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Methyl esters production from Waste Cooking Oil catalysed by iron oxides supported on CaO: Cost and environmental impacts

Claudia Alanis [a], Rubi Romero [b], Liliana Ávila Córdoba [c], Reyna Natividad [b,*]

[a] *Urban and Regional Planning Faculty, Universidad Autónoma del Estado de México, Mariano Matamoros esquina Av. Paseo Tollocan, Toluca, 50130 Mexico*
[b] *Chemical Engineering Lab., Centro Conjunto de Investigación en Química Sustentable, UAEM-UNAM, Universidad Autónoma del Estado de México, Km 14.5 Toluca-Atlacomulco road, Toluca, 50200 Mexico*
[c] *Engineering Faculty, Universidad Autónoma del Estado de México, Cerro de Coatepec S/N Ciudad Universitaria, Toluca, 50110 Mexico*

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ABSTRACT

It was the objective of this work, to assess the midpoint environmental impacts of the catalyst synthesis stage and biodiesel production from waste cooking oil (WCO) as feedstock, depending on the catalyst source, i.e. Fe_2O_3 or $Fe(NO_3)_3 \cdot 9H_2O$, lime or waste clam shells, to produce the applied bifunctional catalyst based on iron and CaO. The cost of biodiesel production depending on the catalyst was also established. In the catalyst synthesis stage, the use of clam shells contributed the most to the midpoint environmental categories, mainly terrestrial ecotoxicity, freshwater ecotoxicity, marine ecotoxicity and human non-carcinogenic toxicity. In the stage of biodiesel production (esterification-transesterification reaction), the scenario contributing the lowest (20.95–22.16 %) to the midpoint environmental impacts is when using $Fe(NO_3)_3 \cdot 9H_2O$ and CaO as iron and lime precursors, respectively. Using waste clam shells increases the environmental impacts. Regarding costs, the clam shells lead to the most expensive process ($0.08 USD/MJ). The source of energy to conduct the biodiesel production was also assessed and it was found that the use of wind turbines leads to the lowest global warming potential (GWP), 11.6 g CO_2 eq·MJ-1, with the catalyst prepared with the iron salt and with the CaO from lime. The presented results were obtained with the commercial software SimaPro® version 9.6 PhD. For the inventory, experimental data obtained at laboratory scale and previously published were used.

It was concluded that based on environmental impacts and costs, it is recommended to use lime instead of clam shells waste as precursor of CaO and $Fe(NO_3)_3 \cdot 9H_2O$ as precursor of iron.

1. Introduction

Most industrial, transportation, and agricultural activities rely on fossil fuels (Ogunkunle and Ahmed, 2019). However, this energy model is becoming less and less viable, as fossil fuels are nonrenewable resources, and their use favors greenhouse gas generation. In this context, biodiesel has become a viable alternative to replace fossil diesel. It can be used in diesel engines without significant modifications. The use of this fuel in transportation, in 2020, during the Covid-19 pandemic, decreased due to mobility restrictions. However, in 2021, several causes motivated the biodiesel market to recover, including subsidies, tax credits, and global interest in decarbonization (OECD-FAO, 2022). Global biodiesel consumption in 2022 was 49.96 billion liters (IEA, 2023), and Europe, the United States, Brazil, and Indonesia are responsible for the growth of biodiesel consumption (IEA, 2022). The biodiesel market is estimated to grow at a compound annual growth rate (CAGR) of 7.76 % from 2021 to 2028 and reach $10.08 billion (Fortune business insights, 2024).

From an economic perspective, since the Ukraine-Russia conflict, the costs of raw materials (corn, sugar cane, and vegetable oil) have risen due to shortages or difficulties in distribution chains. This has caused an increase in the prices of biofuels (Esfandabadi et al., 2022) and this is one of the main reasons for European countries starting to use waste cooking oil (WCO) as feedstock for biodiesel production. For WCO, approximately 16.5 million tons are produced globally each year (Hosseinzadeh et al., 2022).

From an environmental point of view, the use of WCO to produce biodiesel has attracted attention because has a negative environmental potential effect (Alanis et al., 2022; Chung et al., 2019) Biodiesel from waste cooking oil is a second-generation biofuel and an alternative for

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* Corresponding author.
E-mail address: rnatividadr@uaemex.mx (R. Natividad).

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transportation sustainability, renewability, biodegradability, and availability purpose (Hajjari et al., 2017). The process to produce biodiesel can be either homogeneous or heterogeneous (Mandari and Devarai, 2021), with the latter offering the advantage of catalyst re-use and higher purity than with the former. The heterogeneous process implies the synthesis of a solid catalyst demanding reagents and energy in most of the typical cases, like co-precipitation and incipient wetness. Nowadays, there is a wide variety of heterogeneous catalysts to accelerate biodiesel production from WCO and the literature on this regard is vast.

The biodiesel production from WCO proceeds mainly by two stages, i.e. esterification of free fatty acids (FFA) and transesterification of triglycerides. In a typical process, the former is conducted using an acid catalyst and then the transesterification occurs by adding a basic catalyst. Nowadays, however, there are bifunctional heterogeneous catalysts that have been demonstrated to be able to reduce one stage of the typical biodiesel production process since allow to concomitantly conduct esterification and transesterification reactions in the same reactor. In addition, a bifunctional catalyst eliminates the low catalytic activity commonly observed with acid catalysts (Dai et al., 2021). In this context, a bifunctional catalyst that has been demonstrated (Enguilo et al., 2021; Ceron-Ferrusca et al., 2021) to efficiently produce methyl esters from WCO is based on iron oxide supported on calcium oxide. (Alanis et al., 2022) conducted the life cycle assessment (LCA) of such a process based on the results of Enguilo et al., 2021. It was concluded that the iron precursor exerts and important effect on the mid-point environmental impact categories.

Regarding the catalyst synthesis stage for biodiesel production, it has been demonstrated (Al-Mawali et al., 2021; Al-Muhtaseb et al., 2021) that such a step implies the highest environmental burden of the process. In this sense, it has been suggested that the use of biological waste like cow teeth or food waste, like eggshells and oyster shells, as CaO precursor to conduct the esterification-transesterification of WCO may lead to lower the cost of biodiesel production and to decrease the associated environmental impacts (Alsaiari et al., 2023; Aworanti et al., 2023; Sochima et al., 2023). This, however, is still a hypothesis. Therefore, the objectives of this work were: *i)* to contrast the environmental impacts of synthesizing $Ca_2Fe_2O_5$—$CaFeO_3$/CaO catalyst using as precursors of iron, Fe_2O_3 and $Fe(NO_3)_3{\cdot}9H_2O$; and as precursor of CaO, lime and clam shells; *ii)* to establish whether the use of food waste, clam shells in this case, as precursor of CaO to prepare an iron and CaO based catalyst, does lead to a decrease in costs and environmental impacts of biodiesel production when using WCO. To achieve so, a life cycle assessment (LCA) of biodiesel production from WCO under three scenarios was conducted. In Scenario 1 (S1) and Scenario 2 (S2), the source of CaO was $Ca(OH)_2$ while in Scenario 3 (S3) was $CaCO_3$ from waste clam shells. For Scenario 1, Fe_2O_3 was used as precursor of the iron oxide while the precursor for the same phase for Scenarios 2 and 3, was $Fe(NO_3)_3{\cdot}9H_2O$. The experimental data for the inventory were taken from previous works (Enguilo et al., 2021) and (Cerón-Ferrusca et al., 2021), conducted by the authors research group. The difference between both works is the CaO precursor, being lime in the first one and clam shells in the second one.

It is worth noticing that both assessed aspects in this work, environmental impacts and costs, are relevant because they are two of the three parts in which sustainability is regularly discretized.

2. Methodology

To achieve objective one, a contribution analysis of mid-point environmental impacts was conducted based on ReCiPe midpoint method characterization per functional unit (1 g of prepared catalyst) for catalyst synthesis from shells ($CaCO_3$) and lime ($Ca(OH)_2$). A more detailed description is provided in Section 2.2.2. The commercial software used for this purpose was (SimaPro® version 9.6 PhD). The experimental data for the inventory were taken from previous works (Enguilo et al., 2021) and (Cerón-Ferrusca et al., 2021).

Strictly speaking, only in ~50 % of the works presented in Table 1 it can be said that biodiesel is obtained. According to ASTM D6751–07b, biodiesel is the set of monoalkyl esters of long-chain fatty acids derived from renewable lipids such as vegetable oils, which must comply with a minimum of 96.5 % methyl esters (EN-14,103) among other parameters. Nevertheless, the physicochemical characterisation of the product reported in the selected experimental studies (Enguilo et al., 2021 and Ceron-Ferrusca et al., 2021) to conduct this work, indicated that biodiesel was produced (European Norm UNE-EN14214) in the work of Ceron-Ferrusca et al., 2021. In the case of Enguilo et al. (2021), however, the only parameter that is not within norm is the minimum content of methyl esters. Regarding acid value, this was found to be 0.54 mg KOH/g, which is very close to the maximum limit established by UNE-EN14214 (0.5 mg KOH/g). Regarding kinematic viscosity at 40 °C, this was found to be 5.0 mm^2/s which is also in the maximum limit of the established accepted range of 3.5–5.0 mm^2/s (EN ISO 3104). Thus, even when the methyl esters content is lower than the minimum established value, it was decided to call it biodiesel in this work since the difference is relatively low and the other two parameters are within limits.

