

Selection of Hydrogen Donors for the Production of Renewable Diesel by *In Situ* Catalytic Deoxygenation of Palm Oil

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Recently, advanced biofuels have gained interest and importance; as a result, the world production of renewable diesel has quadrupled in the last 5 years. Renewable diesel is a type of biodiesel whose functional properties are similar, or even superior, to those of fossil diesel. It also looks more attractive than conventional biodiesel (FAME), as an option for the partial or complete replacement of liquid fossil fuels in the effort to reduce GHG emissions generated in the transport sector of diesel engines. However, when obtained by hydrotreating, the use of gaseous hydrogen makes the process lose sustainability due to its petrochemical origin (at least 90% comes from the reforming of naphtha) and high risk in its safe handling, besides increasing the costs due to its intensive operating conditions (temperature and pressure). A possible solution to this remarkable disadvantage is the generation of hydrogen *in situ*, which can be carried out by hydrogenation by catalytic transfer, using a donor, usually a solvent that transfers hydrogen. This is an interesting alternative for the hydrogenation of organic compounds, with substantial advantages over processes that use molecular hydrogen, in addition to moderate process conditions and reduction of the intensive energy consumption. In this sense, it is necessary to evaluate potential donors as renewable sources of hydrogen to obtain renewable diesel from palm oil. To carry out the previous analysis, in this study, a multicriteria decision method (MACBETH) is applied, using criteria such as renewable origin, decomposition products, cost, availability and toxicity, which are considered to select the most appropriate family of organic compounds that act as hydrogen donor: alcohols, polyols, aldehydes, ketones, carboxylic acids, condensed aromatic homologs, amines and alkanes. Results show that alcohols, polyols and carboxylic acids are the most suitable organic families for the generation of hydrogen *in situ* during hydroprocessing. According to sustainability and taking into account the atomic economy, the availability and biorefinery origin, it was found that the most promising compounds to use as donors in each of these families are the ethanol, glycerine and formic acid, which are then evaluated using thermodynamic analysis.

1. Introduction

The enormous influence that fossil fuels have had on the industrial development of society during the past century and a half is evident. However, this progress has been concomitant with the increase in environmental and social problems. Environmental problems are related to pollution and the generation of greenhouse gases (Klass, 1998; Lotfalipour, et al., 2010), while social problems are linked to the uneven distribution of gas and oil reserves globally. Countries with access to the largest reserves establish the price of these fuels (Hill, et al., 2006), generating inequality in industrial development among nations. Therefore, research has been focused on the creation of alternative energy sources that are environmentally friendlier, capable of being synthesized independently of the geopolitical zone, and, as far as possible, compatible with current technologies. Biofuels, such as biodiesel, are an important option for this purpose. Nowadays, 3 routes are used for the production of biodiesel (Bezergianni & Dimitriadis, 2013): (1) the conventional one, involving esterification with methanol in the presence of hydroxide (Azócar, et al., 2010; Vyas, et al., 2010); (2) the treatment of triglycerides with

hydrogen (Demirbas & Dincer, 2008; T. M. T. Kalnes, Marker, & Shonnard, 2007; T. N. Kalnes, Marker, Shonnard, & Koers, 2008); and (3) the synthesis by means of a Fischer-Tropsch process, starting from synthesis gas, preferably obtained from a gasification process (Dry, 2002; Leckel, 2009).

Conventional biodiesel, (FAME) presents, among others, some problems such as low stability, bad cold flow properties and a grade of incompatibility with the engine, so the concentration in the mixture with the fossil diesel is not advisable above 5% (Hass, 2004; T. N. Kalnes et al., 2008). Diesel produced by hydrogenation is known as renewable diesel. Renewable diesel is characterized by a high cetane number, low specific gravity, good cold flow properties, storage stability, and it can be mixed at any proportion with fossil diesel.

