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Biodiesel production process intensification using a rotor-stator type generator of hydrodynamic cavitation

Daniele Crudo[a], Valentina Bosco[a], Giuliano Cavaglià[a], Giorgio Grillo[b], Stefano Mantegna[b],
Giancarlo Cravotto[b,*]

[a] *E-PIC S.r.l. Turin, Via Ippolito Nievo 25, Turin,10143, Italy.*
[b] *Dipartimento di Scienza e Tecnologia del Farmaco and NIS - Centre for Nanostructured Interfaces and Surfaces, University of Turin, Via P. Giuria 9, 10125 Turin, Italy.*

Abstract

Triglyceride transesterification for biodiesel production is a model reaction which is used to compare the conversion efficiency, yield, reaction time, energy consumption, scalability and cost estimation of different reactor technology and energy source. This work describes an efficient, fast and cost-effective procedure for biodiesel preparation using a rotating generator of hydrodynamic cavitation (HC). The base-catalyzed transesterification (methanol/sodium hydroxide) has been carried out using refined and bleached palm oil and waste vegetable cooking oil. The novel HC unit is a continuous rotor-stator type reactor in which reagents are directly fed into the controlled cavitation chamber. The high-speed rotation of the reactor creates micron-sized droplets of the immiscible reacting mixture leading to outstanding mass and heat transfer and enhancing the kinetics of the transesterification reaction which completes much more quickly than traditional methods. All the biodiesel samples obtained respect the ASTM standard and present fatty acid methyl ester contents of >99% m/m in both feedstocks. The electrical energy consumption of the HC reactor is 0.030 kWh per L of produced crude biodiesel, making this innovative technology really quite competitive. The reactor can be easily scaled-up, from producing a few hundred to thousands of liters of biodiesel per hour while avoiding the risk of orifices clogging with oil impurities, which may occur in conventional HC reactors. Furthermore it requires minimal installation space due to its compact design, which enhances overall security.

Keywords: Biodiesel; Process intensification; Hydrodynamic cavitation; Palm oil; Waste cooking oil**.**

1. Introduction

A huge gap currently exists between classic production processes and the most recent generation of enabling technologies which provide process intensification, higher efficiency and sustainability. Over the last few years, chemists and chemical engineers have found that sonochemical methods furnish attractive solutions to bottle necks in chemical processes [1]. Sonochemical effects arise from the action of ultrasound (US) waves and/or hydrodynamic cavitation (HC). The use of sound energy and in a liquid flow can result in significant gains in process intensification by generating cavitational events inside reactors. When a liquid passes through a constriction, it undergoes a sudden increase in its velocity of liquid at the expense of local pressure. If this local pressure falls below the vapour pressure, a number of cavities will be generated and subsequently collapse, while pressure is recovered downstream of the mechanical constriction.

Several authors have studied reactors since the early 1990s in order to optimize cavitation distribution [2–4]. The last decade has seen the chemistry of flowing systems become more prominent as a method of carrying out chemical reactions, which can range from the lab scale up to kilogram-scale processes [5, 6]. Flow reactors stand out above their classic batch sonochemical process counterparts thanks to their greater efficiency, flexibility and lower energy consumption [7].

