



doi:10.5281/zenodo.20073191

# Homogeneous Catalysis Of Sunflower Oil Using Vortex Fluidic Device (VFD) As A Means Of Mitigating Climate Change

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## ABSTRACT

Currently, biodiesel synthesis is undergoing rapid technological reforms in both industry and academic institutions. At present, the major challenge of biodiesel synthesis is the cost of production compared to conventional biodiesel. Thus, numerous studies on the use of technologies and different methods to evaluate optimal conditions of biodiesel synthesis technically and economically have been carried out. The dynamic thin film (changes of height and length due to free movement of material as a result of fluid exposure to air and vapour) in VFD is generated by the continuous addition of fluid to a rapidly rotating glass sample tube surface. In this work, virgin sunflower oil was subjected to the transesterification process and the maximum FAME yield of 99.72 wt. % was obtained. Replacing NaOH with KOH is effectively reduces cost, improves green chemistry, and increases the safety of NaOH. Vortex fluidic flow chemistry is effective in synthesising fatty acid methyl esters from sunflower oil sourced triglycerides at room temperature. Despite less reactivity of NaOH, less methanol and catalyst were used compared to other transesterification with the conversion taking place at 98.04 FAMEs wt. %. Optimum operational conditions are 1: 6 volumetric ratio of sunflower oil to methanol and 1.1 moldm<sup>-3</sup> for a combined flow rate of 3.5mlmin<sup>-1</sup> in a tube length of 200 mm, external and internal diameters tube of 20 mm and 17.7 mm respectively, rotating at 6150 rpm. This work furthers the viability of using less reactive, but a cheaper catalyst for biodiesel synthesis. This process is effective in converting fatty acid from virgin sunflower oil to methyl esters at room temperature without the use of co-solvent, with no soap formation and a down-streaming process.

## Keywords

Homogenous alkaline catalyst; Transesterification; biodiesel; VFD

## 1. INTRODUCTION

Today, the price of petroleum-based diesel has been fluctuating now and then, coupled with the challenges of climate change and its emerging mandate obligations have made it necessary for another alternative to liquid fuel. This is especially for the transport and manufacturing industries that 75 – 80 % rely on diesel for their activities [1]. However, upgrading conversion technologies is expected to drive the biodiesel market to robustly advance shortly. Biodiesel specifically has the potential for providing the global population with energy that is sustainable and renewable. The major prospects for biodiesel are manufacturing and transportation industries, which mostly rely on liquid oils for power generations [2]. The global major energy demand ~80 – 85 % is fossil-based fuel, out of which 55-60 % is consumed by

the transport sector [3]. However, the rise in the cost of biodiesel production due to yet unsolved economic, social and technical challenges is a course of concern. In some quarters, however, biofuels generally are receiving subsidies and tax exemption to become a competitive alternative to fossil fuels [4]. Biodiesel can be used in most fuel engines with little or no modification [5], and several countries are utilizing biodiesel for their transport networks [6]. Biodiesel has the potential for providing us with energy that is clean, sustainable, and renewable [7]. The major prospects for biodiesel are manufacturing and transportation industries, which mostly rely on liquid oils for power generations [3]. Biodiesel is traditionally synthesized from animal fats or vegetable oil triglycerides through the acid, base, or in a process called esterification/transesterification [8]. However, the rate of alkali catalyzed transesterification is higher compared to the acid-catalyzed transesterification process [9, 10]. The transesterification reaction is a reaction between vegetable oil and alcohol in the presence of a catalyst that results in the formation of fatty acid methyl (FAME) and glycerol [11].

Feedstocks for biodiesel production come from various sources which include plants and animal kingdoms, however, vegetable oils are major sources for making biodiesel. The edible sources include; Rapeseed oil which accounted for 84% of the world's biodiesel production in 2008, with 13% being produced from sunflower oil [12]. Other feedstocks used for biodiesel production include soybeans, palm oil, safflower, coconut, and peanut [13]. Edible vegetable oils are referred to as "First-Generation" biodiesel feedstocks and are still the most common feedstock used, with over 95% of the world's biodiesel being produced using edible oils [69]. The key advantage of using edible feedstocks is that the plantations and infrastructure to convert the crops into biodiesel are already well established, making it easier to produce biodiesel on a large scale [13, 14]. Other non-edible feedstocks are also employed for biodiesel production to reduce pressure on food crops and to reduce cost of biodiesel production. For instance, Seecharan et al. [15] conducted a laboratory biodiesel production with refined and unrefined used vegetable oil. This study confirmed the importance of pre-treatments of used vegetable oils before converting them to biodiesel. The refined vegetable oil when catalyzed with  $\text{NaOCH}_3$ , the biodiesel yield was 78.9% while unrefined vegetable oil produces only 48.4% yield with the same catalyst at 6; 1 methanol to oil ratio, 60 °C, and 30 minutes reaction time in a conical flask.