The LCA to achieve objective 2, was conducted according to the International Organization for Standardization (ISO 14,040 and 14,044 standards) (ISO 14044, 2006). According to this methodology, Life Cycle Assessment implies the following stages, 1) definition of goal and scope, 2) life cycle inventory (LCI), 3) life cycle impact assessment (LCIA), and 4) results interpretation. The description of each one of these phases can be found below.

2.1. Goal and scope

The literature describes various types of bifunctional catalysts for obtaining biodiesel (Cerón Ferrusca et al., 2023). These used metals (acid sites) such as iron (Enguilo et al., 2021), strontium ((Al-Saadi et al., 2020), lanthanum (Rattanaphra et al., 2021) zirconium and tungsten (Mansir et al., 2021) are deposited on supports (basic sites) such as calcium oxide (Simbi et al., 2022), carbonates (Dai et al., 2021), and alumina (Al-Saadi et al., 2020), among others. CaO has gained

Table 1
Inventory data for biodiesel production from WCO, according to the functional unit (1 MJ) with a heterogeneous process with different catalyst and iron precursor.

	Inventory item	Unit	Scenarios			Data quality
			1	2	3	
Inputs	Methanol	g	13.72	15.59	18.86	Experimental
	Electricity consumption	kWh	1.06	0.56	1.06	Experimental
	Shells, calcium carbonate ($CaCO_3$)	g			1.99	Experimental
	Lime ($Ca(OH)_2$)	g	1.00	1.01		Experimental
	Pig iron (Fe_2O_3)	g	0.10			Experimental
	Vermiculite $Fe(NO_3)_3 \times 9H_2O$	g		0.10	0.20	Experimental
Outputs	Biodiesel	MJ	1	1	1	Experimental
	Methanol	g	1.51	1.66	3.02	
	FAME	%	89	88	84	Experimental
	Air emissions: evaporated (CH_3OH)	g	0.15	0.17	0.30	Reference (Chung et al., 2019)
	Spent catalyst	g	1.10	1.01	2.19	Experimental
	Methanol recovered	g	10.70	10.54	12.82	Experimental
Hours		h	2	1	2	Experimental

significant relevance because it can be obtained from low-cost or waste materials such as quicklime (Enguilo et al., 2021), waste clam shells (Cerón-Ferrusca et al., 2021), or eggshells (Tshizanga et al., 2017). However, few works evaluate the life cycle of biodiesel production using bifunctional catalysts from waste or low-cost materials with waste cooking oil (Alanis et al., 2022) it is essential to mention that most of these works only evaluate one stage of the process. Alanis et al. (2022), does take into account the separation of the by-product (glycerol), recovery of the alcohol and the catalyst, and concludes that the bifunctional catalyst using $Fe(NO_3)_3{\cdot}9H_2O$ presented less environmental impact with a contribution of 35 %.

The goal of this LCA was to assess and contrast the environmental impacts of biodiesel production with heterogenous catalysts under three different scenarios. The feedstock was WCO and the catalyst was a mixture of iron and calcium oxides; however, three different scenarios regarding the catalyst synthesis were assessed. In Scenario 1 and Scenario 2, the source of CaO was $Ca(OH)_2$ while in Scenario 3 was $CaCO_3$ from waste clam shells.

For Scenario 1, Fe_2O_3 was used as precursor of the iron oxide while the precursor for the same phase for Scenarios 2 and 3, was $Fe(NO_3)_3{\cdot}9H_2O$. The catalyst loading was 7 wt%, iron content (III) (10 wt %) and molar ratio methanol/WCO 12:1. For biodiesel production, in a similar way than in other works (Alanis et al., 2022; Caldeira et al., 2015; Pasha et al., 2021; Sheinbaum et al., 2013), the functional unit (FU) was 1 MJ of energy from biodiesel produced from WCO produced in food courts, based on its calorific value (37.27 MJ $L - 1$). The study was from gate to gate processes directly involved in producing biodiesel from WCO such as raw material and catalysts (lime $Ca(OH)_2$ and clam shells ($CaCO_3$) disposed in restaurant), including stages of catalyst synthesis and reusability; and esterification-transesterification reaction, leaving outside of the limits of the system, WCO pre-treatment, the mixing operations, loading and use in vehicles.

2.2. System description

All the stages constituting the biodiesel production system as well as the system boundary are shown in Fig. 1, where two-unit operations are observed: catalyst synthesis and reusability; and biodiesel production with WCO via esterification-transesterification catalysed by heterogeneous bifunctional catalysts synthesized under three scenarios. Under Scenario 1, the precursors of the catalyst are Fe_2O_3 and $Ca(OH)_2$; in Scenario 2, the catalyst precursors are $Fe(NO_3)_3{\cdot}9H_2O$ and $Ca(OH)_2$; in Scenario 3 the precursors are $Fe(NO_3)_3{\cdot}9H_2O$ and $CaCO_3$ from waste clam shells. It is worth pointing out that the use of waste clam shells as raw material for the catalyst synthesis, implies a pre-treatment to remove soluble salts, solid particles, and moisture. Since we are dealing with a residue, up-stream processes (oil extraction, cultivation, or mineral extraction) were not considered for the analysis. The outputs included air emissions of methanol (CH_3OH) and the emitted gases (CO_2) during catalyst synthesis, specifically in the activation stage by calcination. There were also considered as output wastewater emissions, spent catalyst, glycerol ($C_3H_8O_3$) and fatty methyl esters content (FAMEś%) of biodiesel production. The outputs associated to electricity consumption were the emitted gases per produced and consumed kWh. It is also worth noting that emulsification or saponification problems were not observed during the reaction stage.

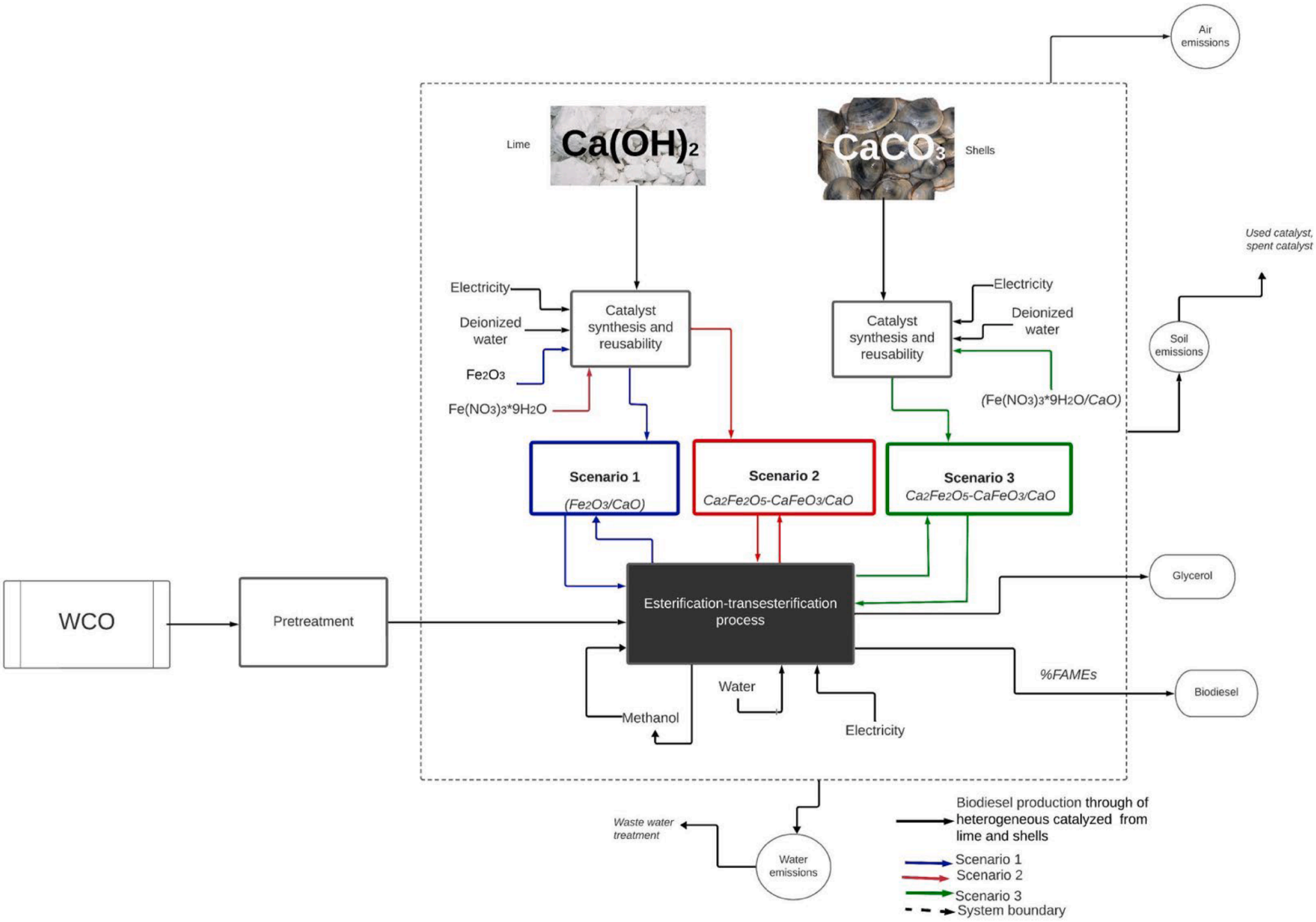

Fig. 1. System boundary of biodiesel production from waste cooking oil through esterification-transesterification reactions catalyzed by iron oxides on CaO (from lime and clam shells).

