Feasibility of Waste Engine Oil Electrooxidation with Ni-Co and Cu-B Catalysts

Paweł P. Włodarczyk * and Barbara Włodarczyk *

Institute of Environmental Engineering and Biotechnology, University of Opole, ul. Kominka 6a, 45-032 Opole, Poland
* Correspondence: pawel.wlodarczyk@uni.opole.pl (P.P.W.); barbara.wlodarczyk@uni.opole.pl (B.W.);
Tel. +48-774-016-048 (P.P.W. & B.W.)

Abstract: To implement a circular economy policy, methods of using waste products as a starting point for other technologies are constantly researched. One of the waste products that should be disposed of after use is waste engine oil (WEO). Despite the permanent introduction of the electrification of cars, the number of combustion vehicles (and, thus, the production of WEO) is constantly increasing. For these reasons, the reuse of WEOs is extremely important; e.g., to use these oils for energy purposes. One of the potential uses of this type of oil is as fuel for fuel cells (for direct electricity production). To lower the production costs of electrodes for fuel cells, catalysts that do not contain precious metals are being constantly sought. This work shows the research of WEO electrooxidation feasibility with Ni-Co and Cu-B catalysts. The results showed the feasibility of the electrooxidation of WEO emulsion on Ni-Co and Cu-B electrodes in an electrolyte (a water solution of sulfuric acid). Moreover, it was shown that the electrooxidation of the WEO emulsion occurred for all concentrations of WEO in the emulsion for all measurement temperatures (20–80 °C). The highest current density obtained in the measurements was 11 mA cm⁻² (at 60 °C) for the Ni-Co electrode.

Keywords: waste engine oil; use of by-product; Cu-B catalyst; Ni-Co catalyst; electricity production; environmental engineering; fuel cell

1. Introduction

The number of motor vehicles in the world is constantly increasing. The upward trend also continues in Poland [1]. Here, a total of over 1.8 million vehicles were registered in 2021 [1]. Figure 1 shows the number of newly registered cars (for the first time in the country) in Poland for the years 2011–2021. The temporary drop in sales in 2020 was a result of changes in the car sales market because of COVID-19 [2]. Since 2021, car sales have increased again.

Figure 1. Number of newly registered motor vehicles in Poland for the years 2011–2021 [1].

The data (Figure 1) include all motor vehicles; e.g., cars, vans, trucks, agricultural tractors, special vehicles, motorcycles and mopeds. Currently, there are over 38 million
registered vehicles in Poland, including over 10 million inactive registrations [1]. Inactive registrations are vehicles excluded from traffic, scrapped or left in parking lots, for example. However, over 28 million are active registrations; i.e., vehicles that travel on Polish roads. Each vehicle uses both engine oils and transmission oils. These oils are also regularly changed, generating huge amounts of waste oils. It should be noted that these are data from only one country.

The European Parliament voted to end the sale of combustion cars from 2035 [3]. However, the replacement of most vehicles in the EU will not abruptly happen. Vehicles of this type will be used for many, many years to come. Thus, the generation of huge amounts of waste oils (which have to be disposed of or reused; e.g., for the production of new oils or energy production) will also take place for a long time. For these reasons, the disposal of waste engine oils (WEOs) is a very important issue [4–6]. In most countries of the world, WEOs, waste transmission oils, used oil filters or oil packaging are classified as hazardous waste [7,8].

The parameters of waste oils (e.g., the amount of other contaminants or the oxidation state) depend on the type of oil, amount of improvers, operation time, operation temperature, oil oxidation rate, presence of different metals (e.g., catalytically active) and mechanical shear forces [7,9–12]. Moreover, the oil can be polluted by exhaust gases or other combustion products [13,14].

Activities for ecology require that the disposal process should be carried out in a way that has the least impact on the natural environment [15,16]. One of the rational forms of the disposal of used oils can be their industrial use [7,16,17]. Examples of ways to use waste oils include the restoration of oil properties by filtration, reprocessing oils to form a substitute fuel, recycling by adding a refining process, deep regeneration (re-refining) or re-using oil directly as a fuel [18–25]. Unfortunately, a few methods pose a threat to the natural environment [24,25].

One of the methods of WEO utilization may be electrooxidation. It is the research direction that (with a high probability) will allow, in the future, fuel cells to be powered by WEOs. Fuel cells operate without any intermediate steps (e.g., a combustion process) and are characterized by a high efficiency [26–31].