The aim of this work is to draw attention to a new means of generating HC, using rotation generators, and their easily operated applications in loop and flow through modes [8]. The reduced energy consumption, environmental and economical impact of this type of reactor mean that it is well suited to industrial scale up and biodiesel production in particular. Most commercial oil transesterification technologies that use cavitation are currently based on acoustic cavitation (US), which is simpler to implement than HC, but also less energy efficient. Cavitation in US reactors (horns and baths) occurs in a confined region around the vibrating surface meaning that there are evident limitations to its operational scale up. Lab scale HC, on the other hand, is either generated by multiple-hole orifice plates or simple Venturi restrictions that require high power pumps because of the considerable pressure losses caused by the restrictions. An innovative design has been presented by Kumar and Pandit [9], who used a high-speed homogenizer consisting of an impeller inside a cage-like stator with numerous slots where cavitation generates. Another rotor and stator based design has been presented by Badve *et al.* [10]. In this set up, a rotor presents a solid cylinder with indentations on its surface meaning that cavity regions are established inside the indentations due to high speed rotation. A similar design with two counter spinning rotors has also been presented by Petkovšek *et al.* [8], which, like all the other machines presented, needs an extra pump for operation. This means that the cavitation generator causes extra pressure losses in the existing system. The same Authors have very recently improved the system making it more energy efficient thanks to an extremely compact generator and pump [11]. This device has been outstanding in waste water treatment [12]. This work tests a novel commercial unit (patent pending) that was designed by E-PIC S.r.l. (Turin, Italy). It is characterized by rotor-stator apparatus equipped with a three-phase, 7.5 kW electric engine, while absorbed power is constantly monitored (Fig. 1).

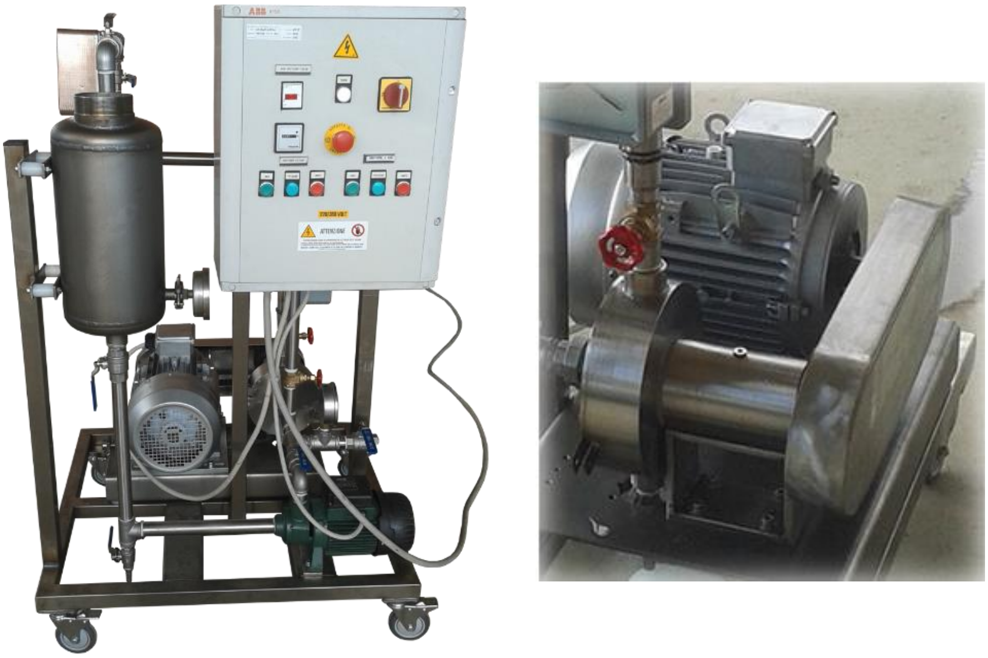

Figure 1. Rotor-stator generator of HC.

A concerned effort has been made to search out new highly efficient reactors that produce biodiesel while saving time and energy. Inefficient mass transfer is one of the main limitations in biphasic heterogeneous reactions, such as transesterification. Although many types of vigorous mixing have been investigated to address this requirement, most of them suffer from high energy demands [13-15]. Optimal mass/heat transfer is doubtless the key to enhancing biodiesel production while cavitational effects may also play a significant role. The two main routes to acoustic cavitation are high-intensity ultrasound and HC (passage of liquid at high flow rates through a constriction), although rotating HC generators are the most recent innovation.

