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**DOCTORAL THESIS**

**(abstract)**

**Contributions on the analysis of  
mineral and biodegradable  
hydraulic fluids for industrial  
systems**

***Scientific leader,***  
**Prof.univ.dr.ing. Sorin CĂNĂNĂU**

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## KEYWORDS

Rheology, Mineral Hydraulic Oil (H46), Biodegradable Hydraulic Oil (HETG46 and HF-E46), Viscosity, Thermal Degradation, FTIR Spectroscopy, Pressure, Flow Rate.

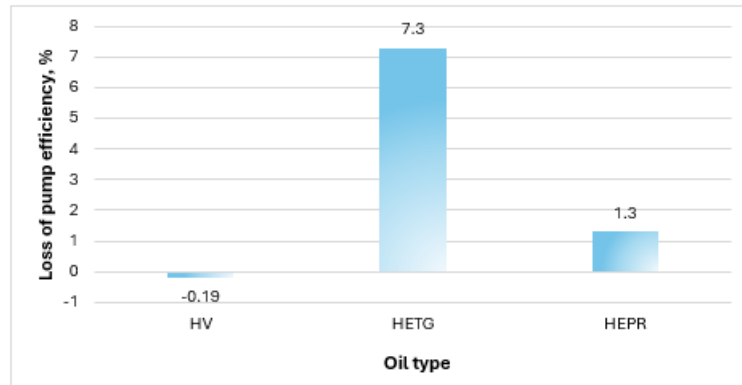
## SUMMARY OF THE CHAPTERS PRESENTED IN THE DOCTORAL THESIS

Chapter 1 entitled "*Current state of research on hydraulic lubricants*" presents general notions regarding mineral and biodegradable hydraulic oils used in hydraulically driven systems, their impact on the environment and at the same time the increased interest in the study of the performance of oils, as well as the understanding of the behavior of the oils under different operating conditions.

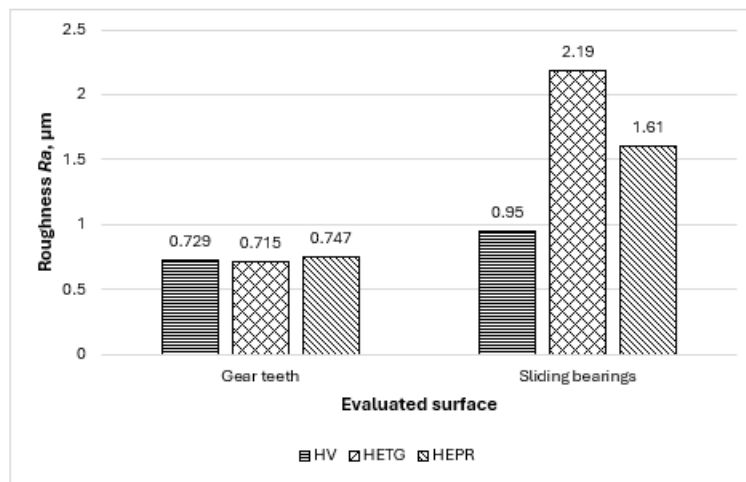
Mineral oil-based fluids, although they exhibit good lubricating properties in industrial applications, have a negative impact on the environment, such as water and soil contamination and greenhouse gas emissions. Thus, the implementation of biodegradable oils as substitutes for mineral oils was considered. By obtaining and implementing biodegradable oils, in addition to reducing the impact on the environment, they have a viscosity index and high lubrication, extend the life of the equipment and at the same time stimulate the agricultural economy by harnessing renewable resources [8], [26].

In the experimental study by Majdan et al. [31] the impact of two biodegradable hydraulic oils (HETG-vegetable oils and HEPR-polyalphaolefins) was evaluated compared to one mineral hydraulic oil (HV) on the wear of the hydraulic gear pump, using a special cyclic pressure loading test bench. The parameters evaluated were the efficiency of the hydraulic pump, the roughness of the surfaces of the internal components and their material losses.

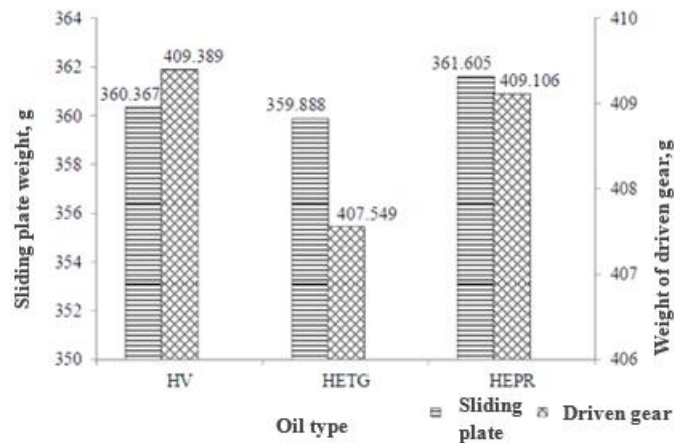
The results obtained showed that the pump when using HEPR and HV oils recorded the smallest variations in efficiency, Figure 1.2, and material losses, Figure 1.4, compared to the situation in which it used the HETG biodegradable oil. The use of HETG oil resulted in a higher degree of wear compared to the other two oils, characterized by component weight losses, high values of the plain bearing roughness (Figure 1.3) and decreased pump efficiency. The pump wear results indicate that the materials from which the components are made are of high quality, with reduced wear in all cases. Although the use of biodegradable oil resulted in higher pump wear and efficiency losses compared to the other two oils tested, the operation of the pump was not negatively influenced.



**Figure Error! No text of specified style in document..1 Loss of hydraulic pump efficiency after testing with different types of oil [31]**



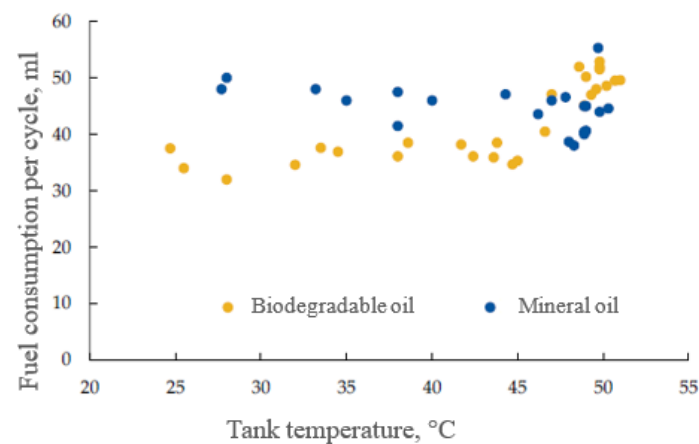
**Figure 1.3 Comparison of roughness of hydraulic pump components after testing with different oils [31]**



**Figure Error! No text of specified style in document..2 Comparison of material losses of bearing bushings and driven gears by weight [31]**

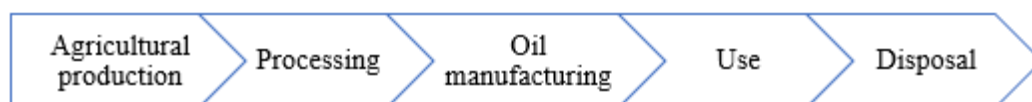
The viscosity influence of two hydraulic oils, namely HLP (mineral hydraulic oil) and HEES (biodegradable hydraulic oil based on synthetic esters), on the operating efficiency of a crawler excavator, was analyzed by Deuster et al. [35]. The study highlights that, in addition

to reducing the environmental impact by using biodegradable oils, their rheological properties, especially viscosity, directly influence the energy efficiency of excavators. By using biodegradable hydraulic oil, its viscosity impacts losses in the hydraulic system and, implicitly, fuel consumption Figure 1.6. This paper shows that by using biodegradable oils, the volumetric and mechanical efficiency of hydraulic components increases, which is reflected in reduced fuel consumption, compared to mineral hydraulic oil.