Regarding the production process, two stages are presented: catalytic hydrotreating to obtain paraffins, and a catalytic isomerization stage to produce mixtures of n- and -iso paraffins with water and gases as methane, propane and carbon oxides. However, this reaction is not precise enough and presents each of the chemical mechanisms by which oxygen is removed. Three reactions that present competition are specifically presented in the deoxygenation process: hydrodeoxygenation, decarbonylation and decarboxylation (Romero et al., 2018). One of the main aspects in hydroprocessing is to provide enough hydrogen to carry out the chemical conversion. Molecular hydrogen is a very important raw material for both the chemical and petroleum industries, mainly due to its positive atomic economy (Toledano et al., 2013). Currently, commercial hydrogen is being produced mainly from non-renewable raw materials: about 90% is obtained by reforming natural gas or fractions of naphtha, while the rest is obtained from coal gasification and water electrolysis (Martínez-Merino, et al., 2013). All the above processes have the disadvantages of relying on fossil and non-renewable raw materials and being environmentally damaging or highly intensive in energy consumption. Additionally, the difficult handling of gaseous hydrogen and the large quantities required hinder energy optimization of the process, since hydrogen gas at high pressure presents a considerable safety risk and implies a high cost of infrastructure at industrial scale. This generates an economic barrier for the development of a sustainable industry such as biorefineries, especially at the initial stages (Gilkey & Xu, 2016).

The possibility of generating the hydrogen *in situ*, instead of feeding it to the hydrotreating reactor, is thus envisaged. The generation in situ of hydrogen can be achieved by Aqueous Phase Reforming (APR) or by Catalytic Transfer of Hydrogen (CTH). APR requires the presence of water at intense conditions of temperature and pressure, while CTH requires a substance that donates hydrogen to an acceptor, that is, a donor, usually a solvent, that transfers hydrogen (M.A. Mayorga et al., 2020). As an alternative to overcome these limitations, the proposal is the use of hydrogen generated from biomass (biohydrogen), which is not in the gas phase, but is taken to the catalyst by a donor, capable of generating it on the surface of the catalyst, facilitating its handling and enhancing its effect. This study proposes to analyze the use of biodonors capable of generating hydrogen *in situ* through hydrogenation by CTH, whose storage and transport is much cheaper than molecular hydrogen (X. Jin, 2014). In this way, the problems related to the low solubility of gaseous hydrogen, such as the use of high pressures with the consequent safety problems, could be avoided if it is generated directly on the active sites of the catalyst, allowing a process with a lower working pressure.

In the CTH, the donor, which acts as a source of hydrogen, must be catalytically and/or thermally decomposed into molecular hydrogen, carbon oxides and light hydrocarbons, not to be confused with a simple dehydrogenation. It is ideal that the donor is renewable, so biorefinery streams will be used, with oxygenated components such as alcohols, aldehydes, ketones and carboxylic acids, (e.g. methanol, ethanol, formic acid and glycerol), including condensed aromatic compounds, some alkanes and amines (Ramírez-Corredores, 2013). These solvents are relatively cheaper than molecular hydrogen (X. Jin, 2014). In this work, a multicriteria decision method (MACBETH) is used for the selection of a hydrogen donor. Alternatives and criteria are proposed for choosing the most suitable in the process. Finally, the catalyst has an important role in this process, as it should generate hydrogen from a donor for using it later for hydroprocessing *in situ* of the palm oil with the objective of renewable diesel production (Manuel A Mayorga, et al., 2019).

2. Methodology

The procedure to evaluate the selection of donors is described below. It is important to consider that the hydrogen used must be of renewable origin. Due to the obstacles that may arise in its availability, it is intended to test the use of hydrogen donors, mainly oxygenated compounds such as alcohols, aldehydes, ketones and carboxylic acids, presenting the additional advantage of reducing the level of decarboxylation during the hydrodeoxygenation process and the pressure required by the system, favoring conversion, yield and selectivity. In the first instance, the criteria for selection are established, in order to make the multicriteria decision and finally a thermodynamic analysis is made to show the feasibility of the decomposition of the hydrogen donors.

2.1 Criterion

Since high pressures are used in the hydroprocessing of biomass, a great advantage of using hydrogen donating solvents is to operate at much lower pressures. Additionally, the donor makes the stabilization of the primary decomposition of the products more effectively, preventing carbonization and coking and resulting in higher efficiencies. Therefore, the selection of the solvent can have a great effect on the performance of the liquid produced. To select a good donor, the compound must be considered to contain mobile carbon-hydrogen bonds, dehydrogene easily, have high solvation capacity, high liquefaction yields and reduction in the formation of coal and coke. It is also necessary to consider that, in addition to its renewable origin and production in biorefinery, the hydrogen donor has low cost and high availability, liquid phase at the operating conditions according to its phase diagram, adequate viscosity, thermodynamic feasibility in its dehydrogenation and decomposition, and an appropriate catalytic cycle. Thus, they are considered as first organic solvents, protic and with any polarity, which generally corresponds to oxygenated compounds, such as alcohols, polyols, aldehydes, ketones, carboxylic acids, aromatic polycyclic (condensed aromatic), amines and alkanes.