With the aim of reducing energy consumption in biodiesel production, we have thoroughly investigated the effect of cavitation on the transesterification reaction of triglycerides over the last few years [16]. We have also investigated combinations of different enabling technologies both in batch and in flow processes [17-19]. Hybrid processes for biodiesel production that function upon a combination of sonochemical reactors and other intensification techniques have been thoroughly reviewed [20] as have continuous flow technologies [21]. In 2005 Gogate and Pandit described HC as a technology for the future [22], and one year later, a Chinese group from the Zhejiang University of Technology compared US and HC conditions for biodiesel production [23]. The techniques displayed similar enhancement effects in the transesterification reaction of soybean oil, both giving shorter reaction times and lower energy consumption than the conventional mechanical stirring method. Several authors have confirmed these results [24, 25] and have highlighted HC's easier scale-up compared to US methods. The micro level turbulence created by HC may be able to overcome the mass transfer limitations of a triphasic reaction such as transesterification.

The high cost of vegetable oils and the ethical issues that surround competition with the food industry for oil crops means that much attention has been focussed on non-edible oils and used frying oils which are a renewable source [26-28].

The recycling of waste cooking oil (WCO) that had been used for frying has been investigated under HC conditions [29-32]. HC has also been used as a pre-treatment for residual algal biomass's conversion into cheap feedstock for yeast cells that produce lipids [33].

This work highlights the potential industrial applications of a rotating generator of HC for biodiesel production even with waste unfiltered cooking oil feedstock.

2. Material and methods

2.1 Material

Transesterification reactions were carried out using two different feedstocks; refined and bleached palm oil, purchased from Olitalia (Italy), and waste vegetable cooking oil, a mixture of vegetable oils collected from several fast foods restaurants in Turin (Italy).

Methanol, 99.8% grade, sodium hydroxide, 98% grade, isopropyl alcohol, 99.0% grade, and hydrochloric acid, 36% grade, were purchased from Sigma-Aldrich (Milan, Italy). Distilled water was used for dilutions during analysis and biodiesel washing.

2.2 Experimental procedure

A series of preliminary tests were conducted on refined and bleached palm oil with the aim of defining optimal process conditions. Three working temperatures (35 °C, 45 °C and 55 °C) and three different cavitation reaction times (10, 15 and 25 min) were compared by monitoring triglyceride's conversion to methyl esters. It was clear that the transesterification was much faster at 55 °C, reaching equilibrium after 15 min. Once temperature and time were defined according to the characteristics of the reactor, we were able to compare two different flow-rates: 250 and 390 L/h. At 390 L/h, the average residence time in the HC reactor is around 10 sec with a negligible temperature rise (less than 1 °C), which gave excellent results in both transesterification steps. The pilot unit in Figure 2 shows all the components of the rotating generator of HC used in the investigation.

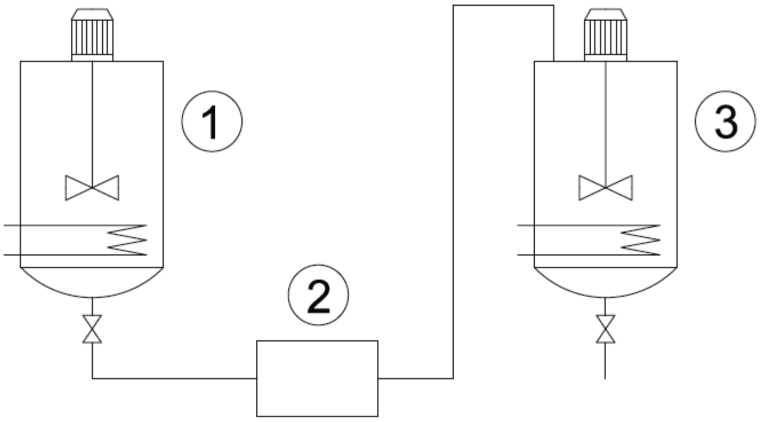

Figure 2. HC pilot unit: (1) Conditioned fed tank, (2) Rotor-stator type HC reactor, (3) Collector tank and separator.

