemical and physical benefits over different alcohols, because the high charge of response with triglycerides. It is important to say the high toxicity of methanol, flammability, and the reality that methanol is specially constructed from nonrenewable fossil fuels such as methane gas [10].

Ethanol, however, comes from renewable raw materials: sugar cane, sugar beet, greens and different cellulosic. So ethanol is of specific interest particularly due to the fact it's far much less high-priced than methanol in a few regions. It is also feasible to obtain through dry distillation of wood,however this method isn't relevant to huge scale. However, the ethyl ester formation is relatively more tough than that of methyl ester. The formation of a stable emulsion for the duration of the ethanolysis is a problem. Ethanol and methanol do now no longer dissolve with the triglycerides at room temperature and the mixture have to be agitated automatically to allow diffusion. During the reaction, typically an emulsion is formed. In the methanolysis this emulsion breaks down rapidly forming a decrease glycerol-rich layer and a higherlayer rich in methyl ester [11].

However in ethanolysis this emulsion is extra stable and complicates the separation and purification of the ethyl esters. The emulsion is precipitated partially through the formation of monoglycerides and diglycerides intermediates containing each polar hydroxyl groups and nonpolar hydrocarbon chains.

The stoichiometric ratio for transesterification requires three moles of alcohol and one mole of triglyceride to supply three moles of esters and one mole of glycerol. The transesterification is an equilibrium reaction requiring an extra of alcohol to drive the reaction to the right. The extra of methanol can be recovered after the reaction [12]. For maximum conversion it's far recommended to apply a molar ratio of 6:1. However this ratio will rely on the kind of feedstock used. An excessive molar ratio alcohol/oil impacts the separation of glycerol because of accelerated solubility. When the glycerol is maintained in the solution the opposite reaction movements to the left. The glycerol can react returned into a monoglycerides and diglycerides. The product will include monoglycerides and diglycerides, which crystallize very easily withinside the biodiesel and may cause clogging of filters and different engine problems.

2.0.8 TEMPERATURE AND PRESSURE:

The transesterification may be carried out at atmospheric pressure and at distinct temperatures depending on the used feedstock used. The temperature impacts the yield of received biodiesel. Often the transesterificationis finished at a temperature close to the boiling point of alcohol (at 60 ° C while theusage of methanol [13].

Biodiesel also can be produced through methods at high pressures and high temperatures (200-250 ° C) the use of huge quantities of methanol and without pretreatment of the raw material. Methods the usage of methanol at supercritical situations at 350 ° C and 43 MPa had been also used. However, the prices of these methods are high [14].

2.0.9 REACTION TIME:

Freedman et al (1986) found the increase in fatty acid esters conversion when there's an increase in reaction time. The reaction is slow at the start due to mixing and dispersion of alcohol and oil. After that the reaction proceeds very fast. However the most ester conversion became completed within < 2hr similarly increase in reaction time does now no longer increase the yield product i.e. biodiesel/mono alkyl ester. Besides, longer reaction time results in the reduction of end product (biodiesel) because of the reversible reaction of transesterification resulting in lack of esters in addition to cleaning soap formation.

2.0.10 PHASE SEPARATION:

After the transesterification reaction, the reaction mixture (combination) is separated in two phases. The lower phase, the glycerol phase, may be separated through decantation or centrifugation.

Some ways to promote phase separation,

- The addition of water, glycerol.
- Cool down the mixture.
- Application of excessive voltage to facilitate separation of the polar phase (glycerol and residues of alcohol and catalyst) from the nonpolar phase (esters and alcohol).

Figure 4. Phase Separation

2.0.11 PURIFICATION OF BIODIESEL:

After separation of phases, each the biodiesel and glycerol phase must be washed and purified. In the industry (enterprise) the methanol is recovered through heating the ester phase. Glycerol and traces of catalyst may be eliminated from the biodiesel segment through washing with acidified water with a purpose to neutralize the traces of catalyst.

Figure 5. Biodiesel **Figure 6**. Biodiesel Washing.

2.1 MATERIALS AND METHOD

2.1.0 MATERIALS:

Waste Cooking oil was obtained as an open-handed gift from Shree fast food, Rajkot, Gujarat, India. Methanol less crude glycerol was produced using current biodiesel synthesisprocedure.

Figure 7. Waste Cooking Oil

3.0 EXPERIMENTAL PROCEDURES

3.1.0 PREPARATION OF BIODIESEL:

The experimental part of the work includes the production of biodiesel fromWaste cooking oil. Methanol has been used in the transesterfication. NaOH has been selected as catalyst. The reaction takes place in an flat bottom flask on magnetic stirrer in a continuous process. The flask is placed on magnetic stirrer maintained at a desired temperature by one electrical heater.

