

# Reuse of Ashes from Digestate Solid Fraction as a Catalytic Support for the Conversion of Vegetable Oils in Biofuels

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This experimental preliminary study investigates the reuse of ashes from Solid Fraction of Digestate (SFD) to synthesize catalysts for biofuels synthesis from vegetable oils. The digestate derived from anaerobic digestion of pomace, lees and agrifood waste. SFD pellets were incinerated at 700°C to produce ashes. Ashes were washed with HCl solution and optionally converted into zeolites (alkali fusion method followed by hydrothermal synthesis). These three synthesized supports (ashes, washed ashes, and ash-derived zeolites), were impregnated to produce Ni-Mo supported catalysts. Ni-Mo catalysts were comparatively tested by a laboratory-scale batch reactor, performing DeOxygenation (DO) of commercial rapeseed oil into green diesel (40 bar of H<sub>2</sub> at room temperature and 320°C). The best DO performances were obtained by the Ni-Mo catalysts supported on SFD ashes washed with HCl (80% oil Conversion and 55.1% Diesel Yield). These performances were also associated with a greater specific surface area of Ni-Mo catalysts supported on SFD ashes washed with HCl than the others in this study. Ni-Mo catalyst from SFD can be of interest for developments in the circular-economy approach.

## 1. Introduction

Anaerobic Digestion (AD) is a biological process that converts putrescible organic matter into biogas (mainly a mixture of CO<sub>2</sub> and CH<sub>4</sub>) and a humid solid residue named digestate (Ward et al., 2008). Around 180 million tonnes of digestate per year are produced in the EU28 (Saveyn & Eder, 2014).

In view of new production chains fed with biomass and waste (i.e., the concept of biorefinery), the integration of AD with other unitary processes must be pursued to create virtuous cascades in the circular economy sense (Macarthur, 2017). The digestate may be better exploited before its return to the biosphere, e.g., the Solid Fraction of Digestate (SFD) may be used as a solid fuel for thermochemical processes, such as incineration (to produce heat and power from flue gases (Gao et al., 2020; Mammarella et al., 2024)) or gasification (to produce syngas, a mixture of mainly H<sub>2</sub> and CO, valuable as a gaseous fuel or feedstock (Freda et al., 2019)).

In both those thermochemical processes, ashes form as the main solid by-product, which must also be circularly managed. Ashes may contain heavy metals, metal oxides, and mineral salts, whose composition strongly depends on the feedstock and influences the ashes disposal strategy (Mammarella et al., 2024).

Incineration is widely used in Europe for waste and biomass treatment (Levaggi et al., 2020). It consists of the total oxidation of the feedstock, usually at a temperature above 500°C (Zajac et al., 2019), to produce heat and power from flue gases (Gao et al., 2020). In Europe, the incinerated waste amount has grown by 117%, from 32 Mt in 1995 to 70 Mt in 2018, in turn increasing ash production (Levaggi et al., 2020).

Gasification is also used to treat biomass (IEA Bioenergy) and waste: the production of valuable syngas involves the formation of high quantity of ash (Gao et al., 2020). Worldwide, annual production of biomass ash due to gasification is growing fast (almost 500 million tons/year) (Guo et al., 2023).

As to agricultural waste, the related ash production in Europe is about 0.75 Mt/y; this kind of ash generally has significant amounts of potassium, calcium, silicon and aluminum (Mammarella et al., 2024).

The reuse of these ashes to synthesize zeolites is an interesting possibility (Ju et al., 2021), thanks to Si and Al contents (Fukasawa et al., 2017). Zeolites are materials of interest for industrial applications, e.g., as adsorbents and catalytic supports (Ferella et al., 2017). The general reuse of ashes as catalysts or catalytic support is documented in the literature for methane reforming (Wang and Lu, 2007) and biodiesel production by oils (Odude et al., 2019).

Based on these premises, this work presents a preliminary study on the unprecedented reuse of SFD ashes as catalytic support for the DeOxygenation (DO) of vegetable oils in green diesel, according to a circular economy model (Mammarella et al., 2024). DO is a hydroprocessing of oils that leads to the conversion of fatty acids in their triglycerides mainly into normal alkanes (Kumar et al., 2010), which are known overall as green diesel. The main DO reactions are: decarbonylation (DCO), decarboxylation (DCO<sub>2</sub>), and hydrodeoxygenation (HDO). DO is usually carried out by a heterogeneous solid catalyst; e.g., the synergistic effect of Ni and Mo with a solid acid support improved conversion from triglycerides to hydrocarbons through DCO, DCO<sub>2</sub> and HDO reactions (Ameen et al., 2017; Lucantonio et al., 2023). Works concerning DO are comprehensively referred to in the recent review by Lucantonio et al., where a comparative analysis of several experimental studies and industrial cases has been reported (Lucantonio et al., 2023).