In this work biodiesel is produced from a double step transesterification reaction and by dosing 75%, by volume, of the methoxide solution in the first step and the remaining 25% in the second step. This choice comes from a preliminary series of tests on fatty acid methyl esters (FAME) conversions performed on lab scale. The WCO was first filtered under vacuum over a five-layer cotton gauze placed on a Buckner funnel (Ø 15 cm) to remove solid impurities. The filtered oil was heated at 50 ˚C under vacuum for 2 hours to remove the residual water. The free fatty acids (FFA) content of WCO was measured in order to calculate the additional catalyst (NaOH) to be added to neutralize the oil. For FFA analysis, isopropyl alcohol (10 mL) was added under stirring to 1 mL of WCO. A solution of NaOH in distilled water (1:1000) was then slowly added dropwise until a pH of 8.5 (pH-meter) was reached. Total FFA content, expressed as acid number, is evaluated according to the following equation:

$$\text{Acid number (mg NaOH/g oil)} = \frac{v}{m_{oil}}$$

v = Volume of added NaOH solution (ml)
m_{oil} = Mass od neutralized oil (g)

The feedstock (i. refined and bleached palm oil and, ii. WCO) was poured into the first tank (1) and heated over the reaction temperature, at 60 ˚C; then 75% by volume of methoxide solution was added to the oil and premixed for 30 sec, to obtain a homogeneous mixture, at a reaction temperature of 55 ˚C. The sodium methoxide solution was prepared by mixing NaOH with methanol under stirring at room temperature for several hours, until the entire solid phase was dissolved. This mixture was sucked by the rotating generator of HC (2), at a flow rate of 390 L/h, and subjected to controlled cavitation and discharged into the stirred collector tank (3). The rotor-stator type HC reactor is a commercially available unit designed by E-PIC S.r.l. (Turin, Italy). It can achieve controlled HC by forcing fluids through its rotating cylinder, which is equipped with a 7.5 kW electric engine and absorbed power is constantly monitored. During high-speed rotation, rotor channels are periodically aligned with stator channels. The processed liquid is accelerated in the radial direction in the cavitation chamber and is subjected to a pressure wave, when flowing through the free channels, which results in cavitation. Shockwaves are produced when the cavitation bubbles collapse and the reagents are mixed on a micron scale, increasing the surface contact area between the methanolic methoxide solution and the oil phase. The maximized mass transfer and alcohol vapour generated in the hot spots strongly accelerate the transesterification reaction.

The mixture is maintained at the same temperature for 15 min under moderate stirring in the discharge tank (3). Glycerol is then finally allowed to separate from the crude biodiesel and unreacted oil, via the action of gravity, in the same tank in 30 min. After glycerol separation, crude biodiesel and the mixture of mono- and diglycerides are transferred to the premixing tank (1) and stirred for 30 sec with the remaining 25% by volume of methoxide solution, at the reaction temp. of 55 ˚C. The mixture is again sucked by the HC reactor, at the same flow rate of 390 L/h, and discharged into the second tank (3), where it is maintained at the same temperature for an additional 15 min under moderate stirring. After 30 min, two clean layers of glycerol and biodiesel are separated in the tank and collected from the bottom.

Before washing with water, the crude biodiesel is analyzed to evaluate conversion and the total soap content. Crude biodiesel is finally washed with distilled water, in a ratio of 1:1 v/v to biodiesel, five times and total soap content is again measured.

All final product analyses were confirmed by INNOVHUB, stazioni sperimentali per l'industria innovazione e ricerca, Laboratorio Sostanze Grasse e Derivati (Milan, Italy).

A simple routine qualitative test to evaluate FAME content was carried out after each experiment by adding methanol (27 mL) to the biodiesel sample (3 mL) in a test tube and shaking it for about 30 sec, at room temperature; since biodiesel is soluble in methanol, good FAME conversion implies no visible precipitate or cloudiness. The total soap content test is performed by adding biodiesel (10 g) to isopropyl alcohol (100 mL) under stirring in a beaker, then dil. HCl (0.01 N) is added dropwise until pH 4.5. Total soap content is evaluated by the following equation:

$$\text{Total soap content (ppm)} = \frac{q*N*PM}{m}*1000$$

q = Volume of added HCl solution (ml)
N = Normality of HCl solution
PM = Molecular weight of NaOH (g/mol)
m = Mass of biodiesel (g)

FFA were quantified in WCO; the WCO acid number was 2.18 mg of NaOH 98% per 1 g of oil. For refined and bleached palm oil, the catalyst ratio was 3.67 g of NaOH 98% per 1 L of palm oil, this gives a total amount that rounds off to 5.7 g / L of oil.