The alcohol (methanol or ethanol) and catalyst NaOH are mixed preferably at the temperature close to the main reaction temperature to avoid significant temperature drop in reactor. Palm oil which also is maintained at the constant temperature - is added to the mixture of alcohol-catalyst. After reaction finished the product is poured into a funnel to obtain a separated two phases. Upper phase isbiodiesel and downer is glycerol.

The influence of ratio alcohol to oil, the amount of catalyst, the reaction time, the rate of mixing, the reaction temperature on the yield of produced biodiesel is studied.

The performance of the experiments includes the following steps:

- Measure quickly the catalysts NaOH on an electronic scale.
- Mix the alcohol and the catalysts in a flat bottom flask under stirring to ensure complete solution.
- The waste cooking oil is preheated to the reaction temperature in a electric heater. Then the oil is added to the alcohol/catalyst solution in the flask (FBF). The reaction is maintained for 1-2 hours under stirring at the set temperature $(65 \degree C)$

The solution is poured into a separation funnel since the reaction is completed, i.e. two large phases are formed. The biodiesel product at the topand the by-product glycerol in the bottom of the funnel.

 Biodiesel and glycerol have different specific gravities and separated using gravity decanter. The biodiesel was washed with hot water till no more alkaliwas found. Recovered alkali could be recycled while glycerol was the byproduct of the process.

Figure 8. Biodiesel reaction process

3.2.1 TRANSESTERIFICATION REACTION:

Reaction 2. Transesterification Reaction

3.2.2 MECHANISM:

STEP-1:

Monoglyceride

Reaction 3. Transesterfication Reaction

4.0 ESTERIFICATION

Esterification is reaction wherein reactant (alcohol and acid) from a ester as areaction product. Ester is common in organic materials.

ESTERIFICATION REACTION:

Reaction 4. Esterification Reaction& Mechanisms

5.0 BIODIESEL YIELD ESTIMATION (%)

The biodiesel yield (%) has been calculated using acid value estimation of Palm oil and biodiesel as given in equation[15] .

% FFA Conversion (ppm) = $\frac{\text{AVi}-\text{AVF}}{\text{AVi}-\text{AVF}}$ × 100 AVi

Where: FFA = Free Fatty Acid AVi = Acid value initial

 $AVF = Acid value final$

• Calculation for biodiesel yield% $yield \% = \frac{weight \ of \ methyl \ est \ (gms)}{weight \ of \ oil \ (gms)} \ \times 100$

6.0 RESULT DISCUSSION

Sr. No.	O: M Molar ratio	NaOH $\%$ (w/w)	Volume Oil ml	Reaction Time (t)	Yield
1.	1:6	$\overline{2}$	100	30	50%
2.	1:6	$\overline{2}$	100	45	63%
3.	1:6	2	100	60	66.33%
4.	1:6	2	100	75	75.46%
5.	1:6	2	100	90	79.86%

Table-1: Result of % (Yield of Biodiesel with varying reaction parameters)

Table-2: Result of % (Yield of Biodiesel with varying reaction parameters)

Sr. No.	O: M Molar ratio	KOH $\%$ (w/w)	Volume Oil ml	Reaction Time (t)	Yield
1.	1:6	2	100	30	52%
2.	1:6	$\overline{2}$	100	45	59.23%
3.	1:6	$\overline{2}$	100	60	65%
4.	1:6	2	100	75	76%
5.	1:6	2	100	90	82.30%