The feasibility of WEO utilization by electrooxidation was demonstrated in our previous work [32]. However, the previous work involved the research of electrooxidation on a platinum electrode. The high price of platinum has forced the search for other, cheaper catalysts that do not contain precious metals [33–36]. Another catalyst with good catalytic parameters is nickel. However, pure nickel (e.g., Raney Ni) can be very difficult to use due to the fact that Raney Ni contains small amounts of absorbed hydrogen that may spontaneously ignite; thus, it should not be exposed to air. Therefore, this nickel form is most often supplied as an aqueous suspension [37]. For these reasons, nickel alloys (rather than pure nickel) is safer (and easier) to use compared with pure nickel. Other metals and metal alloys are also used as electrode catalysts; e.g., cobalt and its alloys or a metal alloy with boron [38–44]. It is essential to maintain the good catalytic properties of the new alloys.

Mainly to reduce costs (by reducing the use of precious metals) and also to obtain durable electrodes, it is extremely important to search for new metal (metal alloy) catalysts [34–36,39–47]. This paper presents our research of the electrooxidation feasibility of WEOs with the use of electrodes with Ni-Co and Cu-B catalysts.

2. Materials and Methods

In our research, WEOs from ten different cars were applied. The oils were obtained from a car service station from compression ignition engines (diesel) and spark ignition engines (gasoline and LPG). The mileage of the engines ranged from 10,000 to 30,000 km. Moreover, the age of the cars (and thus the engines) and their condition were not analyzed. Therefore, the engine oil wear was at different levels.

Mixed waste oils from car service stations (from various vehicles) are sent for reprocessing or disposal. Such mixtures of waste oils contain mainly engine oils, gearbox oils
and oils from steering gear, for example. Our research plan assumed the measurements of the electrooxidation of oils using only WEOs.

The oils used in the measurements were collected when cars were serviced (as a result, the oils are exchanged). During the oil sampling, the engine type and mileage from the last oil change were simultaneously recorded. The data of the collected samples of WEOs are presented in Table 1.

| Engine Oil Grade | Type of Engine | Capacity and Power | Oil Mileage |
|------------------|----------------|-------------------|-------------|
| 5W30             | Diesel²        | 1.5; 115 hp⁴      | 30,000 km   |
| 5W30             | Diesel²        | 1.6; 92 hp⁴       | 15,000 km   |
| 5W30             | Diesel²        | 3.0; 245 hp⁴      | 15,000 km   |
| 5W40             | Petroleum³     | 1.0; 68 hp⁴       | 15,000 km   |
| 10W40            | Petroleum³     | 3.0; 180 hp⁴      | 15,000 km   |
| 15W40            | Petroleum³     | 2.0; 105 hp⁴      | 10,000 km   |
| 10W40            | Petroleum³     | 1.0; 72 hp⁴       | 15,000 km   |
| 10W40            | LPG³           | 2.0; 115 hp⁴      | 15,000 km   |
| 10W40            | LPG³           | 1.4; 75 hp⁴       | 10,000 km   |

1 Data derived from the car service station; 2 turbo; 3 naturally aspirated; 4 metric horsepower; 5 fuel injection.

To simulate the real oils sourced from car service stations, the WEOs were mixed in equal proportions (1:1:1:1:1:1:1:1:1:1). After mixing the oils, decantation was performed then filtering using a viscose filter. These treatments allowed the solid contaminants to be cleaned from the oil, resulting in normal engine operation. Figure 2 shows the WEO mixed in equal proportions.

Figure 2. The decanted, filtered and mixed in equal proportions waste engine oil (WEO).

To allow the oil to mix with the electrolyte and to ensure electricity conduction, an oil emulsion preparation was planned. Syntanol DS-10 was used to form the emulsion. That detergent is characterized by its emulsification, solubilization capabilities, dispersion and high superficial activity [48,49]. Moreover, Syntanol DS-10 is biodegradable (e.g., by bacteria) [50,51]. The WEO emulsion was obtained by mixing oil (WEO), water and Syntanol DS-10. The mixing was carried out by means of a mechanical stirrer (1200 rpm). The oil–water–detergent ratio was selected experimentally and was finally 1:2:1. This ratio of ingredients ensured a long emulsion stability of about 20 min. The stability of the WEO emulsion specified in the analysis changed the color uniformity of the images in the emulsion sample. The emulsion was exposed to white light on a white background and a photo was taken every minute. The comparison of changes in the color uniformity allowed the evaluation of the emulsion stability time.
The catalysts were electrochemically deposited onto a copper electrode. A copper electrode in the form of a foam was used. The electrode parameters were 20 × 40 × 5 mm with foamed copper at 100 PPI. Before the deposition, the foam copper was prepared in several steps [43,44,52,53]: the surface was degreased in a 25% aqueous solution of KOH to obtain the complete wettability of the electrode; the electrode was then digested in acetic acid and subsequently washed with alcohol.