In refined and bleached palm oil, the methanol:oil stoichiometric ratio is 13% v/v. A methanol excess of 77.5% by volume was fixed, resulting in a total methanol amount of 1.725 L.

We assumed that WCO is a mixture of different vegetable oils largely composed of sunflower and palm oil, a greater excess of 90% by volume was chosen, which gave a total amount of 1.852 L of methanol.

The matrix of experimental conditions is reported in Table 1.

Vegetable oil **Parameter**	Refined and bleached palm oil	WCO
Oil amount	7.5 L	7.5 L
Methanol amount	1.725 L	1.852 L
NaOH conc. (98% grade)	3.67 g/L of oil	5.67 g/L of oil
Additional treatments	-	Filtration, dewatering
Methoxide – oil premix time (1st step)	30 sec	30 sec
Reaction temperature	55 ˚C	55 ˚C
Reaction pressure	atmospheric	atmospheric
Cavitation number	0.2	0.2
Flow-rate in rotating HC reactor (1st step)	390 L/h	390 L/h
1st step reaction time	15 min	15 min
Biodiesel, unreacted oil – glycerol separation time (1st step)	30 min	30 min
Methoxide – oil premix time (2nd step)	30 sec	30 sec
Flow-rate in rotating HC reactor (step)	390 L/h	390 L/h
2nd step reaction time	15 min	15 min
Biodiesel /glycerol separation time (2nd step)	30 min	30 min

Table 1. Matrix of experimental conditions

3. Results and discussion

We herein report a new, efficient HC protocol for biodiesel production from edible and waste oil. The latter feedstock contains FFA that may lead to soap formation via the undesirable saponification reaction, especially when homogeneous basic catalysts are employed in the transesterification reaction. Methods for reducing or removing FFA from oils are neutralization, extraction by alcohol, steam distillation and the esterification reaction [34]. Many limitations and drawbacks have been documented for all of these methods; however the esterification reaction makes best use of FFA in oils as it transforms FFA directly into FAME. Recent studies have described the adsorption of free fatty acids using an anion-exchange resin such as Dowex 550A [35], as a prior step to the transesterification reaction in order to replace the esterification reaction.

All the rapid tests on unwashed and washed biodiesel samples (from both types of vegetable oil) after the second step of the transesterification gave excellent conversions. The photo in Figure 3 shows an example of the test on unwashed biodiesel from refined and bleached palm oil.

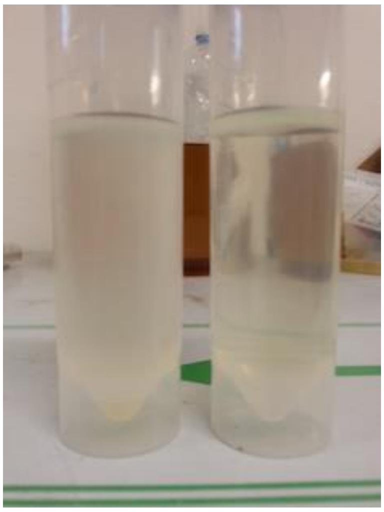

Figure 3. Rapid test on biodiesel (unwashed) from palm oil. Left: sample after the 1st step of reaction. Right: after the 2nd step of the reaction.

The mixture is not clear after the first step of the reaction; some precipitate is visible on the bottom of the test tube. This indicates the presence of monoglycerides and diglycerides. However, no precipitate or cloudiness is visible after the second step.

Comparable results were obtained for both feedstocks (palm oil and WCO). Total soap contents were also evaluated for unwashed and washed biodiesel after the second reaction step, as reported in Table 2.