In Fig. 1, the system boundary is indicated by a black discontinuous line while the solid lines are delimiting independent subsystems (pretreatment, catalyst synthesis and reusability, and the reaction stage).

The feedstock, WCO, was collected from food courts. The fatty acid composition of this oil was determined by gas chromatography and the results were: lauric (C12:0) (0.03 %), myristic (C14:0) (0.16 %), palmitic (C16:0) (12.03 %), palmitoleic (C16:1) (0.17 %), margaric (C17:0) (0.12 %), stearic (C18:0) (4.40 %), oleic (C18:1) (23.58 %), linoleic (C18:2) (52.48 %), arachidic (C20:0) (0.33 %) and linoleic (C18:3) (6.65 %) (Enguilo et al., 2021). The WCO was a recycled product with the Cut-off method, so the emissions associated with the recycled product are only those of its collection.

2.2.1. *Catalyst synthesis and reusability*

As observed in Fig. 2, catalyst synthesis and reusability, is a subsystem in the production of biodiesel from WCO. The catalyst synthesis was conducted by using different iron and Ca precursors. There are in Fig. 2 depicted the stages for each catalyst synthesis depending on the CaO precursor, $Ca(OH)_2$ (green dashed line) or $CaCO_3$ from clam shells (orange dashed line). The inventory for this stage is also included in Fig. 2. It can be observed that the latter implies six additional steps compared to lime. These stages being washing, filtration, drying, crushing, grinding and homogenization. The first stage, washing of the waste clam shells, was conducted during 1.5 h, with an input of 1.5 L of deionized water, and then the shells waste was placed in a sonicator

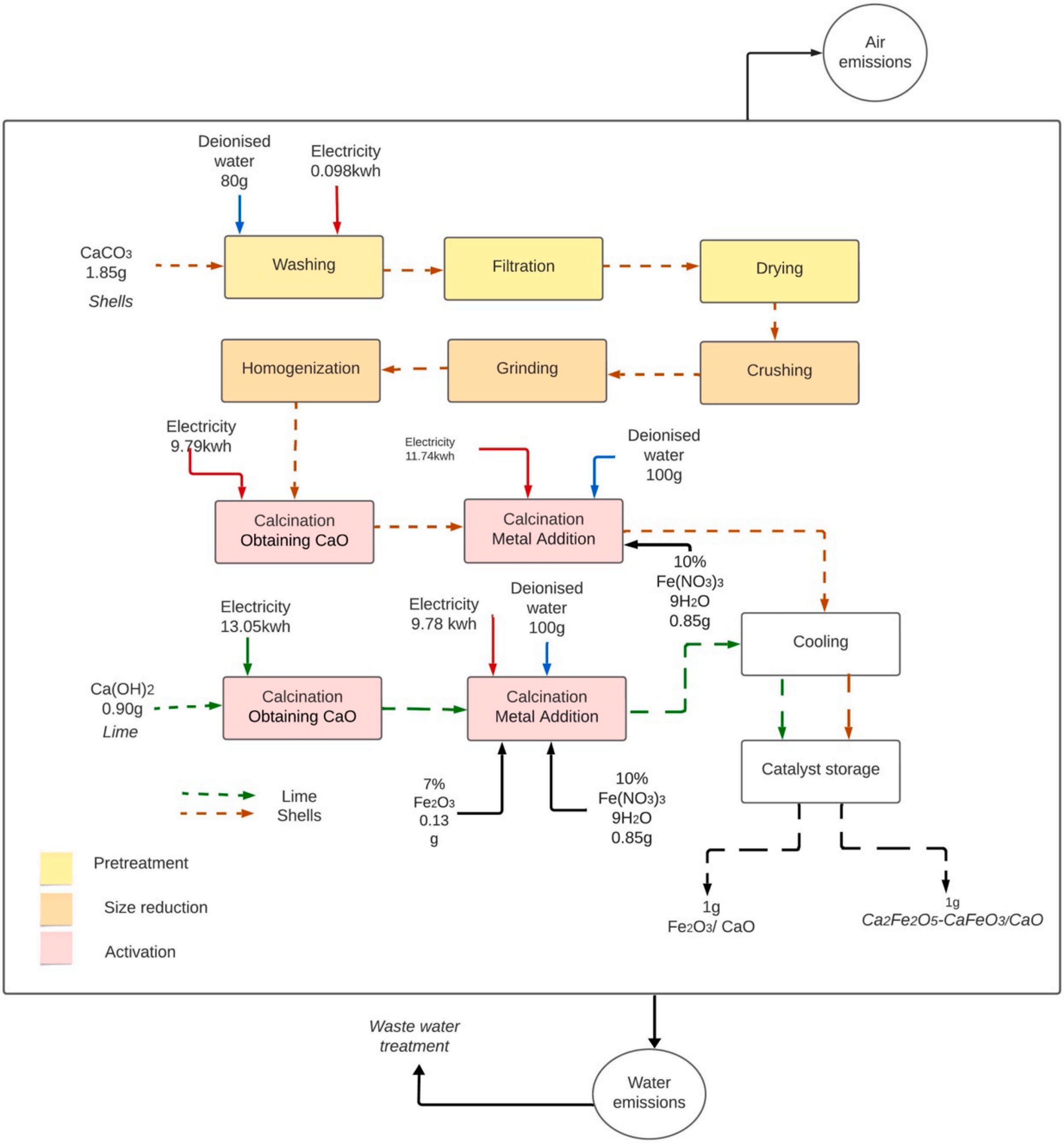

Fig. 2. Stages of catalyst synthesis from clam shells ($CaCO_3$) and lime ($Ca(OH)_2$).

during 20 min to remove the adhered salts and then the shells were dried under room conditions. Then the shells size was reduced by crushing and grinding and homogenized with a 40 μm mesh size. To transform the Ca precursors into CaO, the solids from clam shells were calcined at a 900 °C for 6 h, and 900 °C for 8 h for lime, in a furnace where temperature was increased at a rate of 2 $^{\circ}C{\cdot}min^{-1}$, for the three scenarios. Then, CaO was dispersed into 400 mL of water during 5 min. Two solutions of Iron (III) (1.79×10^{-2} M) were prepared, one per each iron precursor, i.e., Fe_2O_3 or $Fe(NO_3)_3{\cdot}9H_2O$. The solution was dropwise added to the CaO slurry and the mixture was stirred for 4 h. The suspension was filtered and the solid was dried overnight at 100 °C. Then the resulting material was calcined at 800 °C in a furnace for 5 h in scenarios 1 and 2, and it was calcined at 600 °C for 6 h in scenario 3. To reach this temperature, the furnace took 8 h since temperature was increased at 2 $^{\circ}C{\cdot}$ min^{-1}. This thermal treatment affects both, input, and output inventory, especially in the energy consumption and in the emissions items. The electricity consumption in this stage was for scenario 1 and 2, 9`.78 kWh and scenario 3, 11.74 kWh. During this stage, the transformation of $CaCO_3$ and $Ca(OH)_2$ to CaO proceeds by means of temperature. It is worth noting that this is the stage with the highest energy consumption due to the use of a furnace, with emissions of carbon dioxide (CO_2) gas and water vapor. The last stages were cooling and bifunctional catalysts storage.

2.2.2. Biodiesel production: esterification-transesterification reaction stage

According to Cerón et al. (2021) and Enguilo et al. (2021), the esterification and transesterification reaction for the biodiesel production from WCO with bifunctional catalyst, was conducted in a 250 mL batch reactor made of glass, with baffles and connected to a methanol-reflux system. The reactor was operated at constant temperature ($T = 60$ °C). The reaction volume was 150 mL. The type of catalyst dictated the electricity consumption time that was from 1 to 2 h depending on the required time to reach the maximum yield (Cerón et al., 2021; Enguilo et al., 2021). For reaction, the following equipment was also employed: heating and stirring thermal plate (600–1000 rpm), recirculation system, rotary evaporator, centrifuge, and vacuum pump. At the end of each reaction, the spent catalyst and produced glycerol were recovered by centrifugation and settling, respectively. Unreacted methanol was separated by evaporation under vacuum.