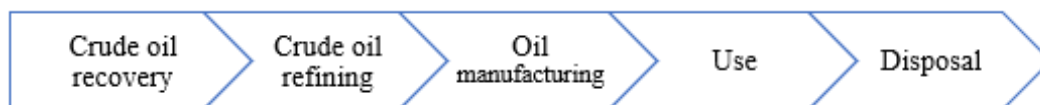


**Figure Error! No text of specified style in document..3 Fuel consumption according to tank temperature [35]**

The life cycle stages of mineral and biodegradable hydraulic oils, Figure 1.8 and Figure 1.9, have also been presented in this chapter.



**Figure 1.8 Life cycle stages of biodegradable oil [21]**



**Figure 1.9 Life cycle stages of mineral oil [21]**

### Objectives of the thesis

In recent years, the economic environment is increasingly required to demonstrate its commitment to a cleaner environment, especially in terms of the sustainability report.

A major concern is to obtain hydraulic oils that correspond to this purpose, respectively their correct choice for the operation of industrial installations. Finally, the use of

biodegradable hydraulic oils is necessary to be evaluated, as they can positively influence environmental, social and governance performance.

Thus, the general objective of the thesis is to highlight the performance of hydraulic systems by using biodegradable oils and comparing them with classic systems using mineral oils.

The thesis aims to achieve the following objectives:

- achieving the current state of research in the field;
- determination of the rheological parameters (viscosity, consistency index and flow index) under the assumption of the validity of the Oswald-de Waele and Newtonian rheological models of three types of fresh hydraulic oils, namely H46 (mineral hydraulic oil), HETG46 (vegetable oil-based hydraulic oil), HF-E46 (biodegradable hydraulic oil based on synthetic esters derived from vegetable oil fatty acids);
- determination of the variation of viscosity with temperature, using the Reynolds thermal model, which is an exponential model;
- development of a working methodology regarding the performance of the rheological study on test oils;
- thermal degradation of the studied oils and at the same time their rheological study;
- performing chemical analysis on fresh and thermally degraded oils, by infrared spectroscopy with Fourier transformation and comparing them, to identify new types of chemical bonds in their structure;
- the realization of an experimental stand for testing oils and determining the hydraulic parameters through which it is possible to identify the efficiency of the hydraulic system;
- simulation of the experimental stand through the Simcenter Amesim program and comparison of the results with those obtained on the experimental stand within the laboratory.

In the doctoral thesis, mineral and biodegradable hydraulic oils were studied in the category of hydraulic fluids/lubricants.

In Chapter 2 entitled "*Rheological Study of Hydraulic Oils*" the rheological properties and classification of fluids are presented, followed by the presentation of the equipment used for testing hydraulic fluids together with the data acquisition and processing system, the rheological models used and the test methodology.

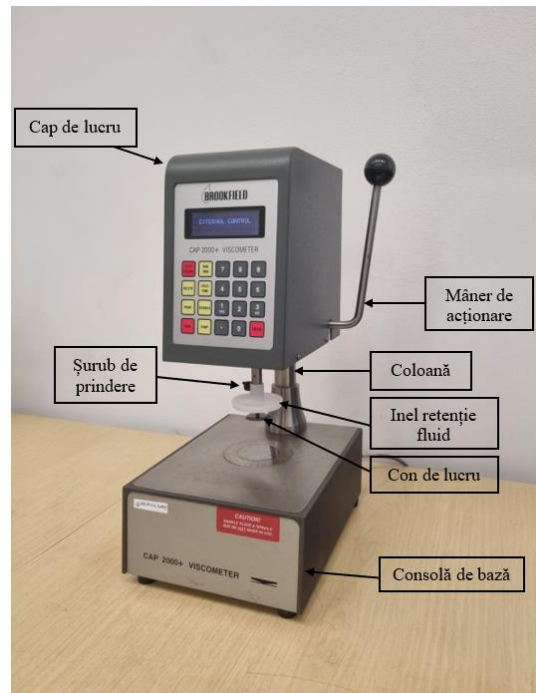
According to [74], rheological properties are mainly defined by *viscosity and elasticity*. While *viscosity* is specific to fluid materials, in which the deformation continues to increase over time, without returning to its original shape after the stress has been removed, *elasticity* is characteristic of solid materials, which, once the external stress is removed, completely regain its original shape. A third property called *plasticity* intervenes, the result of the appearance of the viscosity and elasticity property on the body

The test equipment used in the experiments was a Couette rotary viscometer, with cone-plate geometry, Brookfield CAP2000+, controlled by a CAPCALC 32 software from the Department of Machine Parts and Tribology within POLITEHNICA Bucharest, Figure 2.5, and in Figure 2.6 its components are presented.



**Figure Error! No text of specified style in document..4 Rotary viscometer Couette, Brookfield CAP2000+**





*Figure 2.6 Viscometer components*

This viscometer has been designed for viscosity measurement using con-plate geometry, which makes it possible to apply shear speeds. The geometry of the con-plate viscometer shows uniform shear rate and allows the measurement of normal tension. The viscometer with con-plate geometry is the most widely used device for determining the rheological properties of a Newtonian or non-Newtonian fluid. It is formed between a cone and a plate, and in the middle of them is the fluid [90].

The Brookfield CAP2000+ viscometer is controlled by a software called CAPCALC 32 ("CAPCALC 32 - Brookfield AMETEK" 2018) which allows the control of the viscometer from a computer for automatic testing, programming function, collection and storage of test data, numerical data processing, as well as general setting of the working parameters of the viscometer. Built-in mathematical models help to quickly and easily analyze material flow behavior, flow curves/viscosity can be drawn, can record up to 1000 data points per test, and create a permanent record of the test in cP or mPa·s, generates up to four sets of data simultaneously in 12 types of diagrams and provides flow voltage calculations [91].

The concept of rheological model is the basic tool that characterizes rheology. The rheological model is represented by a mathematical relationship between stresses, strains or strain rate, and material coefficients [78]. By interpreting these models, one can identify the

behavior of fluids, whether they are non-Newtonian fluids dependent or time-independent, or fluids with elasticity or plasticity properties [92].

The oils testing program was carried out through the CAPCAL32 program that has viscometer. With the help of this calculation system, the parameters of the rheological model of the studied oils were determined in the hypothesis of the validity of the Oswald-de Waele model (Law of Power), and the parameters of the Newtonian rheological model were determined by means of the Excel calculation program.

The oils were tested at a constant temperature of 20°C, in a range of shear rate between 100 s<sup>-1</sup> and 2000 s<sup>-1</sup>, equivalent to a cone speed between 50-1000 rpm, using cone number 8, which has a cone radius of 15.11 mm, the top angle of the cone is 3°, and the viscosity measurement range being between 0.312...3.12 Pa·s.

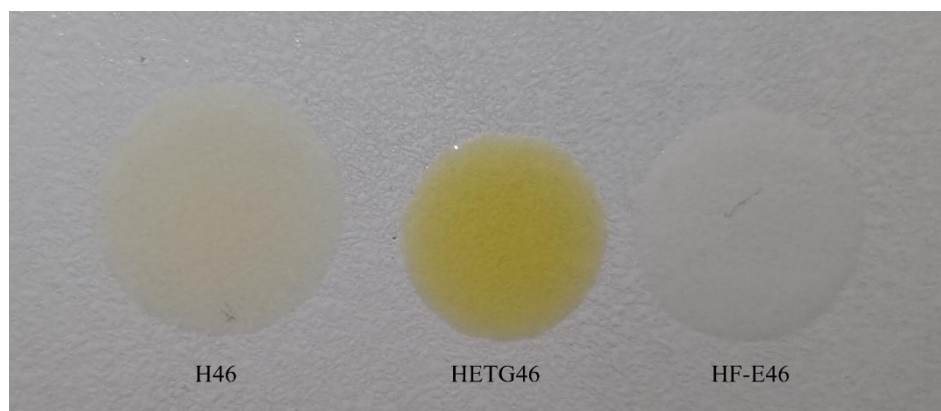
To highlight the variation of viscosity as a function of temperature, the CAPCAL32 software was used, through which the test program was carried out.

The test program has been designed to measure temperature in the range of 15°C... 75°C, at a constant shear rate of 250 s<sup>-1</sup>, 500 s<sup>-1</sup>, 750 s<sup>-1</sup> and 1000 s<sup>-1</sup> respectively, with a cone speed of 125 rpm, 250 rpm, 375 rpm and 500 rpm respectively.

