POLYTECHNIC UNIVERSITY of BUCHAREST

SUMMARY

THESIS

REDUCTION OF FABRICATION COST IN THE MATERIAL INDUSTRY FROM REGENERATION OF INDUSTRIAL USED OILS

Thesis supervisor: Prof. Dr. Ing. Nicolae PANAIT

Author: Drd. Ing. Simion Marian STRATESCU

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Foreword

The justification for choosing the doctoral thesis is given by the need to solve a particularly important problem for the oil products regeneration industry, namely: reuse of oil and grease products so that their loss is as small as possible and reduce the costs of beneficiaries by regeneration of at least 3 times the oils that use them both for cooling the machine and for thermochemical treatments by managing them as efficiently as possible so that neutralization is the last resort.

The proposed theme of the doctoral thesis was the design and realization of a new technology that allows the reuse of the products listed above and in case of neutralization even if they are of several types (hydraulic oil H46, transformer oil type T30, mineral oil, etc.) and are all stored in a single container) their transformation into general purpose Vaseline with a high degree of use.

The aim of the doctoral thesis was to reduce manufacturing costs in the materials industry by regenerating used industrial oils.

To achieve this goal it was necessary to perform the following steps:

- *analysis of the literature on the current state in the country and abroad of the situation of used oil that goes to neutralization or is discharged in various prohibited places given that this product is very toxic to water, soil and air.*
- *experimental research on the possibilities of regeneration at different third companies listed in chapter 3 where the results were very good, with certifications from third parties.*
- *determining the number of possible regeneration cycles for these products.*
- *experimental calculations regarding the viscosity of the analyzed oils.*
- *research on the achievement of technological flows in order to have the highest possible yield.*

The doctoral thesis has its methodological and theoretical-scientific support in the bibliographic study, both regarding the current stage of industrial oils regeneration and for obtaining general purpose greases with a high degree of applicability..

I would like to thank the scientific director of the paper, Prof. Dr. Eng. Nicolae PANAIT for all the support given to the completion of the research plan and all the indications he offered me to complete the research and write the doctoral thesis, I thank the members of the Guidance Commission, professors Mihai BUZATU, Valeriu Gabriel GHICA, Nicolae CONSTANTIN and AUGUSTIN SEMENESCU who offered me their competent support and advice for the elaboration and completion of the thesis.

I thank the teachers from the IMOMM department at the Polytechnic University of Bucharest, for helping me with recommendations in all the activity I have carried out so far.

Keywords:

Smoothness: is the property of the oil to adhere to the surfaces it comes in contact with.
The smoothness means that an oil film remains on the surfaces of the parts in contact, so that at the beginning it has a minimum of lubrication.

Flash point: is defined as the minimum temperature mark at which the mixture of oil and vapor becomes flammable.

Lubrication point: the lubrication point is defined as the minimum temperature mark at which the oil continues to flow**.**

Ignition temperature: the temperature at which the mixture of water, oil, air can be ignited with a mixture of heat source.

Oil density: mass of moisture in the volume.

Burning point: is 40-50 ºC above the flash point.

Freezing point: represents the most volatile temperature at which the oil stops flowing**.**

C O N T E N T

CHAPTER 1. EXPERIMENTAL RESEARCH IN ORDER TO REGENERATE WASTE INDUSTRIAL OILS AND REINTRODUCE THEM INTO TECHNOLOGICAL FLOW

The rationale for choosing this theme is given by the need to solve a particularly important problem for the petroleum regeneration industry. In this sense, the theme of the article is to develop a new technology to reuse oil and grease products so that their loss is as small as possible and in parallel to significantly reduce the costs of beneficiaries by regenerating at least 3 times the oils used both for cooling the machine and for thermochemical treatments by managing them as efficiently as possible so that neutralization is the last solution, but also their transformation into general purpose Vaseline with a high degree of use. This technology applies to different types of industrial oils from, for example, H46 hydraulic oil, T30 type transformer oil to various other types of mineral oils, all of which are stored in a single container.

The purpose of this part of the doctoral thesis is to research the possibility of obtaining a very high yield after the use and regeneration of industrial oil..

1.1. Introduction

The most commonly used liquid lubricants are oils. Lubricating oils consist mainly of base oil and additives. The base oils are mineral, vegetable, animal and synthetic. [1,2,3]

A classification of oils is given in Figure 1. Lubricants are characterized by a number of physico-chemical and functional properties, of which the main ones are:

- viscosity;
- smoothness: it is the property of the oil to adhere to the surfaces it comes in contact with; this causes an oil film to remain on the surfaces of the parts in contact, so that at start-up it has a minimum of lubrication;
- the density of the oil, which defines the mass of moisture in the volume;
- oxidation stability;
- flash point: is defined as the minimum temperature mark at which the oil - vapor mixture becomes flammable;
- burning point: it is 40-50 ºC above the flash point;
- freezing point: represents the most volatile temperature at which the oil stops flowing.