2.2 Multicriteria Decision

After listing all the options of possible donor families and the alternatives to be selected, a multicriteria analysis is made for decision-making. Multi-Criteria Decision Analysis (MCDA) methods are formal approaches that consider multiple criteria to help decision-making processes. Within these methods, MACBETH is recommended, as it is an approach to multicriteria decision-making problems applied by the Multi-Attribute Utility Theory (MAUT), which assigns a value to each criterion for evaluating and thus finding a global criterion. In the first step, once the criteria to evaluate the alternatives have been selected, experts should order them from highest to lowest, according to the importance of each criterion for the final result. Then, they must assign a numerical value that separates an alternative from the one immediately following, taking into account only two criteria at the same time. In the same way, the last criterion must be compared with a non-existent one that have no importance in the final product, so that that the importance of the last one is evaluated with respect to any other unconsidered quality. The weight of each criterion j , w_j , (of n criteria) is calculated with the differences a_{jk} between criterion j and criterion k , as presented by the Equation (1):

$$w_j = \frac{(\sum_{j=1}^n a_{jk} - \sum_{j=0}^{k-1} a_{jk}) / \sum_{j=1}^n a_{jk}}{\sum_{j=1}^n (\sum_{j=1}^n a_{jk} - \sum_{j=0}^{k-1} a_{jk}) / \sum_{j=1}^n a_{jk}} \quad (1)$$

In the second step, according to the theory of utility, each criterion provides a partial description of the global utility for each alternative U_i , so the global utility can be calculated from the partial profits, using a linear combination of w_j and functions obtained from valuations of experts, according to the next expression (Eq. 2):

$$U_i = \sum_{j=1}^n [w_j f_j(C_j)] \quad (2)$$

where $f_j(C_j)$ is a function of preference for each criterion j . And, in the third step, the alternatives are classified from highest to lowest U_i . The final classification is used for the decision, 8 alternatives are proposed (alcohols, polyols, aldehydes, ketones, carboxylic acids, polycyclic aromatic, amines and alkanes) and 5 criteria are established (Availability, Cost, Renewable Source, Decomposition Products and Toxicity).

2.3 Thermodynamic Analysis

A representative donor will be taken from each of the chosen families, according to the literature review and considering the renewal and availability. For each postulated donation, possible decomposition reactions will arise, among which the generation of hydrogen should be highlighted. Then, for each reaction, the equilibrium constant K , between 20 °C (293.15 K) and 526.15 °C (800 K) is calculated for a given operating temperature T . By performing the graph of $\ln(k)$ vs. $1/T$, this behavior can be observed, indicating the reactions that will occur for each donor and analyzing particularly the generation of hydrogen, which is the one of interest.

2.4 Selection

Based on a global analysis given by the above items, the most promising donors are defined for the production of renewable diesel by deoxygenation of palm oil with hydrogen generation *in situ*.

3. Results and Discussion

3.1 Multicriteria Decision

For each of the criteria, w_j is determined by observing how big is the difference between the criteria presented, organized from the most to the least important (1-Renewable Origin, 2-Decomposition Products, 3-Cost, 4-Availability, 5-Toxicity) and other possible. With these, a matrix is elaborated in Table 1, where the difference between the row and the column aims to calculate the assigned w_j for each criterion, according to Equation 1.

Table 1: Calculation of the weight assigned to each criterion.

Criteria	j	1	2	3	4	5	6	Σ	Diff.	Relative	w_j
Renewable origin	1	-	14	11	8	14	14	61	11	1.0000	0.3081
Decomposition products	2	-	-	17	17	2	14	50	14	0.8197	0.2525
Cost	3	-	-	-	5	14	17	36	5	0.5902	0.1818
Availability	4	-	-	-	-	17	14	31	11	0.5082	0.1566
Toxicity	5	-	-	-	-	-	20	20	20	0.3279	0.1010
Others	6	-	-	-	-	-	-	0	0	0.0000	0.0000
Σ									61	3.2459	1.0000