To obtain the Ni-Co alloy, a mixture composed mainly of NiSO₄ and CoSO₄ was used [51,52]. For conducting the electrochemical deposition, temperatures of 20–50 °C and a pH range of 2.0–5.5 were used [54]. To obtain the Cu-B alloy, a mixture of mainly NaBH₄ and CuSO₄ was used [53]. To conduct this process, temperatures of 80–90 °C and a pH range of 2.0–5.0 were applied [44,52]. Current densities for both alloy types in the range of 1–3 A·dm⁻² were used [52,54].

Based on experience from previous research [44,53–57], we chose catalysts (a Ni-Co alloy with 15% Co and a Cu-B alloy with 9% B) for the electrooxidation of the WEO emulsion. The electrodes with the Ni-Co and Cu-B catalysts were used as the working electrodes (WE).

Figure 3 contains a diagram of the measurement setup for the measurements of the WEO emulsion electrooxidation.

Figure 3. Measurement setup for WEO electrooxidation with new catalysts. (A) Electrochemical deposition of the alloys: 1-components; 2—foam copper electrode; 3—chemical bath; 4—power supply. (B) Preparation of the WEO emulsion: 5—WEO samples; 6—filtration; 7—mixed oil; 8—water; 9—detergent; 10—emulsion preparation; 11—mechanical stirrer. (C) Electrooxidation of the WEO emulsion: 12-reactor (electrochemical cell); 13—magnetic stirrer; 14—potentiostat; 15—computer.

The measurements of the WEO emulsion electrooxidation were made using the method of polarization curves. Electrooxidation in a glass vessel (reactor) was made on an electrode (working electrode (WE)) with the new catalysts (Ni-Co and Cu-B) in a water solution of sulfuric acid (0.5 M and 2 M). The measurements were carried out using a potentiostat. A saturated calomel electrode (SCE) was used as the reference electrode (RE) [55–59]. It should be noted that the measurement results were not related to the real
working electrode (WE) surface but only to its dimensions. Figure 4 contains a diagram of the reactor (number 12 from block C of Figure 3).

Figure 4. Electrochemical cell (reactor): 1—WEO emulsion (or pure detergent); 2—auxiliary electrode (AE); 3—electrolyte; 4—stirrer bar; 5—connections; 6—reference electrode (RE); 7—Luggin capillary; 8—working electrode (WE) with Ni-Co or Cu-B catalyst.

Figure 5 shows a view of the measurement setup for the measurements of WEO electrooxidation (block C of Figure 3).

The electrooxidation of the waste oil emulsions in a water solution of sulfuric acid (0.5 M and 2 M) for various concentrations of WEO and pure detergent at different temperatures (20–80 °C) were measured. The concentrations of the WEO emulsion were 0.005%, 0.010%, 0.030% and 0.060% of the reactor volume (electrolyte volume). The electrooxidation of pure Syntanol DS-10 (added to the electrolyte in the same percentage as the WEO) itself was then measured. A comparison of the electrooxidation of pure Syntanol DS-10 with the electrooxidation of the WEO allowed us to assess whether the electric current was generated from the WEO or only from the detergent.

A PowerLab 305D-IL power supply (PowerLab, China) was used to electrochemically deposit the catalyst onto the copper electrodes. A LAC LH06/12 silt furnace (LAC s.r.o., Židlochovice, Czech Republic) was used for the electrode oxidation. The emulsion was mixed using a CAT R17 mechanical stirrer (Ingenieurbüro CAT M. Zipperer GmbH, Staufen, Germany). For the pictures for the emulsion separation analysis, a D5100 camera (Nikon Co., Tokyo, Japan) was used. A technoKartell model TK 22 magnetic stirrer with a hot plate (Kartell S.p.A.—LABWARE Division, Noviglio, Italy) was used to mix the electrolyte with the emulsion in the glass vessel (reactor). For the measurements of the electrooxidation
of the WEO emulsion, an AMEL System 500 potentiostat (Amel S.l.r., Milano, Italy) was used. For the data collection and the electrooxidation analysis, CorrWare software (Scribner Associates Inc., Southern Pines, NC, USA) was used.