To determine the variation of rheological parameters with temperature, the Reynolds model was applied, which is an exponential type of thermal model. The oil sample was initially homogenized for 2 minutes, after which the sample homogenization time for each measured point was approximately 1 minute (soaking time).

Chapter 3 entitled "*Rheological study of fresh hydraulic oils*", the studied hydraulic oils and their properties according to the technical sheets, subchapter 3.1, were presented. In subchapter 3.2 the results obtained from the rheological study of their fresh state were highlighted.

Figure 3.1 shows the visual appearance of the three hydraulic oils used in rheological tests.



*Figure 3.1 Hydraulic oils used in experimental testing*

In the study by Olszak et al. [103] the physicochemical and tribological properties of biodegradable hydraulic oils are highlighted. Following the results obtained, it was observed that these biodegradable hydraulic oils can be considered alternatives to mineral hydraulic oils, due to their high biodegradability, low toxicity and favorable physicochemical properties. These physicochemical properties were also highlighted in the study by Petlyuk et al. [104] and the study by Uppar et al. [105].

This category of biodegradable hydraulic oils exhibits good physicochemical properties such as low toxicity, high biodegradability, favorable viscosity behavior as a function of temperature [35], shear stability, low volatility and high viscosity index. However, they are prone to oxidation and thermal instability compared to mineral oils [27].

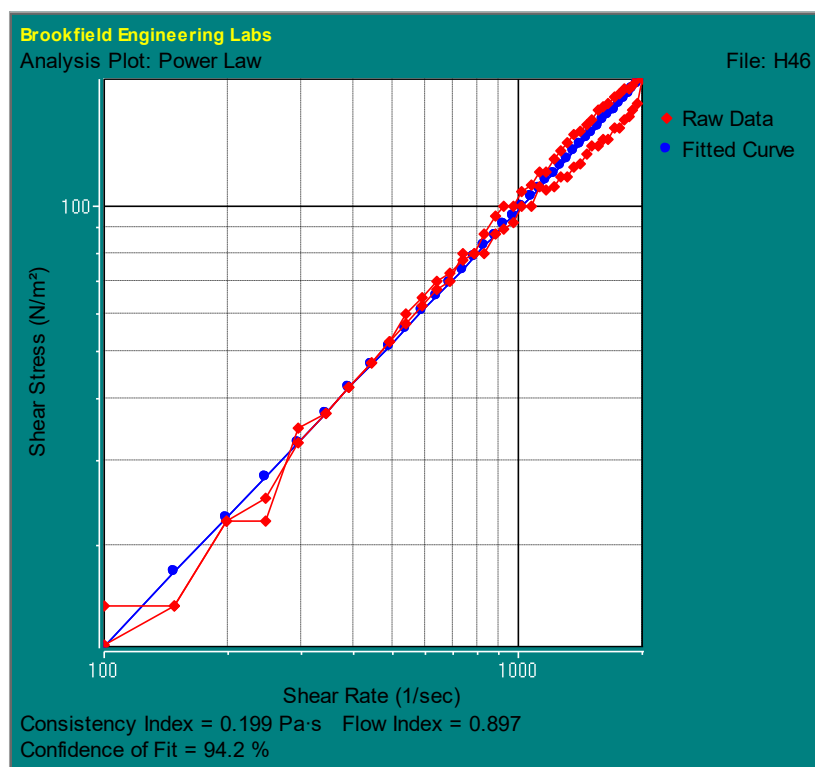
Table 3.1 shows the physicochemical properties of hydraulic oils used in experimental testing.

*Table 3.1 Physicochemical properties of hydraulic oils tested [95], [96], [97]*

| <b>Oil</b>                                          | <b>H46</b> | <b>HETG46</b> | <b>HF-E46</b> |
|-----------------------------------------------------|------------|---------------|---------------|
| <b>Properties</b>                                   | ISO 11158  | ISO 15380     | ISO 15380     |
| Viscosity class                                     | 46         | 46            | 46            |
| Relative density at 15°C, max [kg/m <sup>3</sup> ]  | 876        | 918           | 921           |
| Viscosity index, min.                               | 98         | 210           | 188           |
| Kinematic viscosity at 40°C, [cSt]                  | 44         | -             | 47,2          |
| Kinematic viscosity at 100°C, [cSt]                 | 6,6        | 10            | 9,41          |
| Flash point, min. [°C]                              | 226        | >270          | 320           |
| Flow point, max. [°C]                               | -24        | -30           | -42           |
| Corrosive action on copper blade, 3h, max, 100 [°C] | 1A         | -             | -             |

|                                       |   |     |      |
|---------------------------------------|---|-----|------|
| Acid value, [mg KOH/g]                | - | 0,5 | -    |
| Parts of renewable raw materials, [%] | - | 95  | 76   |
| Temperature to open [°C]              | - | -   | >400 |

Figure 3.6, Figure 3.13, Figure 3.20 show the parameters of the rheological power law model, obtained for H46 oil at a temperature of 20°C, by regression applied to experimental data using the CAPCALC 32 software, assuming the validity of this model.



*Figure 3.6 Numerical regression of H46 hydraulic oil at 20°C using the power law model*

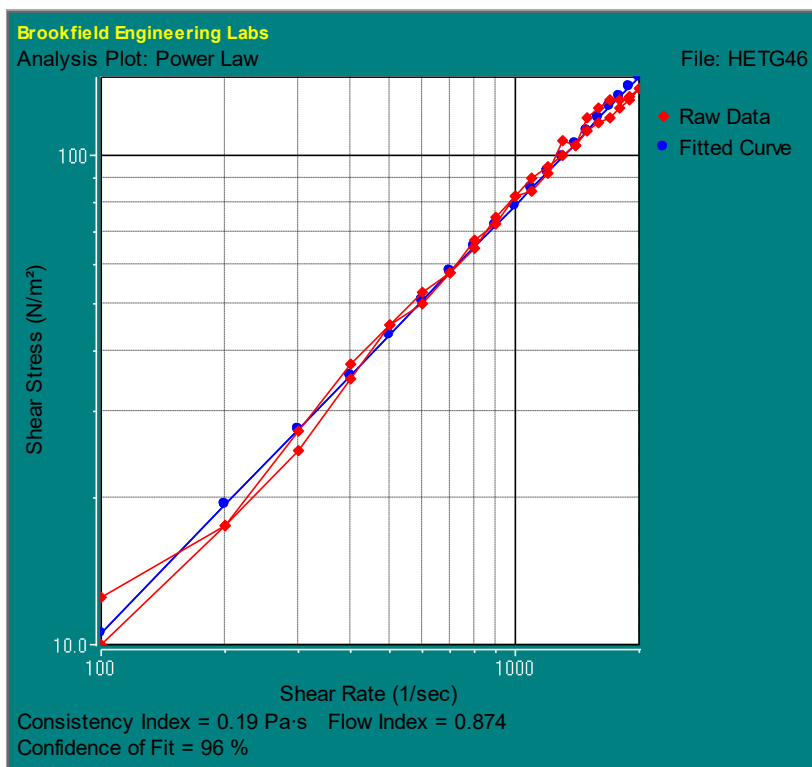


Figure 3.13 Numerical regression of HETG46 biodegradable hydraulic oil at 20°C using the power law