Currently, biodiesel production is mainly by traditional batch process, which requires; a long reaction time, high catalyst consumption, higher volumetric ratio, higher temperature, and high stirring time. However, some of the biodiesel synthesised by batch processes did not meet the global standard for biodiesel production [16], even at very high temperatures. Therefore, overcoming the challenge of mass transfer and low conversion efficiency continuous flow processes were also reported in the literature. Santana et al. [17] compared a batch and a continuous micro reactor for biodiesel production using sunflower oil, ethanol, and NaOH as a catalyst. In a batch reactor, the total FAME yield was 94.06 wt. % while, the micro-device reactor total FAME yield was 95.80 wt. %. Reaction time and residence time were 180 minutes to 60 seconds, respectively. Different volumetric ratios 5; 1 (47.32 wt. %), 9.5; 1 (86 wt. %) and 14; 1 (53.02 wt. %) were measured and reported. At 50 °C and 25 °C, 94.06 wt. % and 86 wt. % total FAME yield was reported respectively. The optimum yield reported from 0.2 wt. % NaOH were 85.25 wt. % and 86.66 while at 0.85 wt. % 89.13wt. % and 89.89 wt. % for batch and micro-reactor, respectively.

From the above-reviewed literature, it has become clear that biodiesel synthesis can be achieved through advanced technologies at varying operational conditions. High cost of biodiesel is related to the inability to have in place cost-effective and eco-friendly biodiesel production technologies, while the conventional approach has been established over the years. This is because, reign of diesel engines will remain preferred over spark ignition engines for heavy-duty applications and power generation plants, and as a result, large-scale biodiesel production is necessary. However, biodiesel production is still relatively high costs due to production and feedstock costs [18]. Some of the technical challenges facing biodiesel production which include low production efficiency, long residence time, high operating cost, and energy consumption can be addressed by various process intensification technologies. Among the novel intensification technologies are microwave, oscillatory flow, microchannel flow, static mixer, ultrasonic, rotating disc, cavitation, zigzag microchannel, and spinning tube reactors in a continuous flow process

[19]. These processes are meant to address the challenges facing the conventional methods of biodiesel production which include low mass transfer, heating, use of excess alcohol and other co-solvents, and providing a continuous flow process [20]. Some of the technologies put forward to intensify mass transfer, reduce reaction time, and enhance conversion rates and yield of biodiesel are reviewed below.

## 2. Experimental

### 2.1 Materials

Virgin sunflower oil was purchased from KTC, United Kingdom. NaOH (98% purity) and methanol (99.9% purity) are obtained from Sigma Aldrich. For FAME analysis, Toluene (analytical grade: 99.9%) was obtained from Sigma Aldrich while Methyl Nonadecanoate, C19 (Analytical grade; CAS – 1731-94-8; Lot No. BCCD1343) and Reference material (FAME Rapeseed oil; Lot No. LRAC1276) were obtained from Sigma Aldrich, UK.

### 2.3 Raw material characterization

Even though the two catalysts belong to the same group of homogeneous base catalyst, however, NaOH is less reactive and economically cheaper than KOH, as such is more favourable to use for biodiesel synthesis. Table 1, compared the unit price of the KOH and NaOH used in this study. As can be seen, NaOH is cheaper than KOH in the market. Therefore, by adopting NaOH, the cost of catalytic biodiesel production was reduced significantly. Similar cost analysis and eventually NaOH was selected for NaOCH<sub>3</sub> and KOH [21] to reduce the cost of biodiesel production.

Table 1  
Comparison of the unit price of KOH and NaOH as of February 2022

	KOH [Purity: ≥99%; CAS No. 1310 – 58-3]	NaOH [Purity: ≥85%; CAS No. 1310 – 73-2]
<b>Price [GB£/5G]</b>	110.00	22.00

<sup>a</sup> Prices are quoted from United Kingdom-Sigma - Aldrich

#### 2.3.1 Fatty acid composition

Before and after the transesterification of the sunflower, both sunflower and biodiesel fatty acid compositions were determined by the method described by Jabbar et. al. [22] and British standard methods [23]. The fatty acid composition was determined using Perkin Elmer Clarus gas chromatography (580S, GC) equipped with mass spectroscopy. The conversion efficiency of the quantifiable saturated and unsaturated fatty acids was determined using GC-MS data obtained from the virgin sunflower oil and the direct transesterification of sunflower oil with NaOH under a VFD-assisted processing platform. The acid value (mgKOH/g) and FFA (%) were determined using various standard methods as described somewhere [24] for precisions and accuracy. The acid value and the percentage of the FFA of the SO and synthesized biodiesel were calculated as in equation 1.