2.3. Life cycle inventory

The life cycle inventory (LCI) with experimental primary quality, was obtained in a heterogeneously catalysed process to produce biodiesel with WCO as feedstock. The inventory data presented in Table 1, was calculated according to experimental results previously reported by Cerón-Ferrusca et al. (2021); Enguilo et al. (2021). The variable of these studies was iron and calcium precursor and the resulting materials of scenario 1, 2 and 3, the source of CaO, was from the lime production hydraulic ($Ca(OH)_2$) and calcium carbonate ($CaCO_3$). Methanol ACS (CH_3OH) 99.9 % was supplied by Fermont. Iron (III) nitrate ($Fe(NO_3)_3{\cdot}9H_2O$) 99.0 % was bought in MERCK and iron (III) Oxide (Fe_2O_3) 99.0 % was obtained from Reasol. The waste shell of the american clam is made up largely of $CaCO_3$, which makes the residues thereof (empty shells), an ideal raw material for obtaining CaO. To conduct the simulation and obtain the environmental impacts of the different scenarios, a commercial software was used (SimaPro® version 9.6 PhD). A mexican database (MX) of Electricity Federal Commission (CFE, for its Spanish acronym), Ecoinvent v.3 database, was used for the modelling of electricity and fuel consumption emissions. Ecoinvent v.3 RoW and GLO database were applied to establish the disposal scenarios of wastewater and solid waste. The LCA system includes material and energy inputs and environmental emissions (water, air and solid) for each stage. The iron precursor, Fe_2O_3, for scenario 1, was considered from the pig iron market (Flowers et al., 2021), for scenario 2 and 3, (iron precursor: $Fe(NO_3)_3{\cdot}9H_2O$), vermiculite market was elected (Chen et al., 2010). The allocation was made based on energy output (1 MJ) in biodiesel.

2.4. Life cycle assessment (LCA)

The SimaPro® version 9.6 PhD (PRé Sustainability, 2022) software was used to quantify the environmental impact categories, the whole processes have consistency, accuracy and specification of data collection. The database of inventory models for inputs were obtained from Ecoinvent v.3 (Ecoinvent, 2019). The method to assess the environmental impact categories was ReciPe Midpoint (H) V1.06 / World (2010) H, and the assessed categories were the following eight: Global warming potential (GWP), Fine particulate matter formation (OFP), Terrestrial ecotoxicity (TETP), Freshwater ecotoxicity (FETP), Marine ecotoxicity (METP), Human non-carcinogenic toxicity (HTPc), Fossil resource scarcity (FFP) and water consumption (WCP). These categories were elected based on previous LCA related to biowaste and biodiesel. Regarding the former, LCA focuses on impact categories such as energy and water use, GHG emissions, land occupation and nutrient depletion (Batool et al., 2024). About environmental impacts of transesterification based on LCA studies, the categories most studied of biowaste are global warming potential, water depletion and particulate matter formation (Arfelli et al., 2023; Papadaskalopoulou et al., 2019; Zeller et al., 2020); and for WCO are human toxicity potential, terrestrial, freshwater and marine ecotoxicity (Alanis et al., 2022; Bhonsle et al., 2022a; Heidari-Maleni et al., 2024; Hosseinzadeh-Bandbafha et al., 2022b; Mohd YUSOF et al., 2019). The categories include climate change, fine emissions (input-related emissions and process-specific emissions) water and soil toxicity, carcinogenic toxicity and fossil fuels scarcity. According to the referred studies, the elected categories are usually the most affected ones.

2.5. Interpretation

To analyse the life cycle impact assessment results, a sensitivity analysis was conducted to evaluate the influence of variables on the energy source (fossil fuels, photovoltaic, and wind turbine) for each scenario based on allocation energy. The environmental impacts to evaluate the sensibility was GWP and was contrasted with sustainability criteria already established by the Renewable Energy Directive (RED) (European Parliament, 2018). The environmental impact of GWP in $kgCO_2eq$, is based on the evaluation of CO_2 emissions, saving targets and comparison to fossil fuels, reported in a previous LCA study (Kiehbadroudinezhad et al., 2023).

Uncertainty analysis is another important issue in LCA: average data is usually used without considering the associated variability, and the results can be misleading when comparing systems (Escobar et al., 2014). In this work, LCI uncertainty analysis with Monte Carlo simulation was used to determine the uncertainties of the LCIA results introduced by the statistical variability gaps in the LCI data. Because the uncertainty of the LCI data is a probability distribution, the Monte Carlo function analysis in software SimaPro® version 9.6 PhD, was evaluated with 1000 iterations at the 95 % confidence interval (significance level or α is 0.05), also considered to assess the toxicity of the scenarios with the probability distribution of chemical products, energy consumption. In Monte Carlo, the absolute uncertainty can be determined after recalculating the procedure. The mean is the average of the environmental values. The median is the middle environmental value. The coefficient of variable (CV) is the ratio between the standard deviation and the mean or a normalized indicator of dispersion in category indicator results (Guo and Murphy, 2012).

3. Theoretical background

There is depicted in Fig. 3, a word cloud related to the search of literature on biodiesel where the font size is directly related to the times that the keyword comes out and therefore indicating trends in biodiesel

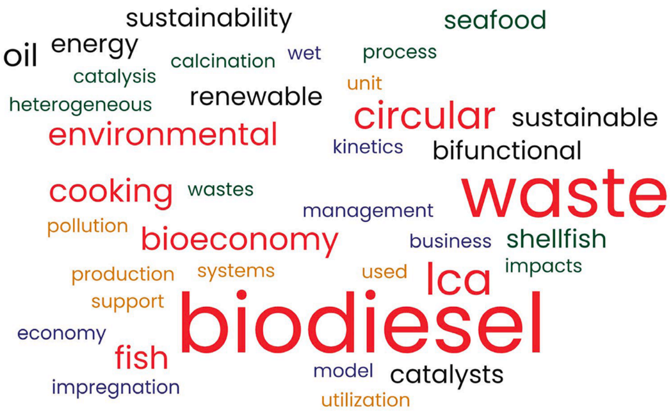

Fig. 3. Word-cloud representing most frequent author's keywords around biodiesel production. Source: own.

production. It can be observed that the words "waste", "circular", "bioeconomy", "cooking", "oil", "sustainability", "seafood" and "shellfish" stand out. It is not surprising that most of these words are related to energy recovering from organic waste, thus generating a form of renewable energy derived from biological sources (bioenergy). The motivation for this is economic and environmental. The former falls within the field of bioeconomy, which entails harnessing renewable biological resources like forests, crops, animals, microorganisms, waste and their by-products and transforming them into enhanced products such as bioactive compounds, bio-based items, and bioenergy (McCormick and Kautto, 2013; Patermann and Aguilar, 2018).

The use of WCO as feedstock instead of virgin oil to produce biodiesel is an efficient method to reduce material cost in biodiesel production, it is likely to lower the biodiesel production cost by 60–90 % (Meng et al., 2008). This is relevant in the context of circular economy, which in bioenergy refers to a sustainable approach where biomass resources are managed in a closed-loop system, minimizing waste, and maximizing resource efficiency (IRENA, 2020).

Table 2 summarizes the published works related to the biodiesel (methyl esters) production from WCO and catalysed by CaO based catalysts. It can be observed that in all cases the produced methyl esters content is 90 % at least at moderate reaction times and relative low temperatures. This is what makes attractive CaO based bifunctional catalysts. A bifunctional catalyst is characterized by having both, Lewis and Brönsted acid sites on their surface and this characteristic enhances the ability of the catalyst to withstand high levels of FFA content in the feedstock compared to basic catalysts.

4. Results and discussion

4.1. Biodiesel production: midpoint impact assessment, scenario 1, 2 and 3

For biodiesel production, there are depicted in Fig. 4, the contribution of the inventory items in which there are significant differences among scenarios (see Table 3). As observed in Fig. 4, Scenario 2 showed a contribution of 20.95–22.16 %; scenario 3, 35.86–39.47 % and scenario 1, 39.59–41–98 %, to environmental impacts across all categories. The hotspots impact mainly affected were GWP, TETP and FFP. The contribution to these impacts is below further discussed. There is in Table 3, all the calculated environmental impact categories. These values are ascribed mainly to electricity consumption for transesterification and chemical inputs such as methanol (Al-Muhtaseb et al., 2021).

It is worth noticing that in all impact categories, scenario 2 (catalyst prepared from iron nitrate and lime) provides the lowest contribution. However, when comparing the estimated values for scenario 1 (catalyst prepared from ferric oxide and lime) and scenario 3 (catalyst prepared from iron nitrate and calcium carbonate from waste shells), the contribution to environmental impacts of scenario 3 is very similar to that provided by scenario 1 although scenario 3 is advantageous since uses a waste to prepare the catalyst.

Table 2
Methyl Esters content produced from WCO using CaO-based bifunctional catalysts.