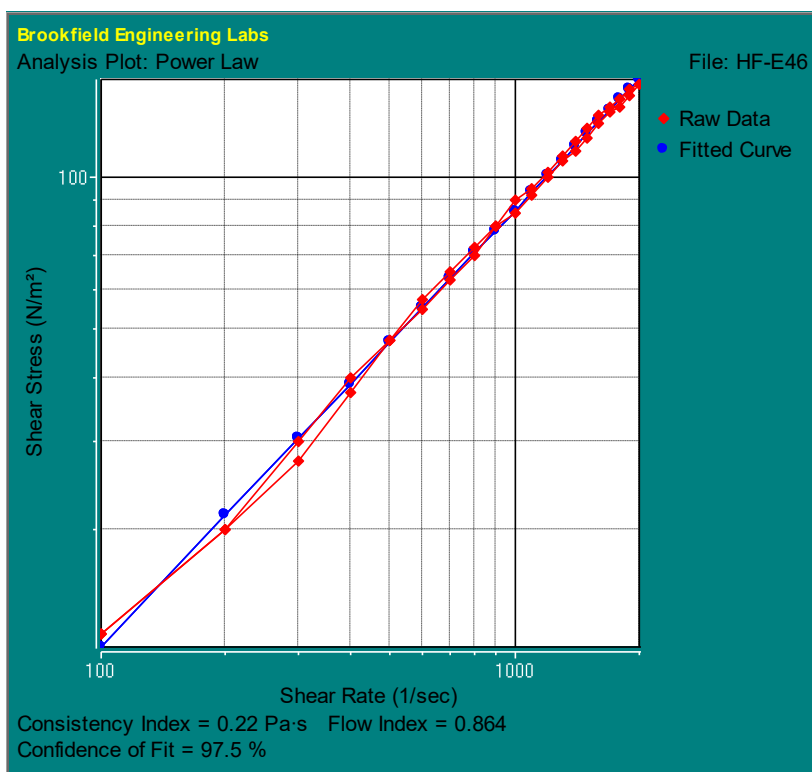


Figure 3.20 Numerical regression of HF-E46 hydraulic oil at 20°C using the power law

Table 3.3, Table 3.6, Table 3.9 summarize the results obtained from the application of the two rheological models. Analyzing the results in the tables, the power law model has a

lower correlation coefficient compared to the Newtonian model, we can say that the model that best approximates the rheological behavior of these fluids is the Newtonian model.

*Table 3.3 Rheological parameters of H46 hydraulic oil at 20°C*

| Oil Type   | Rheological model    |                                    |                                  |               |                                 |
|------------|----------------------|------------------------------------|----------------------------------|---------------|---------------------------------|
|            | Newtonian            |                                    | The Law of Power                 |               |                                 |
|            | Viscosity,<br>[Pa·s] | Correlation<br>coefficient,<br>[%] | Consistency<br>index,<br>[Pa·sn] | Flow<br>Index | Correlation<br>coefficient, [%] |
| <b>H46</b> | 0,0936               | 99,29                              | 0,1991                           | 0,8971        | 94,2                            |

*Table 3.6 Rheological parameters of HETG46 hydraulic oil at 20°C*

| Oil Type      | Rheological model    |                                    |                                  |               |                                 |
|---------------|----------------------|------------------------------------|----------------------------------|---------------|---------------------------------|
|               | Newtonian            |                                    | The Law of Power                 |               |                                 |
|               | Viscosity,<br>[Pa·s] | Correlation<br>coefficient,<br>[%] | Consistency<br>index,<br>[Pa·sn] | Flow<br>Index | Correlation<br>coefficient, [%] |
| <b>HETG46</b> | 0,0752               | 99,49                              | 0,19                             | 0,874         | 96                              |

*Table 3.9 6 Rheological parameters of HF-E46 hydraulic oil at 20°C*

| Oil Type      | Rheological model    |                                    |                                  |               |                                 |
|---------------|----------------------|------------------------------------|----------------------------------|---------------|---------------------------------|
|               | Newtonian            |                                    | The Law of Power                 |               |                                 |
|               | Viscosity,<br>[Pa·s] | Correlation<br>coefficient,<br>[%] | Consistency<br>index,<br>[Pa·sn] | Flow<br>Index | Correlation<br>coefficient, [%] |
| <b>HF-E46</b> | 0,0817               | 99,67                              | 0,22                             | 0,864         | 97,5                            |

Table 3.4, Table 3.7, Table 3.10 show the experimental results of hydraulic oils using the Reynolds thermal model. Analyzing these results, it is observed that the apparent viscosity at the temperature of 50°C ( $\eta_{50^\circ}$ ) decreases with the increase of the shear rate, and the coefficient of variation of the viscosity with temperature ( $m$ ) increases.

*Table 3.4 Characteristic parameters for viscosity variation as a function of temperature using the Reynolds model for H46 hydraulic oil*

| H46                     |                                                |                                                                                          |                                    |
|-------------------------|------------------------------------------------|------------------------------------------------------------------------------------------|------------------------------------|
| Parameters              | Viscosity at 50°C,<br>$\eta_{50^\circ}$ [Pa·s] | Coefficient of variation of<br>viscosity with<br>temperature,<br>$m$ [°C <sup>-1</sup> ] | Correlation<br>coefficient,<br>[%] |
| Shear rate,<br>$s^{-1}$ |                                                |                                                                                          |                                    |

|             |        |       |       |
|-------------|--------|-------|-------|
| <b>250</b>  | 0,0300 | 0,039 | 96,71 |
| <b>500</b>  | 0,0250 | 0,045 | 98,40 |
| <b>750</b>  | 0,0266 | 0,048 | 99,51 |
| <b>1000</b> | 0,0200 | 0,057 | 99,89 |

*Table Error! No text of specified style in document..1 Characteristic parameters for viscosity variation as a function of temperature using the Reynolds model for HETG46 hydraulic oil*

| HETG46                                |                                                |                                                                                       |                                 |
|---------------------------------------|------------------------------------------------|---------------------------------------------------------------------------------------|---------------------------------|
| Parameters<br>Shear rate,<br>$s^{-1}$ | Viscosity at 50°C,<br>$\eta_{50} [Pa \cdot s]$ | Coefficient of variation<br>of viscosity with<br>temperature,<br>$m [^{\circ}C^{-1}]$ | Correlation<br>coefficient, [%] |
| <b>250</b>                            | 0,0300                                         | 0,031                                                                                 | 96,62                           |
| <b>500</b>                            | 0,0250                                         | 0,036                                                                                 | 98,96                           |
| <b>750</b>                            | 0,0199                                         | 0,042                                                                                 | 99,52                           |
| <b>1000</b>                           | 0,0150                                         | 0,058                                                                                 | 99,36                           |

*Table Error! No text of specified style in document..2 Characteristic parameters for viscosity variation as a function of temperature using the Reynolds model for HF-E46 hydraulic oil*

| HF-E46                                |                                                               |                                                                                       |                                    |
|---------------------------------------|---------------------------------------------------------------|---------------------------------------------------------------------------------------|------------------------------------|
| Parameters<br>Shear rate,<br>$s^{-1}$ | Viscosity at<br>temperature<br>50°C, $\eta_{50} [Pa \cdot s]$ | Coefficient of variation<br>of viscosity with<br>temperature,<br>$m [^{\circ}C^{-1}]$ | Correlation<br>coefficient,<br>[%] |
| <b>250</b>                            | 0,0300                                                        | 0,035                                                                                 | 98,18                              |
| <b>500</b>                            | 0,0200                                                        | 0,041                                                                                 | 99,00                              |
| <b>750</b>                            | 0,0166                                                        | 0,050                                                                                 | 99,89                              |
| <b>1000</b>                           | 0,0175                                                        | 0,052                                                                                 | 99,84                              |

Based on these determinations of rheological properties, it was found that:

- Analyzing the results obtained for the correlation coefficients, it is observed that the Newtonian model best approximates the rheological behavior of the three hydraulic oils, the values of the correlation coefficient being greater than 95%. This result indicates that oils have a Newtonian behavior.
- From a thermal point of view, through the Reynolds relationship, it was observed that the variation of viscosity with temperature approximates the experimental values very well, having a correlation coefficient higher than 96%.

At the same time, based on the results obtained, a comparison between the oils was made, where it was found that:

- the biodegradable oils HF-E46 and HETG46 exhibit a very low thixotropy compared to the hydraulic oil H46 where its thixotropy is very well pronounced; the reason why biodegradable oils have a less pronounced thixotropy, compared to the mineral oil H46, is due to the time-dependent structural changes of the tested oil sample, caused by the shear rate, which causes the occurrence of the deformation hysteresis loop [106].
- HF-E46 biodegradable oil has a higher viscosity than HETG46 biodegradable oil and lower than H46 hydraulic oil, where its slope is higher, which means that the viscosity is also high;
- for the two biodegradable oils (HETG46 and HF-E46) no significant differences are identified in terms of viscosity variation with temperature.