$$\text{Acid Value (AV)} = \frac{56.1 \times V \times C}{m} \dots\dots\dots \text{Equation (1)}$$

In addition, the flashpoint of the prepared biodiesel sample was measured by an auto ramp closed cup flash point tester (Setaflash series 3, England) equipped with a coolant block unit. At ambient temperature, 2.0 ml of biodiesel sample was manually injected into the sample cup via the filter orifice and the instrument was set on the expected flash temperature for the biodiesel sample. The temperature ramped at 1 – 2 °C /min until the flash was captured [25].

Table 2 Physicochemical Properties of Sunflower Oil

Property	Sunflower oil
Acid Value	0.2413
Free fatty Acid	0.1215
Fatty acid composition (wt. %)	
Palmitic acid, ME <sup>a</sup>	6.81
Linoleic acid, ME	51.47
Oleic & linolenic acids, ME	35.25
Stearic acid, ME	6.47
Density (gml-1)	0.8765g/cm <sup>3</sup>
Flash point (°C)	181

<sup>a</sup> Methyl Ester

Finally, the physicochemical properties of the two catalysts used in this study are summarised in Table 3, NaOH as supplied by the manufacturer (Sigma Aldrich-United Kingdom).

Table 3

Physicochemical Properties of NaOH catalyst

Property	Sodium hydroxide [NaOH]
Colour	White
Form	Pellets
Solubility colour	Colourless
Solubility (Turbidity)	Clear
100 mg/ml, H <sub>2</sub> O	
Titration with HCl	98%
Impurity	1.0%
As Na <sub>2</sub> CO <sub>3</sub>	
Formulae	HNaO
Molecular formulae	40.00g/mol

#### 2.4 VFD rig setup

The transesterification of sunflower oil was performed using the VFD as illustrated in Figure 1. The VFD was equipped with a 20 mm external diameter glass tube. The sunflower oil and methanol mixed with KOH catalyst were injected via automated pumps at a different flow rate. Products were collected in a graduating cylinder and transferred into a separatory funnel. The product collected is separated into two, sometimes three layers of biodiesel. The lower layer (glycerol) was removed first, followed by the middle layer (biodiesel) then the top layer. In a situation where the output separates into two layers, the bottom layer was removed as glycerol and the top layer was analysed as biodiesel, as shown in Figure 2. The biodiesel collected is always stored in refrigeration until (approximately 2.5 °C) it is used for off-line GC analysis. The biodiesel samples were analysed using Perkin Elmer Clarus gas chromatography (580S, GC) equipped with mass spectroscopy to quantify their FAME content.

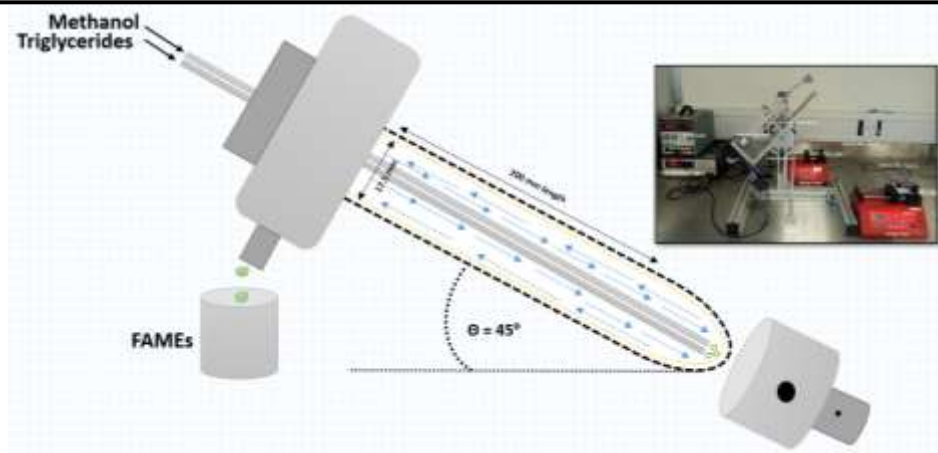


Figure 1. Schematic diagram VFD showing its components used for the conversion of SO and NaOH in methanol to FAMEs.