3. Results

3.1. Electrooxidation of Waste Engine Oil Emulsion on the Ni-Co Catalyst

Figure 6 presents the electrooxidation curves of the pure detergent (blue lines) and the WEO emulsion (black lines) in a 0.5 M water solution of sulfuric acid for temperatures of 20 °C and 40 °C on the Ni-Co catalyst. The concentrations of the pure detergent (blue lines) and the WEO emulsion (black lines) were in the range of 0.005–0.060% (of the electrolyte volume).

Figure 7 presents the electrooxidation curves of the pure detergent (blue lines) and the WEO emulsion (black lines) in a 0.5 M water solution of sulfuric acid for temperatures of 60 °C and 80 °C on the Ni-Co catalyst. The concentrations of the pure detergent (blue lines) and the WEO emulsion (black lines) were in the range of 0.005–0.060% (of the electrolyte volume).
Figure 8 presents the electrooxidation curves of the pure detergent (blue lines) and the WEO emulsion (black lines) in a 2 M water solution of sulfuric acid for temperatures of 20 °C and 40 °C on the Ni-Co catalyst. The concentrations of the pure detergent (blue lines) and the WEO emulsion (black lines) were in the range of 0.005–0.060% (of the electrolyte volume).

![Electrooxidation curves of WEO emulsion (black lines) and detergent (blue lines) for various concentrations of detergent and emulsion on the Ni-Co catalyst for an electrolyte concentration of 2 M.](image)

Figure 8. Electrooxidation curves of WEO emulsion (black lines) and detergent (blue lines) for various concentrations of detergent and emulsion on the Ni-Co catalyst for an electrolyte concentration of 2 M. Electrooxidation: (a) temperature: 20 °C; (b) temperature: 40 °C.

Figure 9 presents the electrooxidation curves of the pure detergent (blue lines) and the WEO emulsion (black lines) in a 2 M water solution of sulfuric acid for temperatures of 60 °C and 80 °C on the Ni-Co catalyst. The concentrations of the pure detergent (blue lines) and the WEO emulsion (black lines) were in the range of 0.005–0.060% (of the electrolyte volume).

![Electrooxidation curves of WEO emulsion (black lines) and detergent (blue lines) for various concentrations of detergent and emulsion on the Ni-Co catalyst for an electrolyte concentration of 2 M.](image)

Figure 9. Electrooxidation curves of WEO emulsion (black lines) and detergent (blue lines) for various concentrations of detergent and emulsion on the Ni-Co catalyst for an electrolyte concentration of 2 M. Electrooxidation: (a) temperature: 60 °C; (b) temperature: 80 °C.

3.2. Electrooxidation of Waste Oil Emulsion on the Cu-B Catalyst

Figure 10 presents the electrooxidation curves of the pure detergent (blue lines) and the WEO emulsion (black lines) in a 0.5 M water solution of sulfuric acid for temperatures of 20 °C and 40 °C on the Cu-B catalyst. The concentrations of the pure detergent (blue lines) and the WEO emulsion (black lines) were in the range of 0.005–0.060% (of the electrolyte volume).
Figure 10. Electrooxidation curves of WEO emulsion (black lines) and detergent (blue lines) for various concentrations of detergent and emulsion on the Cu-B catalyst for an electrolyte concentration of 0.5 M. Electrooxidation: (a) temperature: 20 °C; (b) temperature: 40 °C.

Figure 11 presents the electrooxidation curves of the pure detergent (blue lines) and the WEO emulsion (black lines) in a 0.5 M solution of sulfuric acid for temperatures of 60 °C and 80 °C on the Cu-B catalyst. The concentrations of the pure detergent (blue lines) and the WEO emulsion (black lines) were in the range of 0.005–0.060% (of the electrolyte volume).

Figure 12 presents the electrooxidation curves of the pure detergent (blue lines) and the WEO emulsion (black lines) in a 2 M solution of sulfuric acid for temperatures of 20 °C and 40 °C on the Cu-B catalyst. The concentrations of the pure detergent (blue lines) and the WEO emulsion (black lines) were in the range of 0.005–0.060% (of the electrolyte volume).

Figure 13 presents the electrooxidation curves of the pure Syntanol DS-10 detergent (blue lines) and the WEO emulsion (black lines) in a 2 M solution of sulfuric acid for temperatures of 60 °C and 80 °C on the Cu-B catalyst. The concentrations of the pure Syntanol DS-10 detergent (blue lines) and the WEO emulsion (black lines) were in the range of 0.005–0.060% (of the electrolyte volume).
This was the case for both catalysts (Ni-Co and Cu-B) and for both electrolyte concentrations (0.5 M and 2 M). Therefore, this research made it possible for us to determine the results were not related to the real physical electrode surface but its dimensions, as noted surface of the electrodes. However, these values cannot be directly compared in relation of the obtained current density beforehand.