In this work, the possibility of exploiting SFD to produce effective DO catalysts was explored. Several original Ni-Mo catalysts, derived from SFD ashes, were purposely synthesized, characterized, and tested for the first time for the DO of rapeseed oil in a batch reactor at a laboratory scale. Quantification of DO performances in terms of oil Conversion and Diesel Yield allowed selecting the most promising SFD-derived catalysts.

## 2. Materials and methods

### 2.1 Catalysts synthesis

SFD pellets were produced by BARTIN Distillery (Italy) with digestate from AD of pomace, lees, and waste from the agrifood supply chain.

Ash from SFD (ASFD) was produced at 700°C by air calcination of SFD pellets in a muffle furnace. To increase the content of Si and Al, ASFD underwent acid washing: (i) acid extraction was carried out by treating 30 g of SFD ash in 500 cm<sup>3</sup> of HCl 5 M for 5 h; (ii) the resulting suspension was filtered, washed with distilled water near pH 7 and dried at 105 °C for 4 h (Joseph et al., 2019). The resulting solid was named AWSFD.

AWSFD was used for the synthesis of zeolites (named ZSFD), by the alkali fusion followed by hydrothermal treatment: (i) AWSFD was ground by mortar and pestle with 1.2 parts (by mass) of NaOH and treated at 550°C in air for 1 h in a muffle furnace; (ii) the resulting solid was mixed with deionized water (1:10 mass ratio) and transferred to a Teflon-coated reactor for aging at room temperature for 24 h; (iii) after aging, the reactor was placed in a stainless-steel vessel for hydrothermal treatment (120°C for 24 h); (iv) the solid was filtered, washed with deionized water up to pH 8, dried at 105 °C and finely ground (Ferella et al., 2017; Küçük et al., 2023).

ASFD, AWSFD, and ZSFD became the catalytic supports for this study. They underwent conventional wet impregnation (Sietsma et al., 2006) in water (preferred as a green solvent), to obtain DO catalysts with final contents of 5 %wt NiO and 15 %wt MoO<sub>3</sub>: (i) Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O e (NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>·4H<sub>2</sub>O precursor salts were solubilized in 5 ml of distilled water ; (ii) the support (ASFD, AWSFD or ZSFD) was added after their solubilization and kept there for 3 h; (iii) the suspension was first dried in a rotavapor and then for 24 h at 105°C in an oven; (iv) the solids were calcined at 400°C for 4 h (heating rate 5°C/min), to obtain three DO catalysts Ni-Mo/ASFD, Ni-Mo/AWSFD and Ni-Mo/ZSFD.

### 2.2 Materials characterizations

The moisture and ash content of SFD pellets were quantified according to UNI EN 18134 and UNI EN ISO 18122, respectively. The ultimate analysis of SFD and ASFD was performed through CHNS Perkin Elmer Series II 2400 in order to quantify carbon, hydrogen, nitrogen, and sulphur, according to ISO 16948. The elemental composition of ASFD and AWSFD was determined by X-ray fluorescence (XRF, Spectro Xepos). Crystalline phases in ASFD, and ZSFD were identified by X-ray diffraction (XRD, PANalytical X'Pert PRO) using radiation CuK $\alpha$  ( $\lambda$  = 0,154 nm) with Ni filter.

All three synthesized catalysts (Ni-Mo/ASFD, Ni-Mo/AWSFD and Ni-Mo/ZSFD) were characterized by:

- N<sub>2</sub> adsorption/desorption to quantify specific surface area by Brunauer-Emmett-Teller (BET) method (Physisorption Analyzer NOVA 800 Anton Paar). Before N<sub>2</sub> treatment, each sample was outgassed for 16 h at 150°C.
- Inductively coupled plasma optical emission spectroscopy (ICP-OES, Agilent Technologies 5100) to quantify the elemental amount of Ni and Mo in the synthesized catalysts.

### 2.3 Batch reactor catalytic activity tests

Catalyst pre-reductions and DO catalytic activity tests were conducted in a rig consisting of a 100ml Parr batch reactor (4590 Micro Bench Top Reactor, Magnetic drives, and 4848 Controller), fillable with H<sub>2</sub> (grade 5.0) and or N<sub>2</sub> (grade 5.0) from pressurized bottles.