For the qualification of each alternative (presented in Table 2), technical justifications were taken into account in each criterion. For example, in the dehydrogenation and decomposition of these molecules, besides generating hydrogen, the reaction also generates other by-products as follows. If they are alcohols and polyols, carbon monoxide and alkanes are produced; while if they are aldehydes and ketones, monoxide and alkenes are liberated; and if they are carboxylic acids, they decompose into carbon dioxide and alkenes. Considering that the production of alkenes is undesirable, since they effectively consume hydrogen to saturate alkanes, alcohols and formic acid, the only organic acid that does not decompose in alkenes, but H_2 and CO_2 , can be considered as promising donor. Regarding alcohols, ethanol obtained biotechnologically from the fermentation of carbohydrates is widely available. The decomposition products of each of the families are:

- *Alcohols*: $CO + H_2 + C_nH_{2n+2}$ (carbon monoxide, hydrogen and alkanes)
- *Polyols*: $CO + H_2 + C_nH_{2n+2}$ (carbon monoxide, hydrogen and alkanes)
- *Aldehydes and ketones*: $CO + H_2 + C_nH_{2n}$ (carbon monoxide, hydrogen and alkenes)
- *Carboxylic Acids*: $CO_2 + H_2 + C_nH_{2n}$ (carbon dioxide, hydrogen and alkenes)
- *Condensed aromatics homologs*: $H_2 + C_{10}H_8$ (hydrogen and naphthalene)
- *Amines*: $R-C\equiv N + H_2$ (nitriles and hydrogen)
- *Alkanes*: $H_2 + C_nH_{2n}$ (hydrogen and alkenes)

Now, by the Macbeth method, the utility is calculated from the rating for each criterion j and for each of the alternatives i , according to Equation 4, whose results are shown in Table 2.

Table 2: Calculation of the global utility for each alternative.

Alternative, i	Criteria, j					U_i
	1	2	3	4	5	
Alcohols	10	10	7	8	9	9.0404
Polyols	8	9	8	8	9	8.3535
Aldehydes	8	6	5	5	7	6.3788
Ketones	7	6	5	5	8	6.1717
Carboxylic Acids	9	9	6	8	8	8.1970
Condensed Aromatics	6	4	7	8	4	5.7879
Amines	5	6	7	7	5	5.9293
Alkanes	3	5	9	9	7	5.9394

This table shows that the alternatives with a global utility greater than 8 in descending order are: alcohols, polyols and carboxylic acids. Within these selected families, the following compounds are taken into account:

- *Alcohols*: Ethanol, methanol, isobutanol, isopropanol (tertiary and secondary alcohols release H_2 more easily).
- *Polyols*: Glycerine, ethylene glycol, propylene glycol.
- *Carboxylic Acids*: Formic acid, acetic acid.

Ethanol is chosen from alcohols, since it is generated by the process of fermentation of sugars, used mainly in the beverage industry, which is a competitive advantage in terms of the availability of other alcohols. Regarding polyols, the decision is made for glycerine, because it is obtained in good quantities as a by-product in the soaps and ester biodiesel industry. In contrast to carboxylic acids, the formic acid in its

decomposition by decarboxylation produces H_2 and H_2O , while others acids generate hydrocarbons, predominantly alkenes.

3.2 Thermodynamic analysis

The possible decomposition reactions for formic acid, ethanol and glycerine are presented in Table 3.

Table 3: Decomposition reactions for promising donors.

Chemical Reaction	Chemical Equation
Decarboxylation (dehydrogenation) of formic acid (DCX)	$HCOOH \rightarrow CO_2 + H_2$
Decarbonylation (dehydration) of formic acid (DCN)	$HCOOH \rightarrow CO + H_2O$
Dehydration of ethanol (DW)	$CH_3CH_2OH \rightarrow CH_2=CH_2 + H_2O$
Dehydrogenation of ethanol (DH)	$CH_3CH_2OH \rightarrow CH_3CHO + H_2$
Ethanol methanation (M)	$CH_3CH_2OH \rightarrow CO + CH_4 + H_2$
Etherification of ethanol (E)	$2CH_3CH_2OH \rightarrow CH_3CH_2OCH_2CH_3 + H_2O$
Dehydrogenation of glycerine (DH) by APR	$CH_2OHCHOHCH_2OH \rightarrow 3CO + 4H_2$
Dehydration of glycerine (DW)	$CH_2OHCHOHCH_2OH \rightarrow CH_2=CHCHO + 2H_2O$