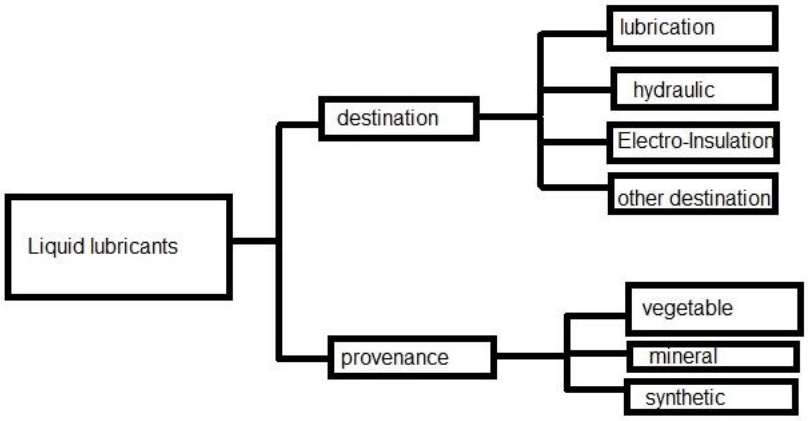


Figure 1. Clasification of liquid lubricants

Soil, water, flora and fauna suffer from waste oils that are thrown away at random. The oil that reaches the soil significantly reduces its productivity and, moreover, prevents it from regenerating in order to be fruitful later.

Underwater life is also endangered if it comes in contact with used oils and used greases, as they form a film on the surface of the water that blocks the absorption of oxygen and the penetration of sunlight. [4.5]

Used oils and used greases are not biodegradable, so they do not disappear, moreover, in contact with other elements they increase their volume and contaminate both water and soil. [6].
Used oils and greases are also harmful to animals or plants. In the case of animals, they have effects similar to those they have on the human body, namely they cause irritation or may even contribute to the appearance of diseases. On the other hand, vegetation also has problems surviving in contact with used oils and greases. [7.8]

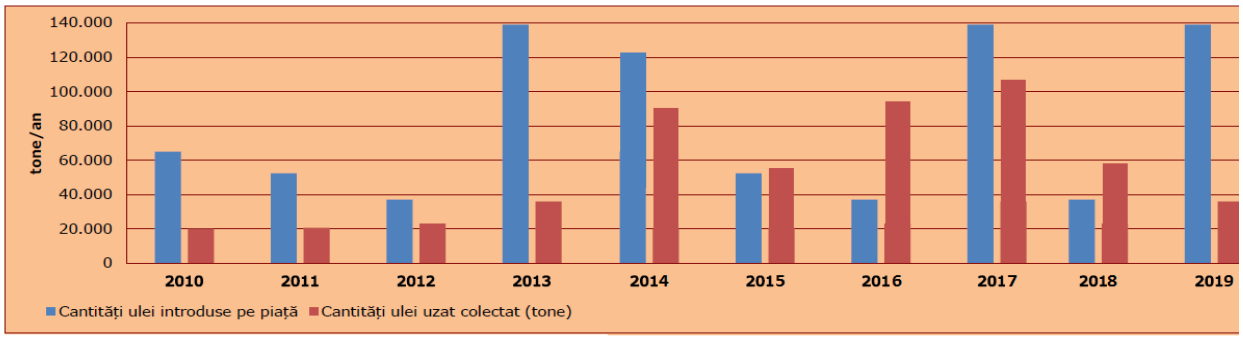
Used industrial oil is an important resource used especially for the manufacture of biofuels, being a viable alternative to specially cultivated plants and occupying large agricultural areas. From a liter of used industrial oil you can get 900 milliliters of biodiesel, and the by-product produces a natural soap, good for the human body.

Industrial grease and oil waste not only harms the environment and man, but also deposits them on the walls of the sewer system causes regular clogging, bad smelling effects, faster corrosion and have a negative influence on the operation of sewage treatment plants and leads to blockage and their destruction.

The frequency of cleaning and maintenance of sewage treatment plants, pumping, pipes and sewerage system increases five times compared to the normal cycle. This leads to a huge increase in community costs. The elimination of these adverse effects is possible if this waste is collected, treated, filtered and reintroduced into the economic circuit in another form. [9,10].

The analysis of the possibilities of regeneration of used industrial oils and their reintroduction in the technological flow is regulated by GD no. 235/2007.