	Refined and bleached palm oil	WCO
crude biodiesel	1450 ppm	1500 ppm
washed biodiesel	30 ppm	30 ppm

Table 2. Total soap content in biodiesel samples

Soap and glycerol are usually removed from biodiesel by washing with water which produces a large amount of waste water with emulsions. After washing with water, the fuel must be dried in order to remove the trace amounts of water. This entails an increased energy cost. A promising alternative is treatment on ion-exchange resins and solid adsorbents [36]. The mechanism of soap removal involves a combination of different phenomena such as filtration, physical adsorption, ion exchange and soap removal by glycerol affinity. In our hands, lab scale filtration using magnesium silicate and an ion-exchange resin was neither faster nor more efficient that water washing. The so called "dry washing", cannot be applied when the total impurities amount in the biodiesel is over 1000 ppm (glycerol, soap, residual catalyst and other trace impurities). This also with the more common Amberlite® and Purolite® that are both based on a gel-type resin built around the same chemical ion-exchange resin protocol or with some peculiar natural adsorbents [37].

Both washed biodiesel sample series have been analysed by INNOVHUB (Laboratorio Sostanze Grasse e Derivati, Milan), who analysed ester contents (standard EN 14103:2011 + ISO 5508:1990) and the methyl ester of linoleic acid. Results are reported in Table 3.

	Refined and bleached palm oil	Waste vegetable cooking oil
Ester content EN 14103 + ISO 5508:1990	99% m/m	99% m/m
Methyl ester of linoleic acid EN 14103 + ISO 5508:1990	0.2% m/m	0.3% m/m

Table 3. FAME conversion in biodiesel samples

A more detailed analysis was performed on the biodiesel sample from WCO by the SSOG who investigated the content of monoglycerides, diglycerides, triglycerides, free glycerol and total glycerol. Results are reported in Table 4.

	WCO
Monoglycerides content	0.51% m/m
Diglycerides content	0.19% m/m
Triglycerides content	0.06% m/m
Free glycerol content	<0.005% m/m
Total glycerol content	0.162% m/m
Density at 15°C	0.89 g/cm^3
Kinematic viscosity at 40°C	4.58 mm^2/s
Oxidative stability at 110 °C	8.04 h

Table 4. Main analytical parameters of biodiesel from WCO (EN 14105; EN 14214).

Overall energy consumption in this work is actual energy as measured by a plug-in power meter. The total actual energy consumption needed to produce biodiesel was 0.030 kWh/L (0.015 kWh/L for each step), which is lower than in the conventional process (0.222 kWh/L) [38]. We converted the specific consumption from kWh/L to kWh/ton (0.030 · 1000 / 0.88 = 34 kWh/ton) by taking the average biodiesel density to be 0.88 kg/L, as the figure varies between 0.86 and 0.90 kg/L. In terms of power consumption, this unit is more easily compared to the commercial technologies advertised by several international companies (J.J. Lurgi, Axens Corporate, Energea GmbH etc.).

Given the fact that the price of raw materials dominates biodiesel production cost (76-84%), the economical sustainability of the process depends on energy consumption [39].

Besides the FAME conversion greater than 99% m/m, the properties of the obtained biodiesel (acid value, density, viscosity, and oxidative stability) satisfy the ASTM standard.

This clearly emphasizes the higher mass transfer efficiency that the rotor-stator generator of HC provides over conventional methods while it is also competitive with classic US and HC reactors in terms of scalability.

Conclusion

In this work, a novel rotor-stator type generator of HC has been efficiently used to produce high quality biodiesel and has given dramatic process intensification. Whereas orifices may become clogged by impurities in conventional HC reactors, this system favours the flow of the suspension mixture. This innovative technology is competitive in terms of electric energy consumption (0.030 kWh per L of biodiesel) and its scalability of up to thousands liters per hour. We believe that cavitational reactors and their synergistic combination with other non-conventional technologies will gain still further recognition as a powerful ally to green chemistry and beyond.

Acknowledgments

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