The catalyst was pre-reduced at 320°C for 4 h, under H<sub>2</sub> (60 bar at room temperature in the reactor volume).

The DO catalytic activity tests were prepared by loading the reactor with about 0.2 g of reduced catalyst, 2 g of rapeseed oil ( $g_{oil}$ ) and hexane (oil-solvent mass ratio 1:10), blanketing with N<sub>2</sub> and then refilling with H<sub>2</sub> (40 bar at room temperature in the reactor volume). The DO tests were carried out at 320°C for 6 h and stirred at 600 rpm. At the end of each test, the final Organic Liquid Product (OLP) was separated from the catalysts by filtration and weighed ( $g_{OLP}$ ).

OLP was processed by transesterification (according to standard procedure AOAC (Association of Official Agricultural Chemists) 969.33) to convert the unreacted rapeseed oil into FAME (Fatty Acid Methyl Esters), then analyzed by a Gas Chromatograph with Flame Ionization Detector (GC-FID). The GC-FID used is an Agilent Technologies 7820A GC System equipped with a non-polar HP-5 19091J-413 Capillary column (30 m x 0,32 mm x 0,25 μm, stationary phase = (5%-phenyl) methylpolysiloxane) capable of effectively separating hydrocarbons in the OLP. The chromatographic run lasted 35 minutes, during which the column was initially kept at 50°C for 5 min, then heated to 280°C (10°C/min), and finally kept at 280°C for 7 min. The eluent gas was H<sub>2</sub> at 2 ml/min; the injector was at 250°C and FID at 300°C. Through the comparison with a standard solution of saturated linear alkanes C<sub>7</sub>-C<sub>40</sub>, it was possible to classify and estimate the composition through area percentages of the peaks corresponding specific hydrocarbons and FAME ( $Area\%_i$ ) in the OLP and obtain the Conversion of Triglycerides ( $X$ ) (Eq(1)) and Diesel Yield ( $DY$ ) (hydrocarbons C<sub>15</sub>-C<sub>18</sub> yield) (Eq(2)).

$$X = \text{Conversion of Triglycerides (\%)} = \left[ 1 - \left( \frac{Area\%_{FAME} / 100 \cdot g_{OLP}}{g_{oil}} \right) \right] \cdot 100 \quad (1)$$

$$DY = \text{Diesel Yield (\%)} = \left( \frac{Area\%_{C_{15}-C_{18}} / 100 \cdot g_{OLP}}{g_{oil}} \right) \cdot 100 \quad (2)$$

### 3. Results

SFD pellets had an ash content on a dry basis of 32.8 wt%, relatively high if compared to lignocellulosic biomasses (typically with ash content on a dry basis below 15 wt% (Mu et al., 2024)).

Table 1 shows the ultimate analysis of SFD pellets and ASFD: the calcination treatment of SFD pellets was carried out effectively since a decrease in carbon content was found, from 34.3 wt% down to 0.5 wt%, i.e., all carbon was combusted.

Table 1: Ultimate analysis of SFD and ASFD

	C	H	N	S
SFD (wt%)	33.5	4.4	4.7	1.1
ASFD (wt%)	0.5	0.2	0.0	0.7

Table 2 shows that the washing process of ASFD into AWSFD led to a very significant removal of Mg, Ca, Fe, Na, and P; a slight decrease in Al and a significant increase in the fraction of Si (from 6.84 to 36.48 wt%) determined an important increase of the Si/Al ratio, a key-parameter for zeolite synthesis (Bukhari et al., 2015). These results are comparable with the literature (Joseph et al., 2019).

Table 2: XRF results of ASFD and AWSFD

	Si	Al	K	Mg	Ca	Fe	Na	P
ASFD (wt%)	6.8	1.9	1.7	1.3	15.3	2.8	2.5	6.1
AWSFD (wt%)	36.5	1.5	1.6	0.0	0.6	0.4	0.0	0.1

Figure 1 compares the diffractograms of ASFD and ZSFD: different crystalline phases appeared in ASFD, demonstrating the heterogeneity of ash (Figure 1(a)); noteworthy, the zeolitization was successful, as confirmed by analcime phase in ZSFD (Figure 1(b)), a zeolite used as catalyst or catalytic support (Gan et al., 2024; Kochaniec et al., 2024). In the ZSFD sample (Figure 1(b)), two other crystalline phases appeared, ascribable to calcium sodium aluminosilicates and a phase of SiO<sub>2</sub>, attributed to the excess of Si in the sample (Table 2).