In Chapter 4 entitled *"Rheological Study of Thermally Degraded Hydraulic Oils"*, hydraulic lubricants were thermally degraded and rheological parameters (viscosity, consistency index, flow index) were determined using the same rheological models (Power Law Model and Newtonian Model).

Depending on the flash point of each oil described in their data sheet, two or three temperatures have been established respectively at which the oils are to be thermally degraded, namely:

- for the mineral hydraulic oil H46, temperatures of 130°C and 150°C have been established;
- for the HETG46 and HF-E46 biodegradable hydraulic oils, temperatures of 150°C, 200°C and 220°C have been established.

The thermal degradation involved heating and cooling the oils in several cycles, as follows: a quantity of 200 ml of oil was used, which was introduced into a convective heat transfer heating equipment, in order to heat the fluid to the set temperature, for 15 minutes, then cooled until it reached a temperature of 22°C. This heating-cooling cycle was repeated 8 times.

Table 4.1 shows the average oil temperatures over the entire thermal degradation cycle.

*Table 4.1 Average temperatures corresponding to the thermal degradation cycle*

| Oil Type | 130°C | 150°C | 200°C | 220°C |
|----------|-------|-------|-------|-------|
|----------|-------|-------|-------|-------|



| Temperature   |      |       |       |       |
|---------------|------|-------|-------|-------|
| <b>H46</b>    | 88°C | 104°C | -     | -     |
| <b>HETG46</b> | -    | 101°C | 131°C | 162°C |
| <b>HF-E46</b> | -    | 94°C  | 142°C | 164°C |

The rheological parameters of thermally degraded oils were determined using the regression analysis method, in the hypothesis of the validity of two rheological models, namely, the Power Law Model and the Newtonian Model. The results are presented in Table 4.2, Table 4.4, Table 4.6.

*Table 4.2 Rheological parameters of H46 mineral hydraulic oil at 20°C*

| H46          | Rheological models            |                                    |                                                     |                    |                                    |
|--------------|-------------------------------|------------------------------------|-----------------------------------------------------|--------------------|------------------------------------|
|              | Newtonian                     |                                    | The Law of Power                                    |                    |                                    |
|              | Viscosity,<br>$\eta$ [Pa · s] | Correlation<br>coefficient,<br>[%] | Consistency<br>index,<br>$m$ [Pa · s <sup>n</sup> ] | Flow index,<br>$n$ | Correlation<br>coefficient,<br>[%] |
| <b>130°C</b> | 0,0922                        | 99,32                              | 0,167                                               | 0,919              | 93,90                              |
| <b>150°C</b> | 0,0927                        | 99,34                              | 0,165                                               | 0,922              | 94,00                              |

*Table 4.4 Rheological parameters of HETG46 biodegradable hydraulic oil at 20°C temperature*

| HETG46       | Rheological models            |                                    |                                                     |                          |                                    |
|--------------|-------------------------------|------------------------------------|-----------------------------------------------------|--------------------------|------------------------------------|
|              | Newtonian                     |                                    | The Law of Power                                    |                          |                                    |
|              | Viscosity,<br>$\eta$ [Pa · s] | Correlation<br>coefficient,<br>[%] | Consistency<br>index,<br>$m$ [Pa · s <sup>n</sup> ] | Curgere<br>index,<br>$n$ | Correlation<br>coefficient,<br>[%] |
| <b>150°C</b> | 0,0836                        | 99,55                              | 0,279                                               | 0,834                    | 96,9                               |
| <b>200°C</b> | 0,0882                        | 99,59                              | 0,229                                               | 0,869                    | 97,6                               |
| <b>220°C</b> | 0,0947                        | 99,62                              | 0,280                                               | 0,851                    | 97,6                               |

*Table 4.6 Rheological parameters of HF-E46 biodegradable hydraulic oil at 20°C*

| HF-E46       | Rheological models            |                                    |                                                     |                          |                                    |
|--------------|-------------------------------|------------------------------------|-----------------------------------------------------|--------------------------|------------------------------------|
|              | Newtonian                     |                                    | The Law of Power                                    |                          |                                    |
|              | Viscosity,<br>$\eta$ [Pa · s] | Correlation<br>coefficient,<br>[%] | Consistency<br>index,<br>$m$ [Pa · s <sup>n</sup> ] | Curgere<br>index,<br>$n$ | Correlation<br>coefficient,<br>[%] |
| <b>150°C</b> | 0,0835                        | 99,64                              | 0,229                                               | 0,861                    | 98,1                               |
| <b>200°C</b> | 0,0941                        | 99,67                              | 0,283                                               | 0,848                    | 98,3                               |
| <b>220°C</b> | 0,0998                        | 99,79                              | 0,195                                               | 0,908                    | 98,3                               |

From a rheological point of view, the data were analyzed in the hypothesis of the validity of two rheological models, namely, the Newtonian model and the law of power model. Using the regression analysis method, rheological parameters of the oils were determined, observing that the rheological model that best approximates the behavior of the three oils, both

fresh and thermally degraded, is the Newtonian model, because it has a correlation coefficient of over 99%, compared to the power law model, where the correlation coefficient is on average 96%.

From the results obtained, by applying the power law model, it was observed that the flow index is less than 1, which means that the oils have a pseudoplastic behavior, that is, they begin to flow regardless of the force that is applied to them. This pseudoplastic behavior is important in industrial applications where a decrease in viscosity can reduce fluid efficiency in hydraulic systems.

The oils studied are part of the category of time-dependent non-Newtonian fluids, which once subjected to shear, changes occur in their structure over time, both in shear stress and viscosity. This category of time-dependent non-Newtonian fluids also includes thixotropic fluids.

In Chapter 5 entitled *"Fourier Transform Infrared Spectroscopy Analysis of Hydraulic Oils"*, a spectroscopy analysis of both fresh and thermally degraded hydraulic oils was performed, and the comparison between them was subsequently performed.

The FTIR analysis of hydraulic oils was performed using the Nicolet iS50R spectrometer (Figure 5.1), equipped with a total attenuated reflection (ATR) accessory equipped with a fully reflective diamond crystal cell and a deuterated triglyceride sulfate (DTGS) detector.

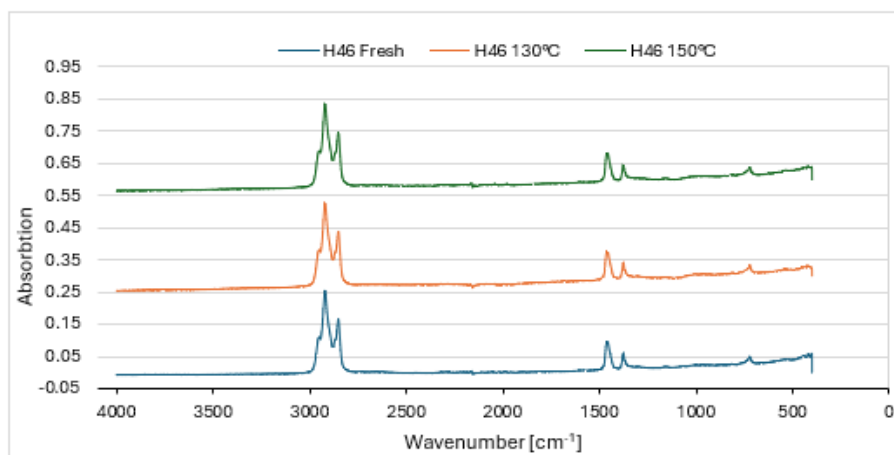


**Figure 5.1 FTIR spectrometer Thermo Nicolet iS50 [120]**

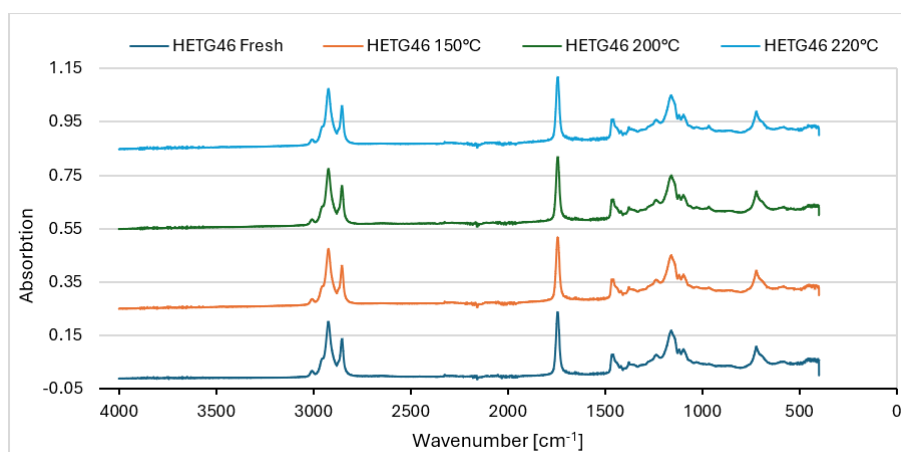
The oil samples were measured at room temperature, where 32 scans between 400 and 4000  $\text{cm}^{-1}$  were performed for each sample, with a resolution of 4  $\text{cm}^{-1}$ . The recording of the

spectrum was achieved by connecting a computer to the spectrometer, and the processing of the results was possible thanks to the OMNIC 32 program.