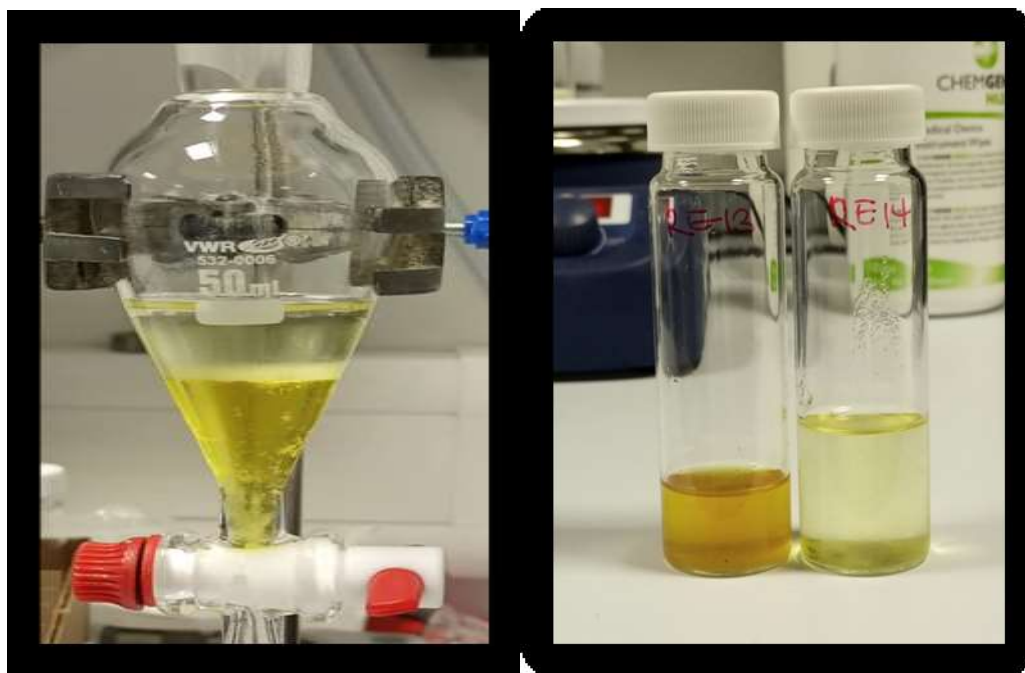


Figure 2: Photographs of (a) the two phases separated and (b) brown and cleared, glycerol and biodiesel using  $1.1 \text{ mol dm}^{-3}$  NaOH concentration.

### 3. RESULTS AND DISCUSSION

Analysing of the synthesised biodiesel using GC-MS and FT-IR, shows almost a complete conversion of sunflower oil to biodiesel FAMEs with 98.04 wt. % and 97.30 wt. % at  $1.1 \text{ mol dm}^{-3}$  and  $0.4 \text{ mol dm}^{-3}$ , respectively, NaOH. The used of the VFD-assisted platform has proved that the nature of the feedstock does not affect conversion to the corresponding biodiesel methyl esters. The GC-MS data show that over five different forms of methyl esters are synthesised using this platform, within 17.0 minutes to 35.0 minutes, retention time. The GC-MS (Perkin Elmer Clarus 5600) was used to identify the total FAMEs content as well as individual FAMEs generated from the VFD-assisted transesterification process. The main FAMEs were palmitic acid (C16: 0), linoleic acid (C18: 3), oleic acid (C18: 1), linolenic acid (C18: 2) and Stearic acid methyl esters (C18: 0) as shown in Figure 3.