Catalyst	Reaction Conditions	Methyl Esters Content (% FAME's)	References
Fe_2O_3/CaO	$T = 65$ °C; CC = 1% wt; M:o = 15:1; $t = 3$ h	92	(Ezzah-Mahmudah et al., 2016)
4Mn-6Zr/CaO	$T = 80$ °C; CC = 3% wt; M:o = 15:1; $t = 3$ h	92.1	(Mansir et al., 2018)
CaO(10 %)-Fe_2O_3(10 %)	$T = 65$ °C; CC = 3% wt; M:o = 18:1; $t = 3$ h	98.3	(Ibrahim et al., 2022)
CaO/Al_2O_3	$T = 60$ °C; CC = 2.5% wt; M:o = 12:1; $t = 3$ h	98.23	(Simbi et al., 2022)
CaO/Al_2O_3	$T = 65$ °C; CC = 1% wt; M:o = 11:1; $t = 4$ h	98	(Kesserwan et al., 2020)
Sn-CaO	$T = 85.15$ °C; CC = 2.2% wt; M:o = 16.1:1; $t = 3.42$ h	97.39	(Bharti et al., 2022)
CaO—CeO_2	$T = 70$ °C; CC = 4% wt; M:o = 9:1; $t = 1.2$ h	90.14	(Suryajaya et al., 2021)
$Ca_2Fe_2O_5$—$CaFeO_3$/CaO	$T = 55$ °C; CC = 6% wt; M:o = 12:1; $t = 5$ h	98.8	(Ceron-Ferrusca et al., 2021)
$Ca_2Fe_2O_5$—$CaFeO_3$/CaO	$T = 60$ °C; CC = 5% wt; M:o = 12:1; $t = 2$ h	90	(Enguilo et al., 2021)

4.1.1. Global warming potential (GWP)

This environmental impact category is a measure of the generated emissions of greenhouse gases by the activity or process that is being assessed, biodiesel production in this case. This measure is important since greenhouse emissions lead to rising temperatures and disruptions in Earth's climate patterns, primarily due to the absorption of heat radiation by the atmosphere, commonly referred to as global warming. GWP is typically expressed in kilograms of equivalent carbon dioxide. This metric holds particular significance as an indicator of the carbon footprint, especially when assessing the sustainability of processes associated with fossil fuel production or energy generation (Chung et al., 2019). The principal input to the environmental impact for biodiesel production, is the use of electricity, mainly produced from fossil fuels in Mexico (Santoyo et al., 2014). The highest GWP was for S1 (0.68 kg CO_2 eq·MJ-1), S3 (0.61 kg CO_2 eq·MJ-1) and finally S2 (0.36 kg CO_2 eq·MJ-1), the GWP is reduced by about half when the iron nitrate is used as precursor of the catalyst with lime, and the FAMES% was 88 % after 1 hour of reaction with S2. A similar FAMES% was obtained with S1 and S3 but after two hours of reaction time, this makes electricity consumption double for those two scenarios. This difference can be ascribed to a different basic/acid sites ratio attained on the catalytic surface under every scenario (Enguilo et al., 2021). For the three scenarios, the second input which contributes the most to global warming was the use of methanol, because its production requires energy from fossil fuels, which results in greenhouse gas emissions.

4.1.2. Terrestrial ecotoxicity (TETP)

Terrestrial ecotoxicity accounts for the environmental persistence (fate), accumulation in the human food chain (exposure), and toxicity

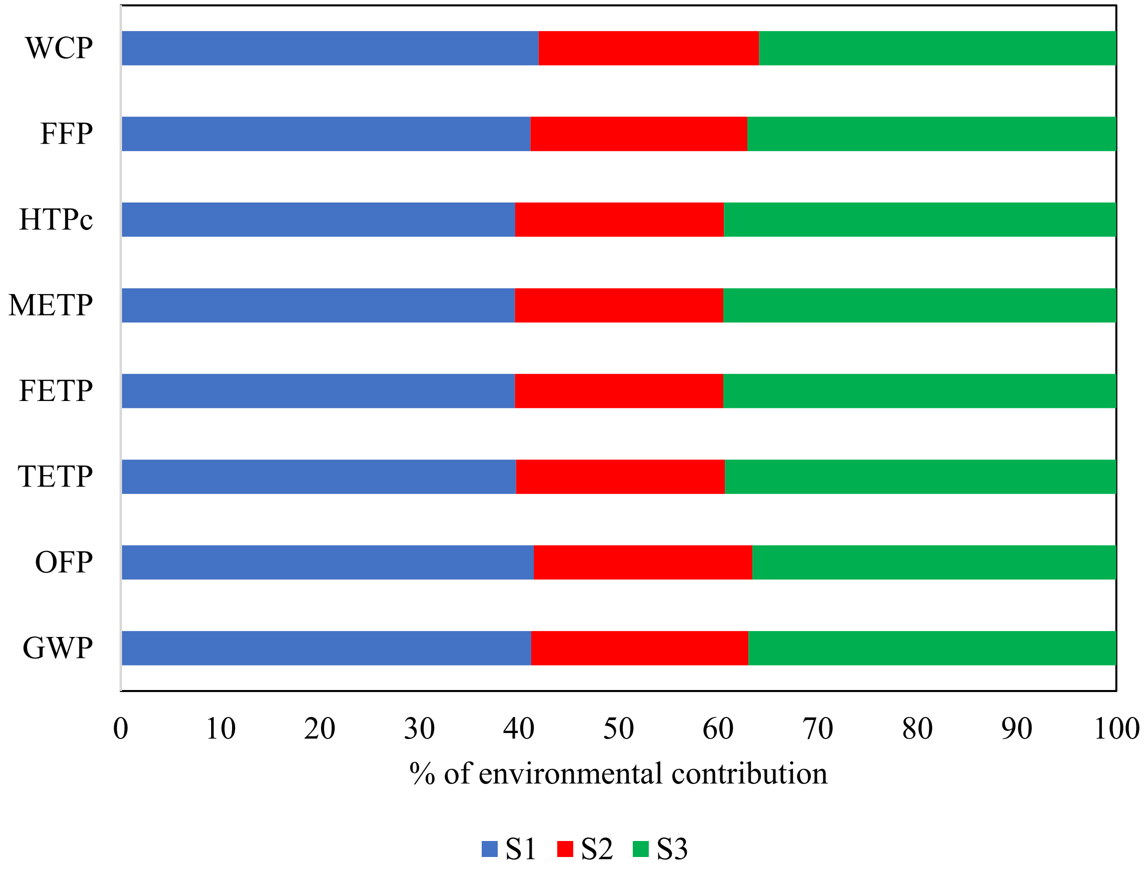

Fig. 4. Contribution of scenario 1 (S1), scenario 2 (S2) and scenario 3 (S3) to environmental impact categories of biodiesel production from waste cooking oil, assessed by the ReCiPe midpoint method.

Table 3
Results of midpoint environmental impact categories associated with biodiesel production from waste cooking oil based on Recipe 2016 (FU = 1 MJ).

Category impact	Unit	Scenarios		
		1	2	3
Global warming potential	kg CO_2 eq	0.68	0.36	0.61
Fine particulate matter formation	kg $PM_{2.5}$ eq	1.32E-03	6.99E-04	1.16E-03
Terrestrial ecotoxicity	kg 1,4-DCB	1.47	0.78	1.45
Freshwater ecotoxicity	kg 1,4-DCB	0.04	0.02	0.04
Marine ecotoxicity	kg 1,4-DCB	0.05	0.03	0.05
Human non-carcinogenic toxicity	kg 1,4-DCB	0.42	0.22	0.42
Fossil resource scarcity	kg oil eq	0.21	0.11	0.19
Water consumption	m^3	1.40E-03	7.41E-04	1.20E-03

(effect) of a chemical (Huijbregts et al., 2016). In this sense, S1 contributes the highest (1.47 kg 1,4-DCB·MJ-1), followed by S3 (1.45 kg 1, 4-DCB·MJ-1) and the last is S2 (0.78 kg 1,4-DCB·MJ-1). Thus, the S2 exerts the lowest impact and this is because the process of purifying the final product is much simpler due to the easy separation of the catalyst (Fe $(NO_3)_3 \cdot 9H_2O/CaO$) and the separation of methanol and glycerine, which directly reduces the material and energy expenditure, which have high contributions to creating indicators of toxic environments (Motevali et al., 2023). Other LCA studies, the TETP has importance of considering, because mainly due to the negative effect of the heavy metals presence in the sludge applied for the agricultural purpose (Yoshida et al., 2018).

4.1.3. Fossil resource scarcity (FFP)

The FFP interpretation was the fossil fuel extraction and costs in production technique location (Huijbregts et al., 2016).On the other hand, the impending exhaustion of fossil fuel reserves and the anticipation of increased costs have never been more critical. This underscores the urgency of transitioning to an alternative energy system (Norouzi, 2022). For S1 (0.21 kg oil eq ·MJ-1), then S3 (0.19 kg oil eq·MJ-1) and the last, S2 (0.11 kg oil eq·MJ-1). In the future, upcoming energy systems will need to undergo substantial and foundational transformations, incorporating non-carbon energy sources like solar, wind, geothermal, and biomass (Norouzi, 2022).