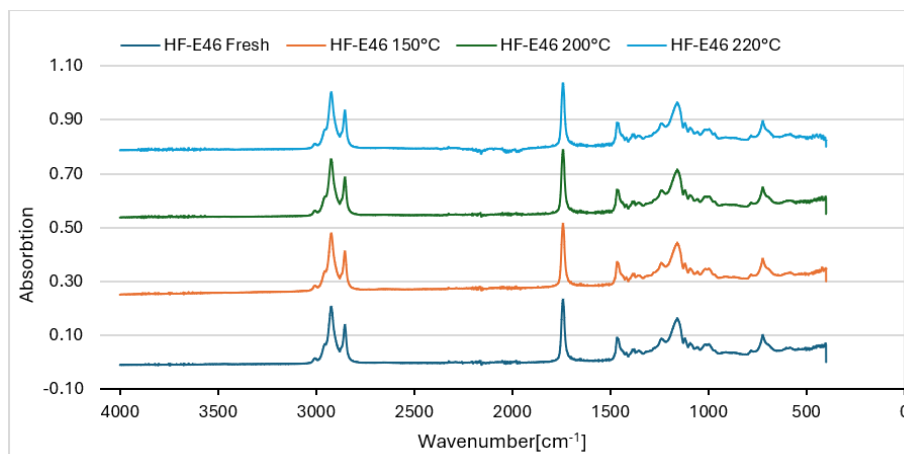
Figure 5.5, Figure 5.11, Figure 5.17 shows the comparative FTIR spectrum between fresh and thermally degraded hydraulic oil.



**Figure 5.5 Comparative FTIR spectrum of H46 mineral hydraulic oil**



**Figure 5.11 Comparative FTIR spectrum of HETG46 biodegradable hydraulic oil**



**Figure 5.17 Comparative FTIR spectrum of HF-E46 biodegradable hydraulic oil**

The main purpose of this analysis was to identify structural changes in fresh and thermally degraded hydraulic oils.

Since the FTIR analysis spectra of mineral and biodegradable hydraulic oils (fresh and thermally degraded) are essentially the same, we can conclude that a new type of bonding does not occur.

This is achieved by the following factors:

- the tested samples are designed to be resistant to high temperatures;
- the period of time in which thermal degradation was achieved was not long enough for structural changes to occur;
- The chemical stability of hydraulic oil at temperature is ensured by the presence of additives (antioxidants, anti-corrosion agents, defoamers, etc.), which reduce the speed of chemical degradation reactions. At very high temperatures (during the test, up to 200–220°C), biodegradable oils show an increase in viscosity, a phenomenon attributed to the formation of compounds identified by the absorption bands (tips) highlighted in the FTIR spectra of the oils tested at different temperatures. To be sure of this, further investigations should be carried out, since the FTIR analysis will identify the types of bonds and not the compounds in the oil, and this cannot be used as proof that a compound or mixture has not undergone any transformation, but only that no new types of bonds have emerged.

In Chapter 6 entitled *"Study of the operation of the hydraulically operated stand by experimental testing"*, the experimental hydraulically operated stand was carried out, in collaboration with the Research Institute for Hydraulics and Pneumatics (INOE 2000-IHP).

The purpose of the hydraulically operated system is to determine the functional characteristics, respectively the variation of the hydraulic parameters as a function of time.

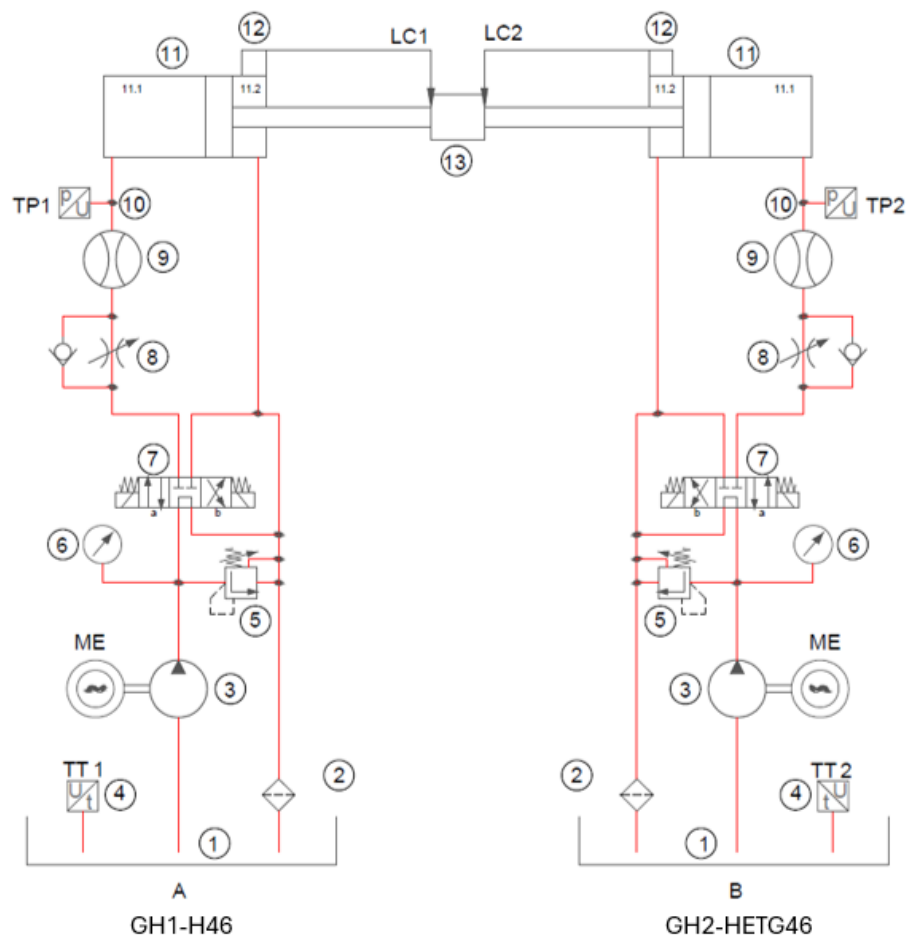
The fundamental hydraulic parameters that characterize the operation of a hydraulic system are the momentum, speed, flow rate and pressure difference between the inlet and outlet connections, which directly influence the efficiency and performance of the hydraulic system [124].

The experimental stand consists of two identical hydraulic drive systems, connected by a connecting element: one system works with mineral hydraulic oil, and the other with biodegradable hydraulic oil. At the same time, thanks to the construction, the possibility of

contamination with another type of oil is eliminated when the installation is not properly cleaned.

The innovation of the stand lies in the fact that the two systems work in interdependence, as follows: the cylinder on the left side works alternately as a motor and as a load, and the cylinder on the right side becoming a load and then a motor, this alternation being possible thanks to the 4/3 distributor of each system.

Figure 6.1 shows the operating diagram of the hydraulic installation, and Figure 6.2 and Figure 6.3 show the hydraulic system in the laboratory together with the electrical panel, Figure 6.5. The oils proposed for the commissioning of the stand were the mineral hydraulic oil H46 and the biodegradable hydraulic oil HETG46.