The thermodynamic feasibility of the above reactions (Table 3) is analyzed in the thermal profiles of the equilibrium constants for the interval proposed (Figure 1). In the case of formic acid, it can be observed that both, DCX and DCN are feasible, with the difference that the first is exothermic, thus with the increase in temperature, it becomes less favorable. However, the DCX for the production of hydrogen is always more favorable than the DCN one that is endothermic. For ethanol, the only exothermic reaction is E, which is feasible in the whole interval, while M is the reaction required for the generation of hydrogen and becomes feasible after approximately 120 °C, where it prevails with respect to the other three decomposition reactions. DH is favored after 100 °C, but in competition with dehydration, which only exceeds after 330 °C. In conclusion, the thermal analysis indicates that the donation of hydrogen is completely spontaneous due to the decarboxylation of formic acid, and in glycerine only after 100 °C, but exceeding formic acid with temperatures higher than 190 °C.

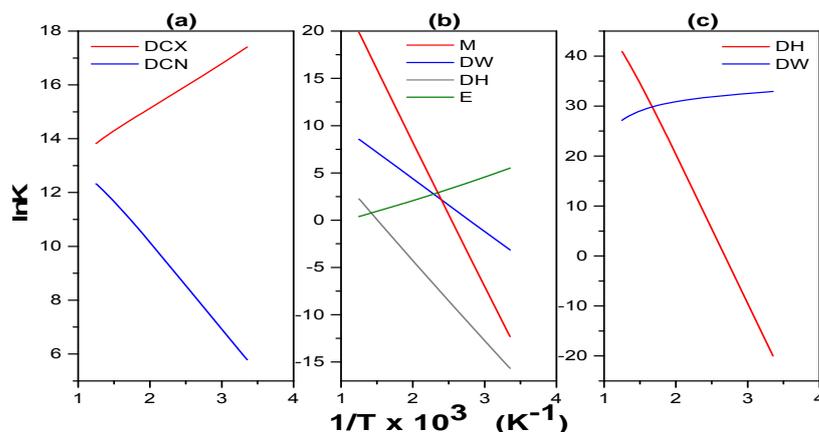


Figure 1: Thermal profile in the decomposition for: (a) formic acid, (b) ethanol and (c) glycerine.

3.3 Selection

Among formic acid, ethanol and glycerine, the last one is discarded for two important reasons. First, one of the main sources of glycerine is the biodiesel industry, but the idea is to substitute this biodiesel with renewable diesel, then glycerine would not be available. Second, the dehydrogenation of glycerine is carried out by APR, which requires water at intensive conditions of temperature and pressure, making the process less sustainable. Formic acid is one of the major byproducts of the biorefinery process. In addition, it has unique properties such as non-toxicity, favorable energy density, and biodegradability, thus it is an economically attractive and safe reagent for energy storage and chemical synthesis. Currently, in many green catalytic transformations, the formic acid of biotechnological origin is an efficient and versatile reagent, which has been researched in numerous applications as a hydrogen donor source in deoxygenation processes. Formic acid can be obtained in biorefinery in many ways, not only by the treatment of lignin or synthesis gas. Another option is for example the hydrothermal conversion of carbohydrates at moderate temperatures (Jin et al., 2008). Meanwhile, ethanol is one of the most important biorefinery products, generated mainly by fermentation of sugars and starches. It is a well-known and optimized technology that generates important volumes of

ethanol that is used primarily in alcoholic beverages, solvents and lately as fuel and partial substitute for gasoline.

4. Conclusions

This research not only presents a study of hydrogen donating substances, useful for an oil hydrotreating process, but also constructs a matrix of options that allows to recognize the differences between the employment of the different candidates. Representatives of each organic functional group are used for the construction of this matrix, which is evaluated using MACBETH that allows to find the optimal donors of the process. The results of the multicriteria algorithm show that the best families are alcohols, polyols and carboxylic acids. The main exponents of each of these families were subjected to a thermodynamic feasibility analysis for the catalytic reactions of decomposition, to evaluate the substances to be used as hydrogen biodonor. Formic acid and ethanol, both readily obtainable substances and process streams in many biorefineries, are available within the constructed set of candidates, with prices that make them easy to be acquired or synthesized.

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