4.2. Catalyst synthesis: assessment of midpoint environmental impact categories

Two primary sources were analysed with respect to their environmental effects. Fig. 5 shows the comparative environmental impacts, and it can be observed that the catalyst synthesis from waste clam shells ($CaCO_3$) had the highest environmental contribution in all impact categories. This is because of important inputs at the pre-treatment stage of shells: deionised water and electricity consumption on the washing stage. For lime ($Ca(OH)_2$), the contribution to environmental impacts was between 30.45–94.62 %. The electricity consumption in the pre-treatment and activation stage to obtain CaO, and metal addition (Fe_2O_3 or Fe $(NO_3)_3 \cdot 9H_2O$) were the main contributors to overall environmental impacts with any of the two assessed primary sources; the GWP for shells was 2.77 kg CO_2 eq and lime 2.62 kg CO_2 eq. Rahman et al. (2022) reported that the high impacts of organic precursors primarily (FeOx) reflect the input of chemicals and organic solvents required for their production. These conditions differ according to the type of waste-derived catalyst and biodiesel feedstock. Thankfully, these waste

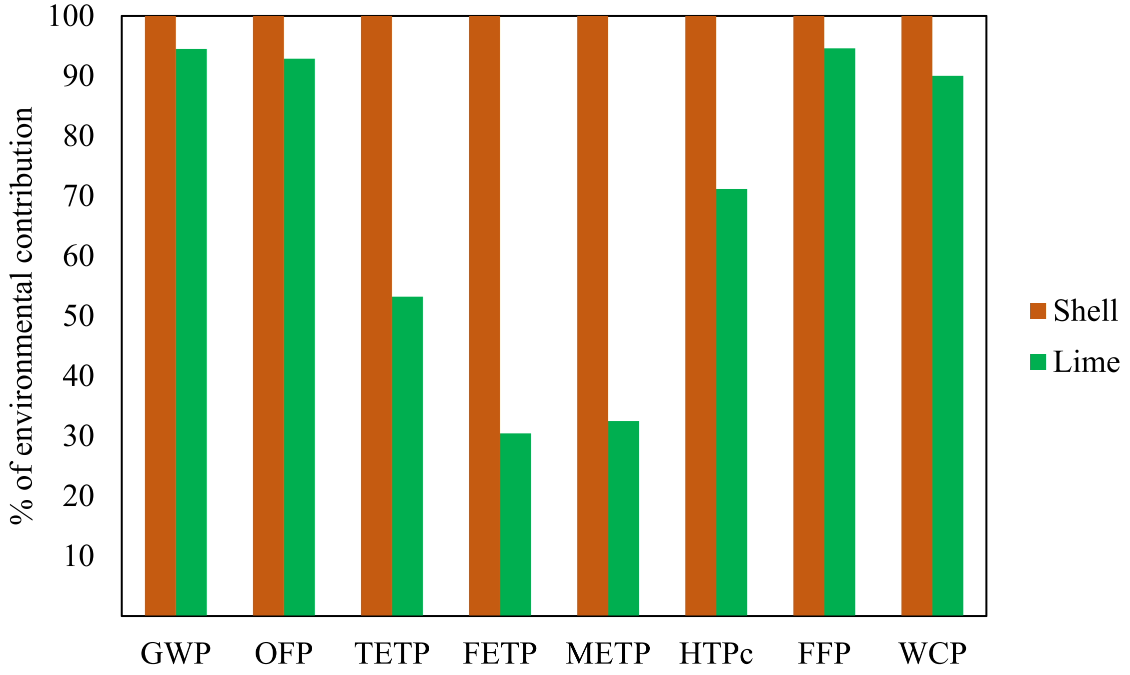

Fig. 5. Contribution analysis of mid-point environmental impacts based on ReCiPe midpoint method characterization per functional unit (1 g of catalyst) for catalyst synthesis from shells ($CaCO_3$) and lime ($Ca\ (OH)_2$).

materials have demonstrated their potential to serve as a starting material for producing catalysts on a laboratory scale. For future improvements regarding synthesis conditions, the use of less energy and chemicals will potentially decrease health and environmental impacts. This will facilitate the making of informed choices when expanding the process for industrial implementation.

There is in Table 1S the value of all the midpoint impact categories shown in Fig. 5 and associated with the production of 1 g of catalyst.

4.3. *Sensitivity analysis*

The Renewable Energy Directive (RED) has set a standard (15–20 gCO_2eq MJ-1) for greenhouse gas emissions in typical biofuel systems based on energy content allocation (European Parliament, 2018). This measure has encouraged the adoption of biofuels instead of fossil fuels. The newly defined objective is to ensure that, by 2030, at least 32 % of the EU's total energy demand is fulfilled through renewable sources. Renewable energy in Mexico refers to the use of sustainable and clean energy sources to generate electricity and power various sectors of the country. Mexico has been making significant efforts to expand its renewable energy capacity in recent years, with a focus on reducing its carbon footprint and increasing energy independence. The Mexican government has implemented policies and initiatives to promote renewable energy development and reduce greenhouse gas emissions. These efforts align with global goals to climate change mitigation and transition to a more sustainable and environmentally friendly energy sector. In 2023, Mexico's Primary Energy Matrix structure consisted of 85 % fossil fuels and 15 % renewable sources (SENER, 2023a). It is worth noting that the share of renewable energies in Mexico's primary energy matrix exceeds that of the United States (6 %) and China (6 %) (SENER, 2023a).

Because of the aforesaid and as strategy to decrease the reliance on fossil fuels, a sensitivity analysis was conducted to establish the effect of using renewable sources of energy such as photovoltaic and wind, on the impact category of GWP. These renewable sources of energy were elected because according to the National Renewable Energy Inventory (NREI), the greatest proven potential for electricity generation, supported by technical and economic studies confirming its feasibility, is found in wind and solar energy (SENER, 2018). The sensitivity analysis was conducted for the biodiesel production stage only and not for the catalyst synthesis stage. Fig. 6 presents the results of such a study.

It can be observed in Fig. 6 that the GWP for any of the assessed scenarios (S1, S2 or S3), is reduced by approximately 92 % with photovoltaic energy and 98 % with energy from wind turbine. Adopting 100 % photovoltaic energy or wind energy, however, does not reflect real-world conditions and therefore a mixture of energy sources was included in the sensitivity analysis presented in Fig. 6. For any of the scenarios, GWP is observed to steeply decrease directly with the contribution percentage of the renewable sources.

There are, in Table 4, summarized the GWP values obtained with the different proposed mixtures. The mixture for 2024 was proposed based on the current ratio of wind energy to photovoltaic energy in Mexico. Nevertheless, Mexico committed to an unconditional 22 % reduction in its GHG emissions by 2030 and 50 % by 2050, compared to the baseline, which implies a 31 % reduction in fossil-based electricity generation, considering 2013 as the baseline year. This international commitment is supported by national regulations, which also include clean energy targets for the medium and long term: 2024, 2030 and 2050 (SENER, 2023b; WRI Mexico, 2020). This was considered for proposing the assessed mixtures for 2030 and 2050. An important reduction of GWP is observed in all essayed mixtures reported in Table 4, however, the most significant reduction is when using 50 % photovoltaic energy and 50 % wind energy, again without energy coming from fossil fuels. This combination was evaluated to compare with the reported in the literature by Kiehbadroudinezhad et al. (2023); who report 0.053 $kgCO_2eq$ •MJ-1 using photovoltaic and wind turbine energy. It is worth noticing that the values reported for S1, S2 and S3 are lower, and this can be ascribed to the activity of the assessed catalysts that allow to obtain a relatively high methyl esters content at lower reaction times and this implies less energy consumption. Energy allocation underscores the significance of clearly identifying, explaining, and justifying the selection of an energy consumption indicator (Arvidsson, 2021). In conclusion, the impact category of GWP can be reduced ca. 45 % by shifting from a carbon intensive electricity source to alternative energy sources combining fossil fuels (50 %) and renewable sources of energy (25 % photovoltaic and 25% wind energy) with lower carbon emissions.