*Figure 6.1 Hydraulic installation operating diagram*



*Figure Error! No text of specified style in document..5 Prototype stand-1*



*Figure Error! No text of specified style in document..6 Prototype stand experimental-2*





***Figure Error! No text of specified style in document..7 Electrical panel with energy meters***

Figure 6.6 shows the schematic of the data acquisition system.

A data acquisition board is used to record experimental data. The transducers for the parameters of interest (pressures, flows, temperatures, power, number of cycles), placed in the installation, are connected to the data acquisition board that is connected to a computer via USB port. The number of cycles of the hydraulic cylinders is counted by the LabView application through a digital input of the data acquisition board, which receives signal via a relay contact (NC1) in the controller module.

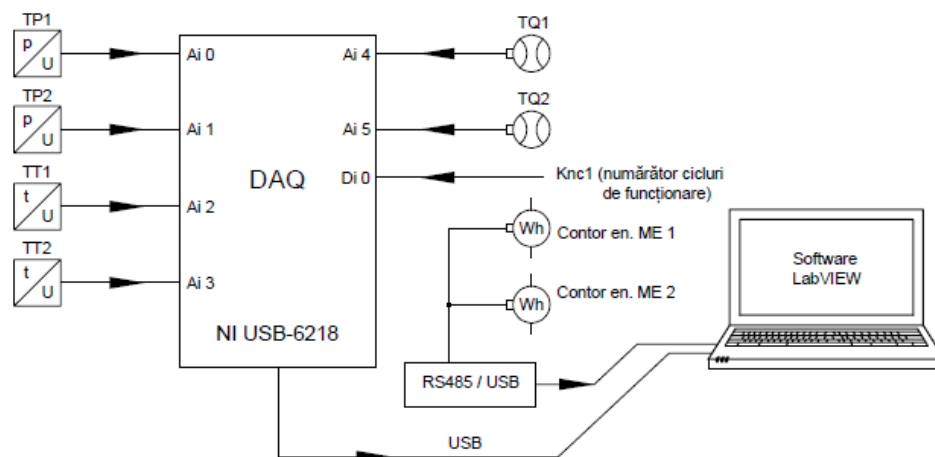


Figure 6.6 Data acquisition system schematic

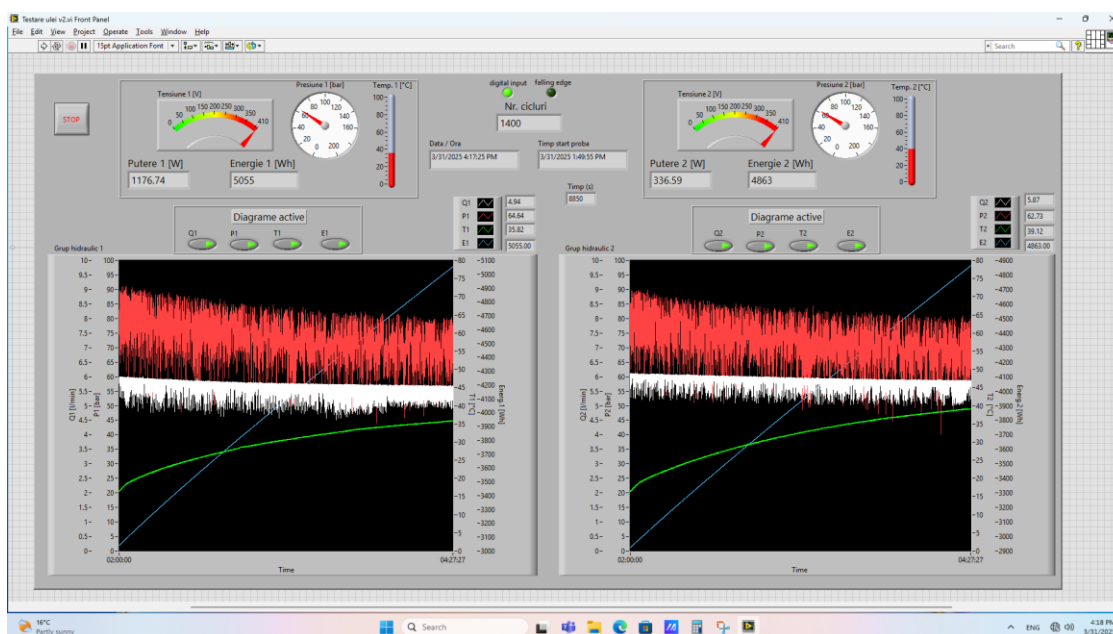


Figure Error! No text of specified style in document..8 The results of the parameters of interest (pressure, temperature, flow, and electricity) record in the LabVIEW app

Table 6.2 shows the hydraulic parameters resulting from an operating cycle of the hydraulic cylinders on the experimental stand. The values shown represent the averages obtained over the entire operating cycle.

Table 6.2 Hydraulic parameters obtained on the experimental stand

| Hydraulic parameters | Unit of measurement | Stand experimental |        |
|----------------------|---------------------|--------------------|--------|
|                      |                     | H46                | HETG46 |



|                |         |        |        |
|----------------|---------|--------|--------|
| Pressure       | [bar]   | 74,25  | 72,89  |
| Debit          | [l/min] | 5,60   | 5,82   |
| Power consumed | [W]     | 819,05 | 789,17 |
| Electricity    | [Wh]    | 4080   | 3917   |
| Temperature    | [°C]    | 29,46  | 31,25  |

In Chapter 7 entitled *"Simulation of the experimental stand in the Simcenter Amesim program"*, the Simcenter Amesim program used for the simulation of the experimental stand was presented, to obtain the same hydraulic parameters obtained on the experimental stand in the laboratory (pressure and flow).

Table 7.2 summarizes the results of the hydraulic parameters obtained from the Simcenter Amesim simulation program. Through these results, the volume efficiency at the pump and the total efficiency of the hydraulic systems were determined.

*Table 7.2 Hydraulic parameters obtained in the simulation*

| Hydraulic parameters                     | Unit of<br>measurement | Simulate Amesim |       |
|------------------------------------------|------------------------|-----------------|-------|
|                                          |                        | H46             | IT46  |
| Pressure                                 | [bar]                  | 79,49           | 79,52 |
| Debit                                    | [l/min]                | 6,45            | 6,45  |
| Torsion Moment                           | [Nm]                   | 5,47            | 5,47  |
| Hydro-mechanical efficiency (0,9 ÷ 0,95) | -                      | 0,92            |       |

The results obtained on the experimental stand (subchapter 6.4), where hydraulic parameters (pressure, flow, temperatures, useful power, power consumed, efficiency of each pumping group) were studied, are to be compared with the results obtained from the numerical simulation (subchapter 7.2).

Starting from hydraulic parameters highlighted in **Error! Reference source not found.** and **Error! Reference source not found.**, comparative graphs were made in which the difference between the results obtained on the experimental stand and those in the simulation are highlighted.

In **Error! Reference source not found.** The difference between the pressures obtained on the experimental stand in the laboratory and those resulting from the simulation is highlighted.

It is observed that the pressure obtained from the simulation of the experimental stand is higher than that resulting from the experimental stand for both mineral and biodegradable hydraulic oil, because in the simulation the pressure drops are neglected, the components in the installation have an ideal behavior, the pipes that connect with the component elements do not show imperfections. On the experimental stand, these factors are present, which further led to pressure losses.

At the same time, in the simulation, the temperature of the oils is constant, compared to the experimental stand, where it increases, according to the **Error! Reference source not found.**, and results in a decrease in viscosity that influences the behavior of the fluid.

Also, the difference between the pressures of the two hydraulic oils obtained on the experimental stand is given by the properties of the oils presented in Table 3.1, more precisely by the kinematic viscosity. The lower it is, hydraulic losses begin to occur, which leads to a decrease in pressure. This observation also applies to simulation.