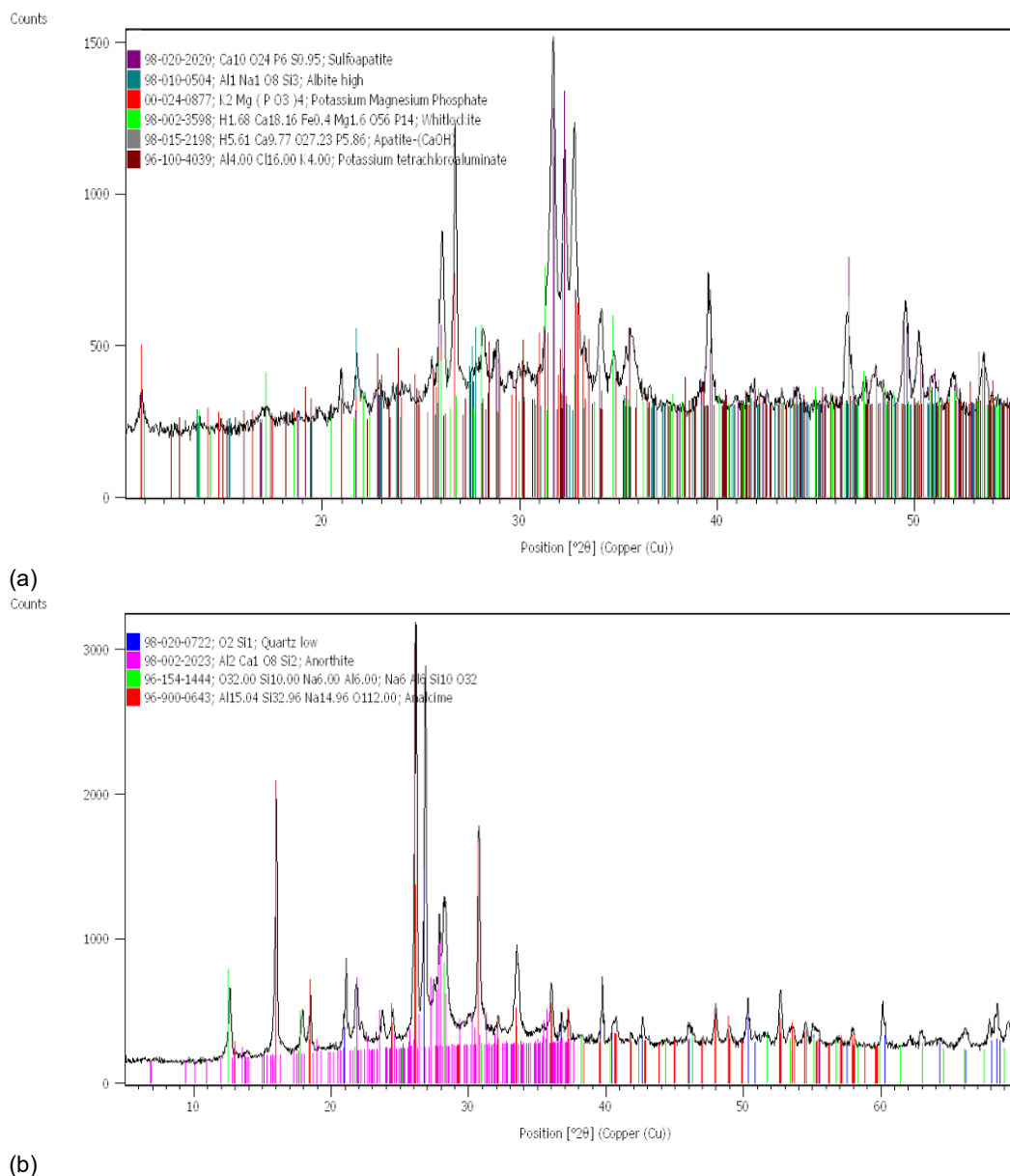


Figure 1: XRD patterns of ASFD (a) and ZSFD (b)

As to synthesized Ni-Mo catalysts, Table 3 shows the amount of active phases, rescaled as NiO and MoO<sub>3</sub>: the impregnation was successful, as experimental amounts were in line with the desired nominal values (5 %wt NiO, 15 %wt MoO<sub>3</sub>).