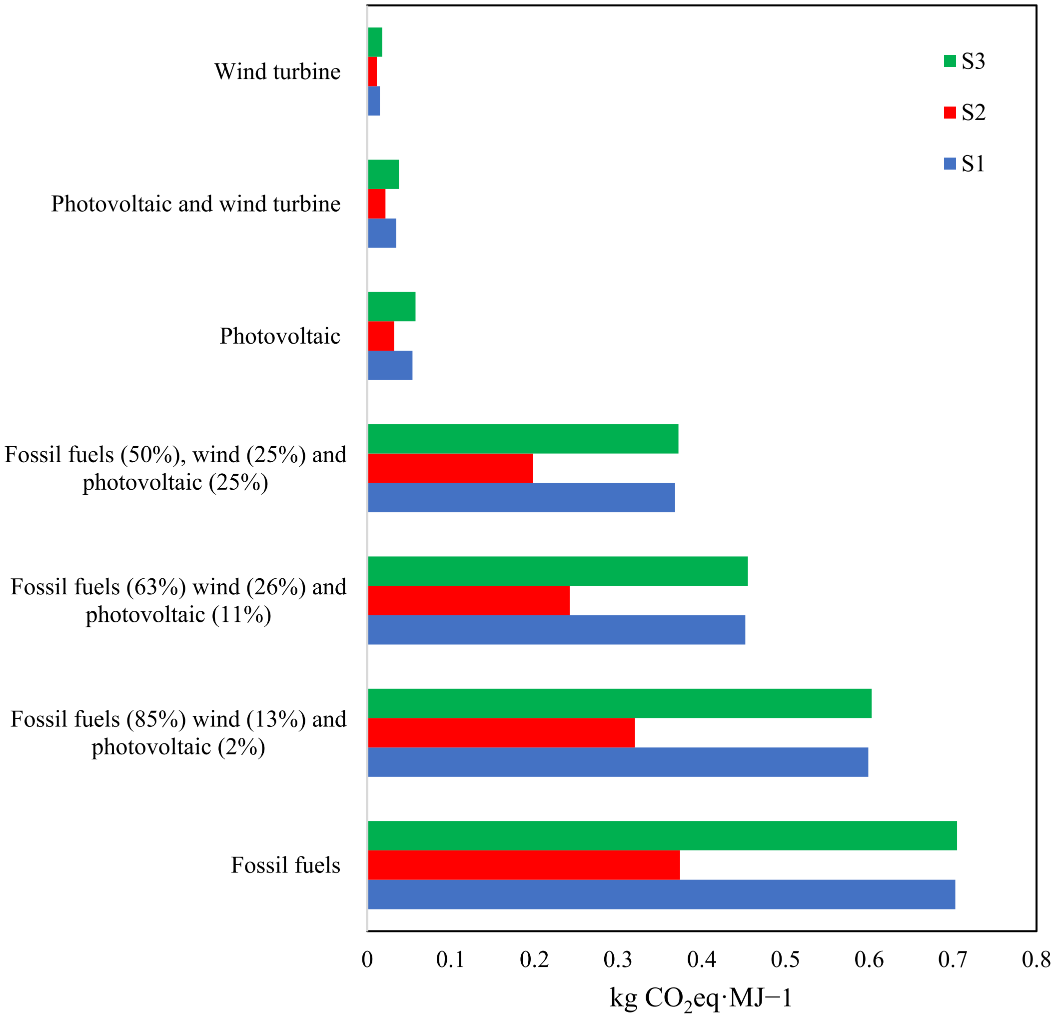

Fig. 6. Effects of electricity consumption by scenarios S1, S2 and S3, using fossil fuels and renewable sources (photovoltaic and wind turbine), on GWP (kg CO_2eq·MJ−1).

Table 4
GWP environmental impact of alternative energy sources.

Biodiesel production scenario	*Energy source*						
	Fossil fuels	*2024 Fossil fuels (85 %), wind (13 %) and photovoltaic (2 %)*	*2030 Fossil fuels (63 %), wind (26 %) and photovoltaic (11 %)*	*2050 Fossil fuels (50 %), wind (25 %) and photovoltaic (25 %)*	*Photovoltaic*	*Photovoltaic (50 %) and wind (50 %)*	*Wind turbine*
	kg CO_2 eq•MJ-1						
S1	0.680	0.599	0.452	0.368	0.048	0.0347	0.0152
S2	0.360	0.32	0.242	0.198	0.026	0.0218	0.0116
S3	0.610	0.603	0.455	0.372	0.049	0.0379	0.018
(Kiehbadroudinezhad et al., 2023)						0.053	

With time, the share of renewable energies as sources of clean energy generation is considered plausible and is expected to be facilitated by a constant reduction in costs. Additionally, the support policies for renewable energies in Mexico, implemented as part of the Energy Reform (DOF, 2015), will keep strengthening the energy market, making renewable energies highly competitive with conventional fuels in the electricity sector.

4.4. Uncertainty analysis

Monte Carlo analysis was additionally conducted to assess the level of uncertainty in every scenario. For three impact categories, i.e. GWP, Terrestrial ecotoxicity and Fossil resource scarcity. The results are summarized in Table 5. It is considered that Recipe method has significant uncertainty if the resultant value is between 0.36–0.38. Typically, uncertainty in each impact category is measured by dividing the standard deviation by the mean (Bhonsle et al., 2022b).

Table 5
Monte Carlo uncertainty analysis for three scenarios.

Impact category		Unit	Mean	Median	SD	CV	2.50 %	97.50 %	SEM	Uncertainty (SD/Mean)
Global warming	S1	kg CO_2 eq	0.71	0.67	0.25	35.81	0.33	1.33	0.01	0.36
	S2		0.36	0.34	0.14	37.53	0.17	0.70	0.00	0.38
	S3		0.67	0.62	0.24	35.81	0.32	1.30	0.01	0.36
Terrestrial ecotoxicity	S1	kg 1,4-DCB	1.52	1.45	0.55	35.85	0.70	2.85	0.02	0.36
	S2		0.78	0.74	0.29	37.67	0.37	1.50	0.01	0.38
	S3		1.44	1.34	0.52	35.90	0.70	2.79	0.02	0.36
Fossil resource scarcity	S1	kg oil eq	0.22	0.21	0.08	35.65	0.10	0.42	2.49E-03	0.36
	S2		0.12	0.11	0.04	36.92	0.05	0.22	1.35E-03	0.37
	S3		0.21	0.20	0.08	35.37	0.10	0.41	2.37E-03	0.35

4.5. Benchmarking

Bearing in mind that GWP is crucial to comprehensively assess the environmental benefits and potential trade-offs of biodiesel production, the GWP values calculated in this study were compared to other studies related to biodiesel production for equivalent functional units (MJ), especially when using waste cooking oil as feedstock and with heterogeneous catalysts. It is worth pointing out that albeit the large amount of literature related to biodiesel production from WCO, most of the works do not conduct a LCA and therefore could not be included in Fig. 7. In this figure, it can be observed that GWP varies in a range of 6.42E-04 to 1.02E-01 kg CO_2•MJ-1. It must be pointed out, however, that the value reported by Foteinis et al. (2020) and Kamal Pasha et al. (2024), was obtained with homogeneous catalysts. The reason to include these values was that they were some of the lowest found with homogeneous catalysts and therefore this extends the spectrum of the present comparison. Nevertheless, it should also be considered that the values obtained in this work (S1, S2 and S3), as well as those from (Alanis et al., 2022; Al-Muhtaseb et al., 2022; Al-Muhtaseb et al., 2021; Khan et al., 2022); and Notarnicola et al. (2023), were obtained at a laboratory scale. In the case of this work and (Alanis et al., 2022), the values in Fig. 7 are assuming the use of photovoltaic energy consumption instead of fossil fuels (see Table 4). The works of Chung et al. (2019), Foteinis et al. (2020) and Kamal Pasha et al. (2024), are at industrial scale.

4.6. Techno-economic assessment

Transesterification is the most widely used and established method for producing biodiesel due to the cost-effective, reliable, and relatively simple process (Singh et al., 2020). The cost and energy viability of large-scale commercial processes must be determined through techno-economic analysis (TEA) (Gowthama Krishnan et al., 2024). Biodiesel quality is improved by the reusability of methanol and catalyst, which also supports reducing the operating cost(Naveenkumar and Baskar, 2020). In the biodiesel production context, it is a common thought that utilizing low-cost materials as feedstock, catalysts or catalyst precursors like waste shells, will minimize biodiesel production costs. According to LCA findings, enhancing the economic and environmental viability of WCO biodiesel hinges significantly on factors such as the purchase price of WCO, reduction in transportation distance, and improvement in energy conversion rates. LCA analysis indicates that conventional feedstock and the associated oil extraction process constitute over 70 % of the total production cost. Hence, utilizing waste by-products as feedstock and catalysts is emerging as the preferred approach for cost reduction in biodiesel production (Gowthama Krishnan et al., 2024). However, this manuscript has shown otherwise.