According to GD no. 235/2007, the producers and importers of oils are obliged to ensure the organization of the waste oil management system, corresponding to the quantities and types of oils introduced on the market. [4].
This obligation may be fulfilled individually or through third parties indicated to the central public authorities for environmental protection, by the responsible persons. Thus, in the period 2010-2013, both the quantities of oils placed on the market and the quantities of waste oils generated show an uneven variation, according to Figure 2 (source: National Agency for Environmental Protection (ANPM)).



Sursa: ANPM

1.2. Experimental research

The technological equipment for regenerating waste oils is shown in figure 3, having the following constructive components:

Oil inlet: the hole through which the oil enters for recycling;

Filter 1: after entering the enclosure with the help of a pump the oil circulates through the filter which leads to its cleaning and clarification, the filter is for the removal of coarse impurities with the size of up to 10 microns - prefiltration operations; Pre-filtration collection vessel; Heater for raising the oil temperature in order to eliminate a quantity of water and to decrease the viscosity index of the oil.

Filter 2: after passing through filter 1, the oil enters the chamber 2 with the help of the pump in order to benefit from its filtration for the second time. The second filter is designed to clean the oil and collect impurities up to 3 microns and collect water particles that have passed through the heater; Enclosure for collecting oil after passing through filter 2; Heater for maintaining and raising the oil temperature.

Filter 3 : final filtration at one micron in which the impurities greater than 1 micron remove any remaining water molecules; Oil collection chamber coming out of filter 3; Regenerated oil transfer pump in the machine; Control panel: to start the filtration installation and implicitly the workflow, it is connected to the electrical installation which starts the circulation pump of the installation

Regenerated oil outlet: after the oil has been filtered using the 3 filters, it is pushed out so that the newly used oil enters the circuit for filtration and regeneration.

The flow of obtaining the regenerated industrial oil is summarized in figure 4, based on the description of the experimental installation.

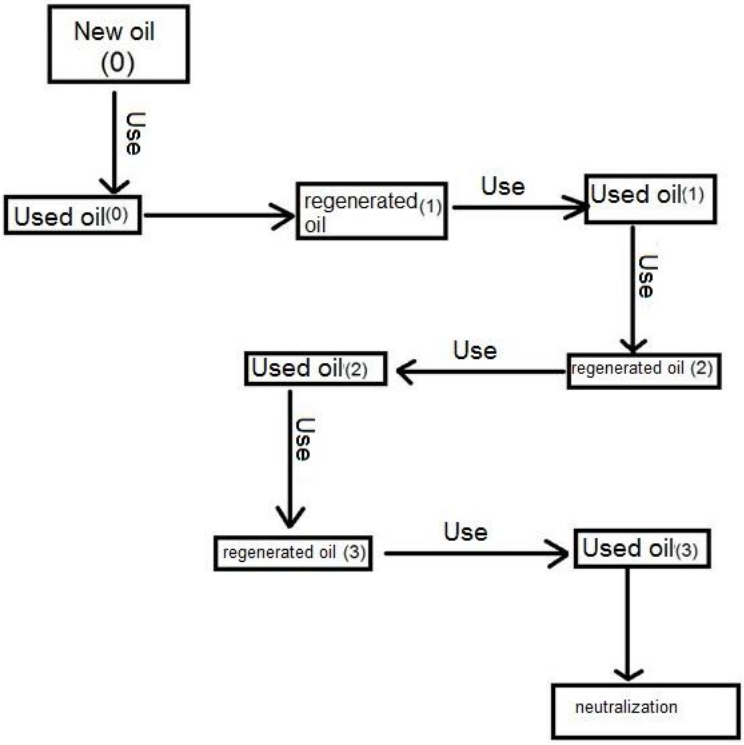


Figure 4. The implementation scheme of the used industrial oil was made at SC Alro SA

The technological solution described in figure 4, was concretely applied to one of the major economic agents in the field of steel production.

1.3. Analysis of experimental data

This analysis is composed of the microscopic analysis of the three categories of oils: new oil, purchased from suppliers, used oil and regenerated oil.1. New H46 hydraulic oil, purchased from suppliers, has the following main physico-chemical properties (Figure 5)
2. used H46 oil has the following main physico-chemical properties (figure 6). In this image you can see a microscopic sample of a hydraulic oil type H46 that had over 8000 operating hours.
It was to reach neutralization (removal of the used product).
3. regenerated H46 oil has the following main physico-chemical properties (figure 7). The microscope sample is taken from a sample of regenerated oil using the technological equipment that is the subject of this article