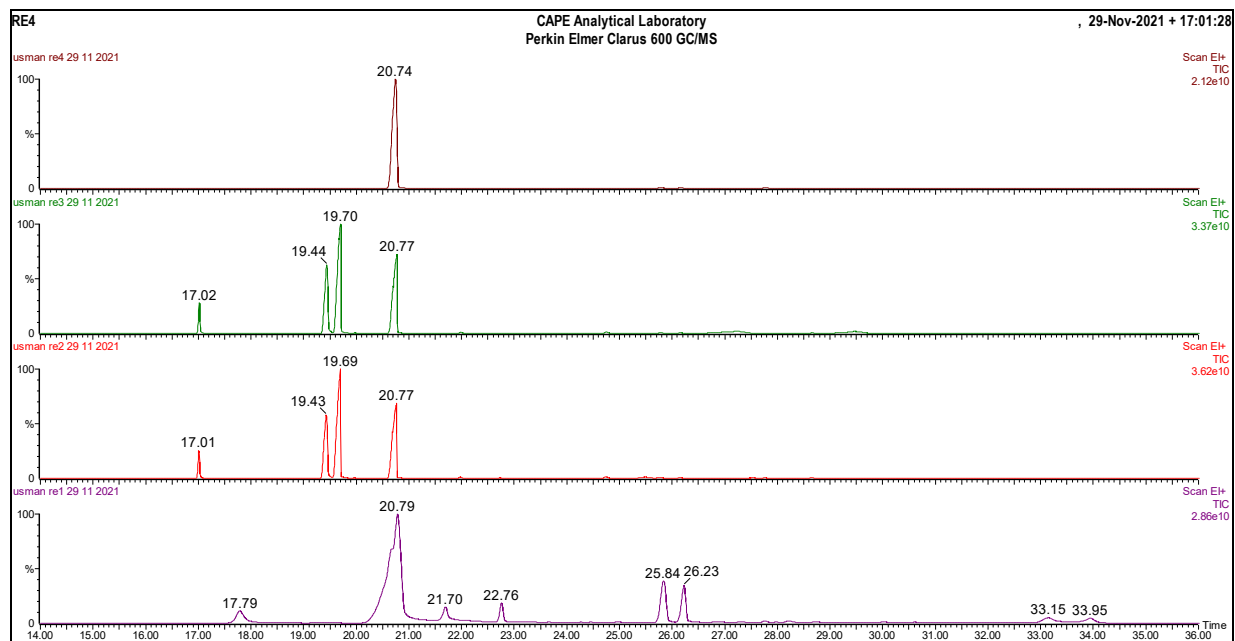


Figure 3: GC-MS of the biodiesel obtained at 0.4 moldm<sup>-3</sup>-NaOH, 1; 1 sunflower oil to methanol ratio at 6, 150 rpm and 45 tube inclination angles.

Similar result was reported by Raston and Britton [26] and Barbaras et al. [27] where the retention time are between 17.03 min to 25.87 min. the specific retention time at 17.05 min, 19.32 min, 21.37 min, 21.59 min, 22.38 min and 25.87 min for the corresponding GC-MS chromatogram reported by Britton and Raston [26]. The formation of FAMES was confirmed by the peaks between 17.00 min – 25.87 min retention time (Figure 4.), these also corresponds to what was obtained from the reference biodiesel.

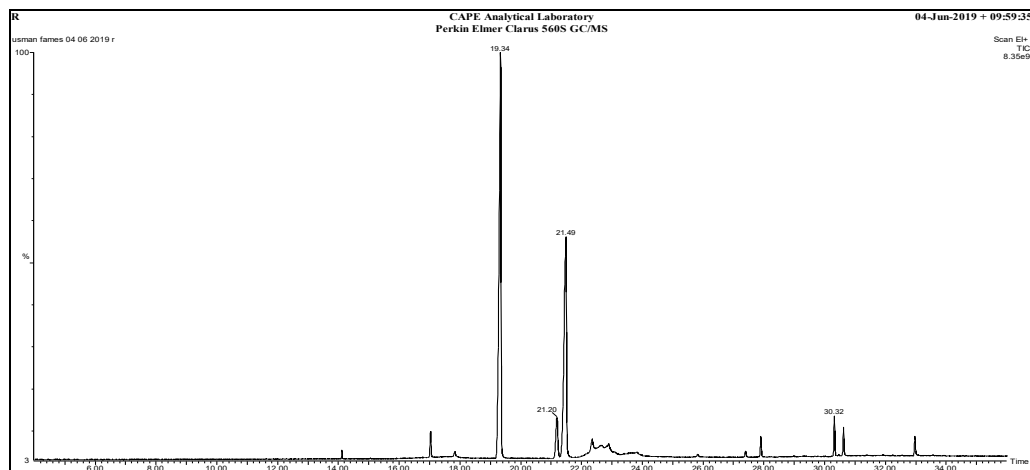


Figure 4: Analytical Standard: AOCs Low Erucic Rapeseed Oil and was prepared using (Ampule of 100 mg, Internal Standard 100 mg , and n-Heptane - 10 ml using GC - MS Analysis according to British Standard Method EN 14103 – 2011.

FT-IR instrument was used to identify the functional groups relating to various stretching and bending vibrations (fingerprint) of the biodiesel synthesised using the VFD-assisted methanolysis, and compared to the spectrum for sunflower triglycerides (Figure 5) used as the feedstock. From the FT-IR data, the peak at 1,163 cm<sup>-1</sup> in the sunflower oil's triglyceride spectrum, corresponds to the stretching vibration of

the C-O group attached to  $-CH_2$  which converts to the peak at  $1,170\text{ cm}^{-1}$  in the methyl spectrum, attributed to the stretching vibration of C-O group attached to the  $-CH_3$ . The absence of the peak at  $3,200\text{ cm}^{-1}$ , correspond to  $-OH$  group, indicates there is no intermediates such as mono and di glycerides as well as unreacted glycerine and methanol in the biodiesel samples [26, 28 – 30].