Fig. 8 depicts the costs of biodiesel production as a function of every scenario. It can be observed that the lowest cost was S1 $\$USD\cdot L^{-1}$ 0.75, then S2 $\$USD\cdot L^{-1}$ 0.76 and S3 $\$USD\cdot L^{-1}$ 0.85. The costs associated to each component of the process are shown in Table 2S and the equation to calculate the cost is also in the supplementary material. Actually, in Mexico the cost of production was reported between 0.46 and 0.78 $\$USD\cdot L^{-1}$ which can decrease if there are improvements in the production process, raw material placed at factory gate and that conversion ratio oil to biodiesel be 1:1 (by volume), but still remains competitive with conventional diesel, with a sale price of 1.08 $\$USD\cdot L^{-1}$(Masera

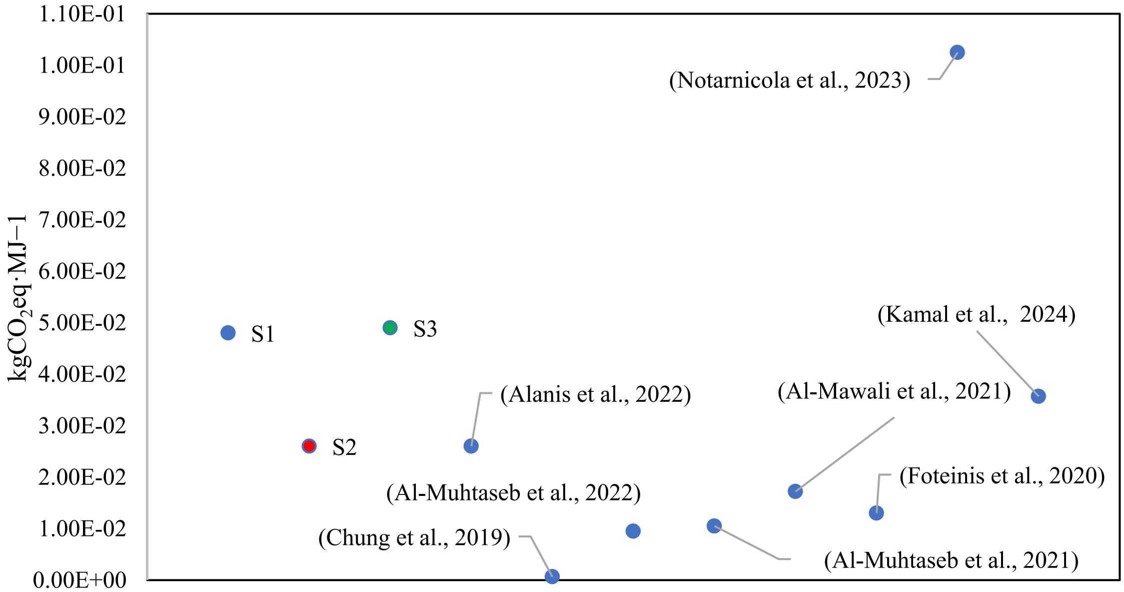

Fig. 7. Average Global Warming Potential emissions of biodiesel production from WCO as feedstock (kg CO_2•MJ-1).

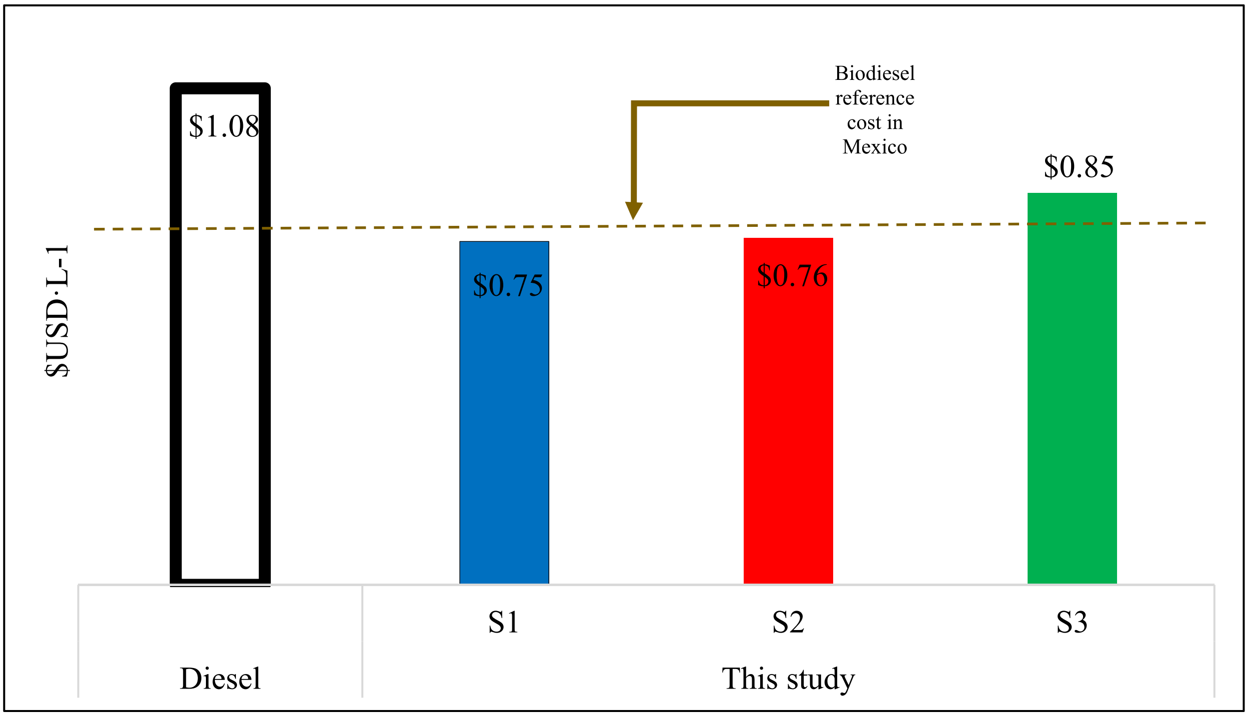

Fig. 8. Comparative costs of production for scenarios (S2, S2 and S3) with diesel and biodiesel reference.

et al., 2019, Riegelhaupt et al., 2016).

The results in Fig. 8 should be taken with caution since the costs of S1, S2 and S3, are based on laboratory scale. These are expected to decrease at industrial scale. The distribution from different cost input in biodiesel production used was from methanol, catalyst and electricity expense (Mohammadshirazi et al., 2014). The cost of biodiesel production for the studied scenarios, consider that the catalyst and methanol were easily recovered and catalyst is able to be reused at least three times (Enguilo et al., 2021). The energy consumption involved in the production of biodiesel from WCO exhibits a more advantageous non-renewable exergy cost, in other words, this process demands fewer non-renewable resources, primarily fossil fuels, in comparison to analogous products and the energy cost is minimum (De Mora et al., 2015). About the catalyst expense from precursors (Fe_2O_3 or $Fe(NO_3)_3{\cdot}9H_2O$), can vary significantly depending on location and specific requirements. The pig iron, which is a fundamental material in the production of steel, tends to be more widely available due to the large-scale steel production industry. Availability of vermiculite may be limited in certain areas, and it can be more accessible in regions where it is actively mined or imported for specific uses.

All the above can be still considered as basic research thus allocating the assessed technology in a Technology Readiness Level (TRL) of 3.

5. Conclusions

The midpoint environmental impacts of the catalyst synthesis for biodiesel production from waste cooking oil (WCO) were established. It was concluded that albeit clam shells being a residue, its use as precursor of CaO does not decrease the environmental impacts of the catalyst synthesis stage, on the contrary, it provides the highest contribution to midpoint environmental impact categories and therefore the use of lime as CaO precursor is recommended instead. The most affected impact categories were terrestrial ecotoxicity (TETP), freshwater ecotoxicity (FETP), marine ecotoxicity (METP) and human non-carcinogenic toxicity (HTPc).

A LCA and cost analysis of biodiesel production under three scenarios was also conducted depending on the used catalyst. It was concluded that the process leading to the lowest contribution to environmental midpoint categories is the catalyst synthesized using as precursors the iron salt and lime. Substituting fossils by photovoltaic or wind turbine as source of energy to conduct the biodiesel production, leads to a reduction on global warming potential (GWP) in all cases. The lowest GWP (11.6 $gCO_2eq{\cdot}MJ\text{-}1$) was found for the process catalysed with the material synthesized from iron nitrate and CaO from lime. The process catalysed with clam shells is the most expensive also.

To reduce the carbon footprint of the biodiesel production from waste cooking oil, two strategies are recommended: election of a highly active catalyst and mixing the energy sources (non-renewable with renewable) to produce 1 MJ from biodiesel. A mixture of 50 % energy contribution from fossil fuels, 25 % contribution of photovoltaic and 25 % of wind energy, leads to a 45 % reduction of carbon footprint.

The initiative presented in this work to produce methyl esters with renewable sources like WCO aligns with the United Nations Sustainable Development Goals (SDGs). The results obtained here contribute to achieve sustainable and affordable energy by transforming waste into energy through the implementation of simultaneous bioprocessing techniques. It directly contributes to several SDGs, including sustainable energy access (SDG7), responsible consumption and production (SDG12), and addressing climate change (SDG13).

CRediT authorship contribution statement

Claudia Alanis: Writing – original draft, Methodology, Investigation. **Rubi Romero:** Resources, Project administration, Investigation, Funding acquisition. **Liliana Ávila Córdoba:** Software, Investigation, Funding acquisition, Software, Investigation, Funding acquisition. **Reyna Natividad:** Writing – review & editing, Visualization, Supervision, Resources, Formal analysis, Data curation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.clcb.2024.100109.

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