Table 3: ICP-OES results: elemental percentage of NiO and MoO<sub>3</sub> in the DO catalysts

Material		Ni-Mo/ASFD	Ni-Mo/AWSFD	Ni-Mo/ZSFD
NiO amount	(wt%)	4.3	3.9	4.7
MoO <sub>3</sub> amount	(wt%)	14.2	12.6	15.8

Table 4 shows the specific surface area of catalysts, according to the BET method ( $S_{BET}$ ): the catalyst supported by SFD ash (Ni-Mo/ASFD) had the lowest  $S_{BET}$  (24 m<sup>2</sup>/g), comparable with the ash-supported catalysts from literature (Miladinović et al., 2020); the washed ash-based catalyst (Ni-Mo/AWSFD) had the highest  $S_{BET}$  (67 m<sup>2</sup>/g) outcome ascribable to the acid washing (Joseph et al., 2019); the catalyst supported with ash-zeolites showed an  $S_{BET}$  (35 m<sup>2</sup>/g) mainly related to the features of the analcime phase (Figure 1(b)) documented to have  $S_{BET}$  of approximately 20 m<sup>2</sup>/g (Park et al., 2012).

Table 4: Specific surface areas by BET method

Material	Ni-Mo/ASFD	Ni-Mo/AWSFD	Ni-Mo/ZSFD
$S_{BET}$ (m <sup>2</sup> g <sup>-1</sup> )	24	67	35

Table 5 summarizes the experimental catalytic DO performances: Ni-Mo/AWSFD was the catalyst with the best  $X$  (Eq(1)) and  $DY$  (Eq(2)) of 79.5% and 55.1%, respectively, with a  $C_{15}$ - $C_{18}$  composition of 64.9 wt%. On the basis of presented characterizations, better performances of Ni-Mo/AWSFD – when compared to the other two investigated catalysts – were attributed to the higher  $S_{BET}$ , in agreement with the literature (Horáček et al., 2014; Wang et al., 2013); this may suggest that a better accessibility to active phases was ensured by Ni-Mo/AWSFD, considering that it had the lowest actual content of active phases (Table 3). Ni-Mo/ZASFD has slightly higher  $DY$  and  $X$  compared to Ni-Mo/ASFD; however, both of them have FAME content greater than 50 wt%, vs. the 24.0 wt% of FAME with Ni-Mo/AWSFD. Despite having an  $X$  lower than that of commercial catalysts studied in the literature (e.g., the Ni/Alumina commercial and Ni-Mo/Alumina catalysts, which in experimental conditions similar to those studied show an  $X$  between approximately 80-100% (Lucantonio et al., 2023), the most active catalyst of this work (Ni-Mo/AWSFD) traces an interesting perspective for future studies, concerning circular catalyst development from waste material.

Table 5: OLP composition (Alkanes with C number < $C_{14}$ , between  $C_{15}$ - $C_{18}$ , > $C_{19}$ ; FAME, i.e., unconverted fatty acids; OTHER compounds; unsaturated (UNS) and branched (BRA) hydrocarbons), Diesel Yield (DY) and Conversion (X)

Sample	< $C_{14}$ (wt%)	$C_{15}$ - $C_{18}$ (wt%)	> $C_{19}$ (wt%)	BRA (wt%)	UNS (wt%)	FAME (wt%)	OTHER (wt%)	DY (%)	X (%)
Ni-Mo/ASFD	1.4	14.8	1.9	1.4	23.4	56.5	0.6	14.3	44.7
Ni-Mo/AWSFD	2.7	64.9	2.3	2.7	3.4	24.0	0.0	55.1	79.5
Ni-Mo/ZSFD	2.3	18.2	0.0	0.0	26.4	50.8	2.3	17.3	51.7

#### 4. Conclusions

In this explorative work, the reuse of SFD ash was studied for the synthesis of three Ni-Mo impregnated catalysts, tested for the DO reaction of rapeseed oil for green diesel production. XRF showed the increase in the Si/Al ratio after the acid washing of ashes, fundamental for zeolites synthesis; XRD patterns of the ZSFD confirmed zeolitic phase formation (analcime). Experimental DO results proved that the Ni-Mo/AWSFD catalyst was the best one in terms of DO Diesel Yield (55.1%) and Conversion (79.5%); this result was explained by the higher specific surface area of the catalyst, compared to those of Ni-Mo/ASFD and Ni-Mo/ZSFD. Therefore, AWSFD proved to be the best support, which would lead to avoiding a zeolite synthesis step. As a catalyst circularly produced from by-products, Ni-Mo/AWSFD has interesting perspectives for future studies varying the concentration of the active phase, adding possible promoters, and testing the support with other active phases with the aim of refining and improving the catalytic properties.

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