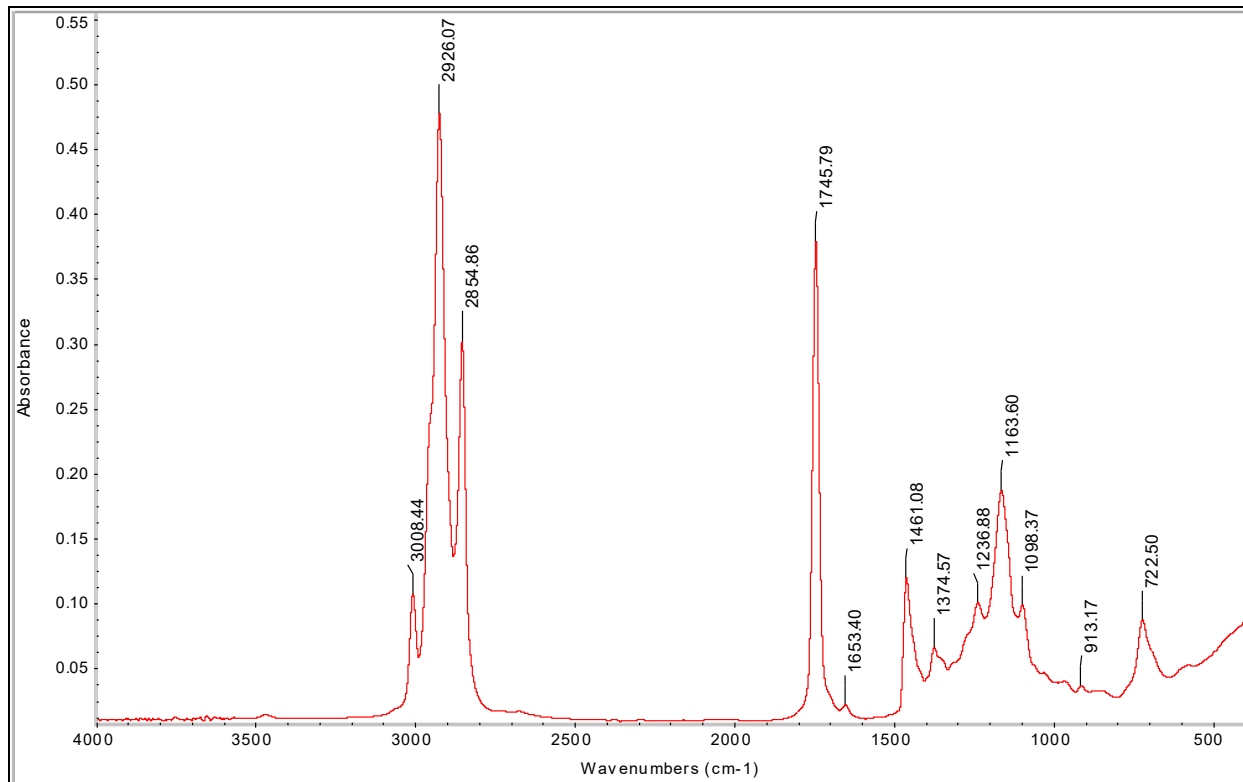


Figure 5: Fourier Transform Infrared (FT-IR) Spectroscopy of Virgin Sunflower

The main absorption peaks of sunflower oil's biodiesel were at  $2,954$  to  $2854\text{ cm}^{-1}$ ,  $1744\text{ cm}^{-1}$ ,  $1377\text{ cm}^{-1}$ ,  $1460\text{ cm}^{-1}$  and  $1196\text{ cm}^{-1}$ , as shown in Figure 6. The strong absorption peak at  $1743.87\text{ cm}^{-1}$  is specific for C=O while the peak at  $1241.70\text{ cm}^{-1}$  correspond to C-O. The formation of FAME was confirmed by the peaks at  $1460\text{ cm}^{-1}$  and  $1196\text{ cm}^{-1}$ , which is correspond to  $CH_3$ - asymmetric and  $CH_3$ -O stretching, as shown in Table 4.