The electrooxidation of WEOs was carried out for both catalysts (Ni-Co and Cu-B) and for all concentrations of emulsions and both concentration of electrolyte as well as for all measurement temperatures (Figures 7b, 9b, 11b and 13b). This was the case for both catalysts (Ni-Co and Cu-B) and for both electro-oxidation curves of detergent (blue lines) and uric acid used as the electrolyte). It was specified that the electrooxidation of the detergent (blue lines in Figures 7b, 9b, 11b and 13b) took place first. At 80 °C, during the WEO emulsion electrooxidation, the obtained current density was lower (black lines in Figures 7b, 9b, 11b and 13b). This was the case for both catalysts (Ni-Co and Cu-B) and for both electrolyte concentrations (0.5 M and 2 M). Therefore, this research made it possible for us to determine the boundary temperature of use of the WEO as a fuel with the use of Ni-Co and Cu-B catalysts.

**4. Discussion**

An SCE was used as the reference electrode in the research. The use of the SCE made the measurements easier. However, all measurement results (Figures 6–13) included in the study were converted and related to a reversible hydrogen electrode (RHE) [58,59].

**Figure 12.** Electrooxidation curves of WEO emulsion (black lines) and detergent (blue lines) for various concentrations of detergent and emulsion on the Cu-B catalyst for an electrolyte concentration of 2 M. Electrooxidation: (a) temperature: 20 °C; (b) temperature: 40 °C.

**Figure 13.** Electrooxidation curves of WEO emulsion (black lines) and detergent (blue lines) for various concentrations of detergent and emulsion on the Cu-B catalyst for an electrolyte concentration of 2 M. Electrooxidation: (a) temperature: 60 °C; (b) temperature: 80 °C.
(in an aqueous solution of sulfuric acid used as the electrolyte). It should be noted that this situation was similar to the measurement results obtained using a platinum catalyst [32].

The current density for all measurements (Ni-Co and Cu-B catalysts, for both electrolyte concentrations and for all measurement temperatures) of the electrooxidation of the WEO emulsion was in the range of 1–11 mA·cm$^{-2}$. The highest value of current density (11 mA·cm$^{-2}$) was recorded for the Ni-Co catalyst (0.010% concentration of the electrolyte volume) at a temperature of 60 °C (a 0.5 M aqueous solution of sulfuric acid) (Figure 7a). The maximum current density (7 mA·cm$^{-2}$) for the Cu-B catalyst was recorded (0.030% concentration of the electrolyte volume) at a temperature of 60 °C (a 0.5 M aqueous solution of sulfuric acid) (Figure 11a).

It should be noted that the values of the current densities were slightly lower than the current densities obtained under the same conditions using the platinum catalyst (about half) [32]. These were comparable values regarding those related to the dimensions of the electrodes. However, these values cannot be directly compared in relation to the surface of real electrodes. This situation occurred due to the fact that the measurement results were not related to the real physical electrode surface but its dimensions, as noted beforehand. The measurements on the platinum electrode were carried out on a smooth surface electrode (with a Pt catalyst) whereas the measurements with the use of the Ni-Co and Cu-B catalysts were carried out on a foam copper electrode with a deposited catalyst [32]. The surface was defined as the visible surface (resulting from the dimensions).

5. Conclusions

Based on the data from the measurements, the feasibility of WEO electrooxidation with Ni-Co and Cu-B catalysts has been demonstrated. In a temperature range of 20–80 °C, the electrooxidation of the WEO emulsion took place for both catalysts (Ni-Co and Cu-B) for all emulsion concentrations and in both concentrations of acid electrolyte. The maximum current density was obtained for the Ni-Co catalyst at a temperature of 60 °C (0.010% of the WEO emulsion; 0.5 M H$_2$SO$_4$). The recorded value of the current density was 11 mA·cm$^{-2}$.

As previously noted (in the Materials and Methods), the measurement results were not related to a real electrode surface, only to its dimensions. However, regarding the dimensions, the comparable size of the electrodes with the Ni-Co and Pt catalysts allowed us to obtain similar current density values (the measurements with the Cu-B catalyst resulted in lower current density values). Such measurement results allow us to hope to obtain catalysts that do not contain precious metals whilst maintaining a similar level of catalytic efficiency. Therefore, there is a chance for a significant reduction in costs when replacing platinum catalysts with a Ni-Co catalyst (or Cu-B, if it is possible to accept the lower performance).

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