### Final conclusions

The study developed within this doctoral thesis, having as main topic "*Contributions regarding the analysis of mineral and biodegradable hydraulic fluids for industrial systems*" resulted in the following aspects:

- *The current state of experimental research on mineral and biodegradable hydraulic lubricants* shows important progress in evaluating their performance. The results obtained from the research highlight the possibility in certain situations of replacing mineral hydraulic oils with biodegradable ones. Biodegradable oils are of interest for applications in the hydraulic industry due to their good physicochemical properties and maintaining energy efficiency compared to classic hydraulic systems. Vegetable oils are considered renewable resources and are characterized by an accelerated rate of biodegradation in the natural environment.
- *The rheological behavior of the three types of hydraulic oils (H46, HETG46, HF-E46) in fresh and thermally degraded condition.* Regarding the rheological study, two rheological models were proposed (the Newtonian model and the power law) by means of which the rheological parameters were determined, namely: consistency index, viscosity and flow index. Through these parameters, it was found that the Newtonian model best approximates the experimental data. The influence of temperature on viscosity was also determined by

using the Reynolds model, which is an exponential model and which, because of its application, resulted in a decrease in viscosity with the shear rate.

- *The analysis by infrared spectroscopy with Fourier transformation* of fresh and thermally degraded hydraulic oils highlighted the fact that between the two variants studied there are no significant differences in terms of the results obtained. The purpose of this analysis was to observe whether new chemical bonds were created because of thermal degradation. These results were due to chemical stability, ensured by the presence of additives in the oil.
- *Realization of the experimental stand* whose main purpose was to determine the functional characteristics for each hydraulic drive system, depending on the type of oil it uses. The results obtained after 1400 cycles (strokes) of operation of hydraulic cylinders, confirm that biodegradable hydraulic oils can be a substitute for mineral hydraulic oils, due to the performance obtained. Thus, the use of HETG46 biodegradable oil has led to an increase in the efficiency of the entire pumping group by 5.9%, an increase in useful power by 2% and a reduction in electricity consumption by 4%. The results show the good energy efficiency of biodegradable hydraulic oil, which is why it can adequately replace classic mineral oil in hydraulic drive systems. The performance of the HETG46 biodegradable oil has also been confirmed through numerical simulation carried out in the Simcenter Amesim program.

### **Personal contributions**

As the purpose of this research was to evaluate the compatibility and efficiency of using a biodegradable oil as an alternative to mineral oil, the following personal contributions were made:

- Going through studies in the literature on mineral and biodegradable hydraulic oils (vegetable oils);
- Study of the performance of mineral and biodegradable hydraulic oils in industrial applications, in accordance with their impact on components and the environment;
- Development of a phased work methodology for the evaluation of the rheological behavior of the three types of hydraulic oils (H46, HETG46, HF-E46) in fresh and thermally degraded state;

- Elaboration of the test program necessary for the operation of the Brookfield CAP2000+ test equipment;
- Making measurements, processing, representation and graphic interpretation of the results necessary for the rheological study within the Department of Machine Elements within POLITEHNICA Bucharest;
- Establishing the temperature regimes for the thermal degradation of oils according to the flash point and carrying out the degradation procedure;
- Realization, processing, representation and graphic interpretation of the results of the FTIR analysis in collaboration with the Center for Advanced Research for Innovative Materials, Products and Processes, POLITEHNICA Bucharest;
- Creation of an experimental stand, consisting of two pumping groups, in collaboration with the Research Institute for Hydraulics and Pneumatics (INOE 2000-IHP) for testing two hydraulic oils (H46 and HETG46);
- Performing experimental tests, processing, representation and graphical interpretation of the results necessary for determining the volume efficiency of the pump and the entire hydraulic group;
- Conducting a simulation through the Simcenter Amesim program, to validate the results obtained on the experimental stand;
- Dissemination of the results obtained in the research by publishing and presenting the results in scientific articles in specialized journals and at international conferences, the list of which is present at the end of this paper.

### **Perspectives of the work**

Based on the results obtained, the author aims to continue the research on these oils in the following directions:

- Study of the influence of biodegradable oils on hydraulic equipment;
- Evaluation of tribological behavior by measuring and analyzing parameters such as coefficient of friction, diameter of the wear trace, its wear rate and roughness parameters;
- Rheological study of other biodegradable hydraulic oils and their comparison with those studied in this thesis.

## Bibliography

- [8] *Bart J. C. J., Gucciardi E., and Cavallaro S.*, “Lubricants: properties and characteristics,” in *Biolubricants*, Elsevier, 2013, pp. 24–73.
- [21] *Bart J. C. J., Gucciardi E., and Cavallaro S.*Eds., *Biolubricants: science and technology*. Cambridge, UK ; Philadelphia, PA: Woodhead Publishing, 2013.
- [26] *Cecilia J. A., Silver Crossbowmen D., Alves Saboya R. M., Tavares De Luna F. M., Cavalcante C. L., and Rodríguez-Castellón E.*, “An Overview of the Biolubricant Production Process: Challenges and Future Perspectives,” *Processes*, **vol. 8**, no. 3, p. 257, Feb. 2020.
- [27] *Berman D.*, “Plant-Based Oils for Sustainable Lubrication Solutions—Review,” *Lubricants*, **vol. 12**, no. 9, p. 300, Aug. 2024.
- [31] *Maidan R., Tkáč Z., Abrahám R., Stančík B., Kureková M., and Paulenka R.*, “Effect of Ecological Oils on the Quality of Materials of Hydraulic Pump Components,” *AMR*, **vol. 801**, pp. 1–6, Sept. 2013.
- [35] *Deuster S. and Schmitz K.*, “Bio-Based Hydraulic Fluids and the Influence of Hydraulic Oil Viscosity on the Efficiency of Mobile Machinery,” *Sustainability*, **vol. 13**, no. 14, p. 7570, July 2021
- [74] *Fly I.*, *Behavior of lubricating oils under pressure*. Bucharest: Didactic and Pedagogical Publishing House, 2004.
- [78] *Tudose R. Z., Volintiru T., and Lungu M.*, *Rheology of macromolecular compounds*. Technical Publishing House., 1982.
- [90] *Bhattad A.*, “Review on viscosity measurement: devices, methods and models,” *J Therm Anal Calorim*, **vol. 148**, no. 14, pp. 6527–6543, July 2023.
- [91] *Brookfield Engineering Laboratories, Inc.*, “BROOKFIELD CAP 2000+, Viscometer Operating Instructions.”
- [92] *Wan Nik W. B., Zulkifli F., Lam S. S., Rahman M. M., Yusof A. A., and Rahman M. M.*, “Experimental Studies on the Rheological and Hydraulic Performance of Palm Based Hydraulic Fluid,” *Jurnal Teknologi*, **vol. 66**, no. 3, Feb. 2014
- [95] *Industrial Cars*, “H46 Hydraulic Oil Datasheet,” Technical Data Sheet.
- [96] *Kajo Lubricants*, “Fișa tehnică a uleiului hidraulic HETG46,” Technical Data Sheet.
- [97] *Shell*, “Fișa tehnică a uleiului hidraulic HF-E46,” Technical Data Sheet.
- [103] *Olszak A., Osowski K., Musiałek I., Rogoś E., Kęsy A., and Kęsy Z.*, “Application of Plant Oils as Ecologically Friendly Hydraulic Fluids,” *Applied Sciences*, **vol. 10**, no. 24, p. 9086, Dec. 2020.
- [104] *Petlyuk A. M. and Adams R. J.*, “Oxidation Stability and Tribological Behavior of Vegetable Oil Hydraulic Fluids,” *Tribology Transactions*, **vol. 47**, no. 2, pp. 182–187, Apr. 2004.
- [105] *Uppar R., Dinesha P., and Kumar S.*, “A critical review on vegetable oil-based bio-lubricants: preparation, characterization, and challenges,” *Environ Dev Sustain*, **vol. 25**, no. 9, pp. 9011–9046, Sept. 2023
- [106] *Malkin A. Ya. and Khadzhiev S. N.*, “On the rheology of oil (Review),” *Pet. Chem.*, **vol. 56**, no. 7, pp. 541–551, July 2016.
- [120] *Thermo Scientific*, “Broșură Nicolet iS50 FT-IR Spectrometer.” Thermo Fisher Scientific.
- [124] *Vasiliu N. and Vasiliu D.*, *Hydraulic and pneumatic drives*, **vol. 1**. Bucharest, Romania: Technical Publishing House, 2005.