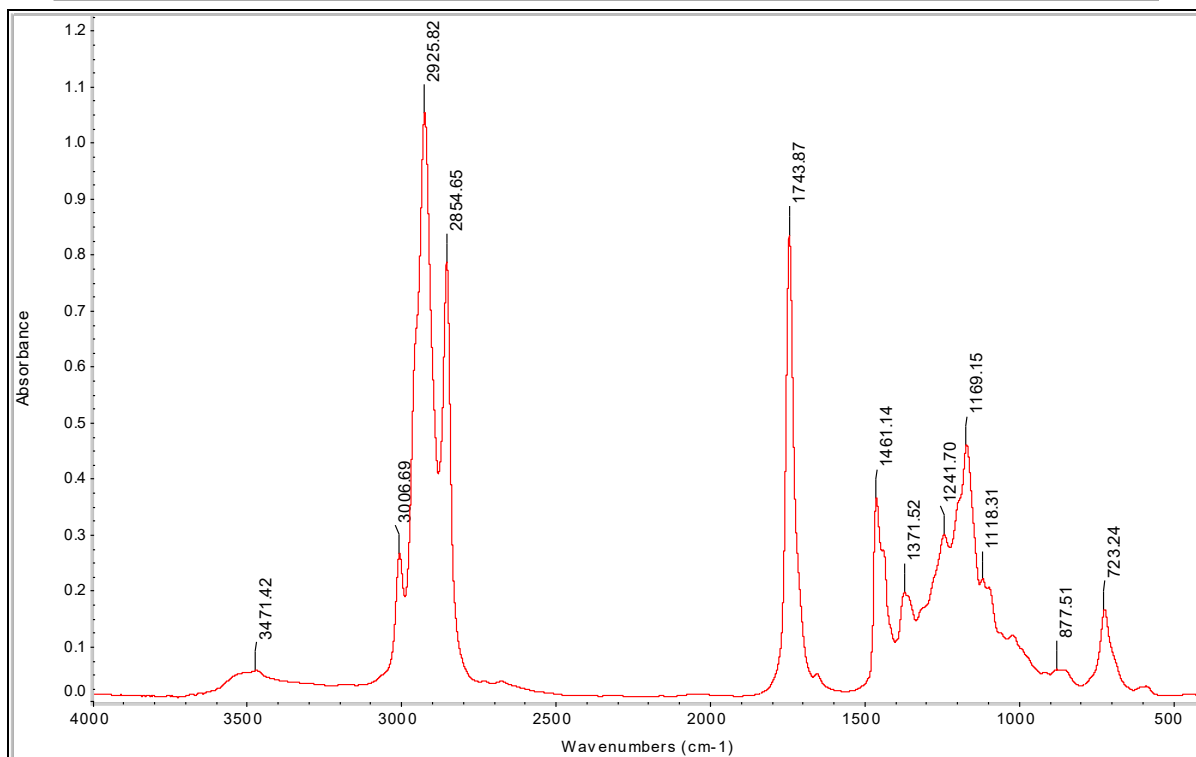


Figure 6: Fourier Transform Infrared (FT-IR) Spectroscopy of the synthesised Biodiesel using NaOH as a catalyst

For the biodiesel synthesised from sunflower using NaOH as catalyst, the FT-IR spectrum was obtained and the various absorption peaks were compared in Table 5. The peak at  $1,163\text{ cm}^{-1}$  in the triglyceride spectrum (Figure 6) corresponds to the stretching vibration of the C-O group attached to  $-\text{CH}_2$  which converts to the peak at  $1,170\text{ cm}^{-1}$  in the methyl spectrum, attributed to the stretching vibration of C-O group attached to the  $-\text{CH}_3$ . The absence of the peak at  $3,200\text{ cm}^{-1}$ , correspond to  $-\text{OH}$  group, indicates there are no intermediates such as mono and di glycerides as well as unreacted glycerine and methanol in the biodiesel samples. The synthesized biodiesel, as in Table 4, are very similar to that of standard of diesel and biodiesel reported in the literature Rashid et al. [30] having long-chain fatty acid esters. The observed bands at  $1743.87\text{ cm}^{-1}$  and  $1461.14\text{ cm}^{-1}$  (Figure 6) are attributed to ester and methyl groups respectively. The presence of two groups namely methyl and esters ( $-\text{CH}_3$  and  $-\text{CH}_3\text{O}$ ) [31] in the biodiesel samples indicate that the transesterification of SO occurred due to addition of methanol and NaOH catalyst. This result is similar to what was reported by Britton and Raston [26]. FT-IR analysis verified that there was no vibration ( $-\text{OH}$ ) absorption peak in the region of  $3200 - 3600\text{ cm}^{-1}$ , indicating there was little acid and methanol in the synthesized biodiesel.