It has been found from the experimental result; the 50% biodiesel yield has been obtained with 1:6 oil molar ratio, 2% (w/w) NaOH catalyst, 65°C reaction temperature and 0.5hr reaction time. However, it has also been obtained from the experimental result, the 52% biodiesel yield has been obtained with 1:6 oil molar ratio, 2% (w/w) KOH catalyst, 65°C reaction temperature and 0.5hr reaction timer. However, It has been found from the experimental result; the 63% biodiesel yield has been obtained with 1:6 oil molar ratio, 2% (w/w) NaOH catalyst, 65^oC reaction temperature and 0.7hr reaction time. It has been found from the experimental result; the 59.23% biodiesel yield It has been found from the experimental result in KOH catalyst; the 66.33% biodiesel yield has been obtained with 1:6 oil molar ratio, 2% (w/w) NaOH catalyst, 65°C reaction temperature and 1hr reaction time. It has been found from the experimental result; the 65% biodiesel yield has been obtained with 1:6 oil molar ratio, 2% (w/w) KOH catalyst, 65°C reaction temperature and 1hr reaction time.It has been found from the experimental result; the 75.46% biodiesel yield has been obtained with 1:6 oil molar ratio, 2% (w/w) NaOH catalyst, 65°C reaction temperature and 1.3hr reaction time. It has been found from the experimental result;the 76% biodiesel yield has been obtained with 1:6 oil molar ratio, 2% (w/w) KOH catalyst, 65°C reaction temperature and 1.3hr reaction time. It has been found from the experimental result; the 79.86% biodiesel yield has been obtained with 1:6 oil molar ratio, 2% (w/w) NaOH catalyst, 65°C reaction temperature and 1.5hr reaction time. the 82.30% biodiesel yield has been obtained with 1:6 oil molar ratio, 2% (w/w) K OH catalyst, 65°C reaction temperature and 1.5hr reaction time.

The research was to access biodiesel production from waste cooking oil in the presence of a homogenous catalyst NaOH & KOH. Three factors were investigated $-$ oil-to- methanol ratio, catalyst loading and temperature – in order to optimize the process. Optimal reaction conditions were obtained at oil to methanol molar ratio (1:6), catalyst loading $(2%)$ and temperature (60 \degree C) which gives 79% yield of biodiesel. Hence, in order to shift the reaction towards formation of FAME, with respect to oil it was assumed that the catalyst was used in sufficient amount. It was observed that the catalyst NaOH showed an excellent activity in mediating the transesterification of waste cooking oil containing FFA to biodiesel production, which is compare to KOH catalyst give more yield.

7.0 SPECTRAL CHARACTERIZATION

Figure 9. 1H NMR of Waste Cooking Oil

the 1H NMR spectra of the refined and used oils are also similar, as shown in figure 9. the two spectra clearly indicate signals at 4.165–4.187 ppm (och2; sn-1, sn-3), 5.104 ppm (och; sn-2) due to the glycerol fraction, 5.199–5.318 ppm (ch=ch), 2.135–2.503 ppm (bisallylic, ch=ch–ch2–ch=ch), 1.96–1.982ppm $(ch2c=o)$, 1.9–2.1 ppm (allylic, ch2–ch=ch–) and 1.47 ppm (ch2–ch2c=o); 1.232 ppm and 1.166 ppm correspond to the alkyl group (ch2)n, and those at 0.77–0.85 ppm are characteristic of terminal ch3 triglycerides comprising both a saturated and unsaturated fatty acid chain.

Figure 10. 1H NMR of Waste Cooking Oil Biodiesel

The waste cooking oil biodiesel was characterized by the 1H NMR. The 1H NMR spectrum of the waste cooking oil biodiesel is shown in Figure 10. To evaluate the conversion of oil into biodiesel, a new signal appeared at 3.518 ppm. This signal indicated the formation of biodiesel, because the peak well matched the range of proton from the methyl ester group $(-\text{cooch3})$. to evaluate the conversion of methyl ester, we used $-\text{ch2}$ protons at 1.64 ppm. these two peaks confirmed the conversion of oil into biodiesel. Other observed peaks were at 0.828 ppm due to terminal methyl protons, a strong signal at 1.214 ppm from the methylene proton of carbon chain, a signal at 1.265 ppm related to $-$ carbonyl methylene proton and at signal at 7.301ppm associated to olefinic proton.

8.0 PHYSICAL PROPERTIES OF BIODIESEL

Types of Analysis	value	Unit	Method
Density	0.9104	g/ml	Measurement
Viscosity	0.589	cm^2/s	Measurement
Free Fatty Acid	2.9	$\%$	Titration
Acid Value	6.1	mg NaOH/g	Titration

Table 3: Some properties of Groundnut acid oil Biodiesel

9.0 CONCLUSION

Biodiesel is eco-friendly. Biodiesel is easy burning alternative gasoline. Biodiesel include no petroleum, however may be mixed with traditional diesel gasoline. This fuel may be used in any diesel engine without any modification. Biodiesel is degradable, non-poisonous and free from Sulphur and lead.

Hence, the primary goal of the observe become to provide biodiesel from Waste Cooking oil and to apply esterification and transesterification with Ethanol and NaOH Catalyst method. The most excellent parameters which gave most conversion of 79 % the use of 2% w/w catalyst, 1:6 oil to alcohol molar ratio,at 65°C in 1.5 hours which is compare to KOH catalyst give more yield.

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