Table 4 Physicochemical properties of biodiesel obtained from SO, methanol, and NaOH

Property	Current study (a) (0.4M)	Current study (b) (1.1M)	Rashid et al. [31]
Acid value (mg KOH/g)	0.22	0.25	0.24
Free fatty acid (%)	0.11	0.125	0.12
Density (gml-1)	0.8233	0.8622	0.88
Flash point (°C)	172	151	
<b>Fatty Acid Composition (wt. %)</b>			
Palmitic acid methyl ester (C16:0)	7.35	5.96	7.0
Linoleic acid methyl ester (C18:2)	35.15	33.24	55.2
Oleic & Linolenic acid methyl esters (C18:1)	56.58	60.19	33.3
Arachidic acid methyl ester (C18:0)	0.92	0.61	3.5
Mean Molecular Weight (g/mol)	876.16g/mol	876.16g/mol	

#### 4. Conclusions

Here, we design and develop the use of a continuous flow process technology for synthesising biodiesel from sunflower oil at room temperature without the necessary use of co-solvent. The process requires mild operational conditions using cheaper NaOH in methanol. The process does not result in saponification - breakdown of the triglyceride fats in the presence of hydroxides. The vortexing process is effective and efficient for biodiesel without the implication of downstream processing due to the easy separation of the glycerol layer from biodiesel. For the first time, a cheaper and basic NaOH was applied for the synthesis of biodiesel using the VFD-assisted transesterification process. It was found that a 97.30 wt. % FAME yield from SO feedstock can be achieved with the VF-assisted transesterification process at room temperature without the use of excess solvent. The 0.4 moldm<sup>-3</sup>-NaOH and methanol to SO ratio of 1; 1 (v/v), 6, 150 rpm and 45° inclination angle relative to a horizontal position, within 8 minutes residence time. Furthermore, the same NaOH was further optimized at 1.1 moldm<sup>-3</sup>. It was found that a 98.04 wt. % FAME yield from SO feedstock can be achieved with the VF-assisted transesterification process at room temperature without the use of excess solvent. The methanol to SO ratio of 6: 1 (v/v), 6, 150 rpm and 45° inclination angle relative to a horizontal position, within 18 minutes residence time. Table 5, compared the VFT to batch processing platforms for the transesterification of sunflower oil with NaOH. Comparison of VFT processing with related continuous flow processing, Table 6, and highlights some advantages in using the VFD. These include lower temperature, methanol ratio without compromising on the FAME yield. This was achieved as a result of shear stress formed within the VFD-reactor tube during processing.

Table 5. Comparison of mild operational conditions for the reported VFD and Batch processes with current study in the biodiesel synthesis

	<b>This study</b>	<b>This study</b>	<b>Saydut et al. [32]</b>
<b>Type of reactor</b>	<b>The VFD</b>	<b>The VFD</b>	<b>Batch</b>
<b>Feedstock</b>	Sunflower oil	Sunflower oil	Sunflower oil
<b>Type of catalyst</b>	NaOH	NaOH	NaOH
<b>Catalyst concentrations</b>	0.4 moldm <sup>-3</sup>	1.1 moldm <sup>-3</sup>	0.5% w/w
<b>Methanol/Oil ratio</b>	1: 1	6: 1	6: 1
<b>Rotor speed</b>	6, 150 rpm	6, 150 rpm	200 rpm
<b>Tube inclination</b>	45°	45°	n/a
<b>Reaction temperature</b>	n/a	n/a	60 °C
<b>Reaction/residence time</b>	8 minutes	18 minutes	2 hours
<b>Total FAME Yield</b>	97.30 wt. %	98.04 wt. %	91.40 wt. %

Table 6. Comparison of VFD Processing of Biodiesel with Related Continuous Flow Processes

Reactor type	Residence time	Temperature	Methanol to oil ratio	Yield %	Reference
Microwave	-	60 °C	15: 1	95.45	[33]
Oscillatory flow	-	60 °C	6: 1	81.9	[34]
Microchannel	180	50 °C	5: 1	95.80	[17]
Static mixer	60 minutes	60 °C	6: 1	97.80	[23]
Spinning disk	-	48 °C	6.8: 1	98.18	[35]
Ultrasonic	15	20 – 25 °C	12: 1	97.85	[36]
VFD (NaOH – 0.4M)	8 minutes	rt	1:1	97.30	This study
VFD (NaOH– 1.1M)	18 minutes	rt	6:1	98.04	This study

## ACKNOWLEDGMENTS

The authors would like to acknowledge the management of Umar Suleiman College of Education Gashua, Bade Local Government Area of Yobe State, Nigeria.

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