REPORT - OIL ANALYSIS

DATE TEST			
CLIENT			
ADDRESS			
EQUIPMENT			
TEST DATE			
OIL TYPE	HD 46		
RESULTS			
MEASUREMENT PARAMETERS	NORMA	Results	
Particles larger than 5µm / ml	(ISO 4406)	1242	-
Particles larger than 15µm / ml	(ISO 4406)	33	-
Particles larger than 30µm / ml	(ISO 4406)	13	-
ISO CODE - impurities	17/12		
Particles	Metal: 80%, Silicone : 20%.		
Viscosity at 40°C	46.8		
Viscosity at 100°C	7.5		
IV	120.6		
Water			
Condition	ALLOWED		

MICROPHOTOGRAPHY

Zoom: 350x

DIAGNOSTIC

The oil has physical properties allowed

Figure 5. New H46 oil quality certificate

In this image you can see a sample of a new oil. You can also see some impurities in the case of a new oil.

REPORT - OIL ANALYSIS

DATE TEST	
CLIENT	
ADDRESS	
EQUIPMENT	
TEST DATE	
OIL TYPE	HD 46

RESULTS

MEASUREMENT PARAMETERS	NORMA	Results	
Particles larger than 5µm / ml	(ISO 4406)	1076	-
Particles larger than 15µm / ml	(ISO 4406)	151	-
Particles larger than 30µm / ml	(ISO 4406)	48	-
ISO CODE - impurities	17/14		
Particles	Metal: 80%, Silicone : 20%.		
Viscosity at 40°C	46.9		
Viscosity at 100°C	7.5		
IV	120.3		
Water			
Condition	Alarm		

MICROPHOTOGRAPHY

Zoom: 350x

DIAGNOSTIC

The oil has physical properties in alarm values due to the quantity of particles

Figure 6. H46 old oil quality certificate

As noted, in the case of used oil, the amount of medium (15 µm) and large (30 µm) impurities is significantly higher than the new oil.

Regenerated oil

REPORT - OIL ANALYSIS

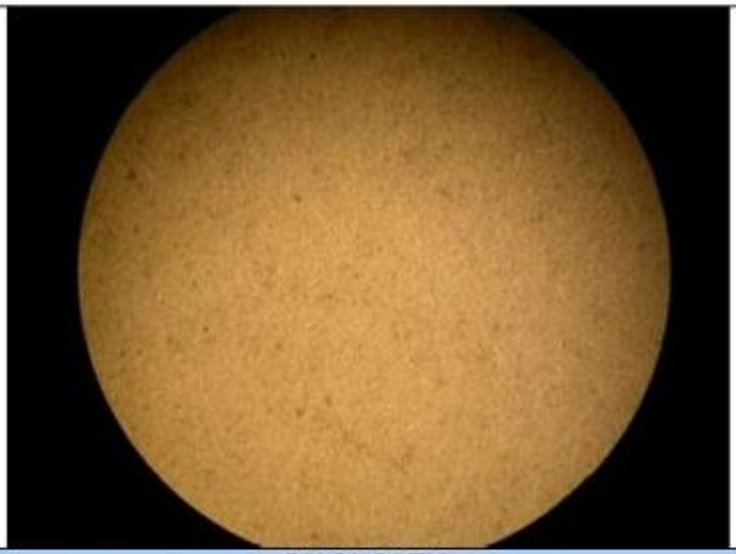
DATE TEST	
CLIENT	
ADDRESS	
EQUIPMENT	
TEST DATE	
OIL TYPE	

REZULTATE

MEASUREMENT PARAMETERS	NORMA	Results	
Particles larger than 5µm / ml	(ISO 4406)	206	-
Particles larger than 15µm / ml	(ISO 4406)	23	-
Particles larger than 30µm / ml	(ISO 4406)	0	-
ISO CODE - impurities	15/12		
Particles	Metal: 50%, Silicone : 45%, Oxide : 5%.		
Viscosity at 40°C	46.3		
Viscosity at 100°C	7.4		
IV	119.5		
Water			
Condition	ALLOWED		

MICROPHOTOGRAPHY

Zoom: 360x



DIAGNOSTIC

The oil has physical properties allowed

Figure 7. H46 regenerated oil quality certificate

In the image above you can see the microscopic examination of an oil sample after regeneration. All physical and chemical properties are the same as new unused oil.

Analysis of the profitability of the presented technology

The calculation of the economic efficiency for the regeneration of the hydraulic oil, with a case study for Tenaris Calarași led to the following result:

The price of a liter of new oil is 6 lei

The price of a liter of H46 regenerated oil is 2.5 lei

New oil price for purchase

15000 x 6 = 90000 lei

Cost of regenerated oil

15000 x 2.5 = 37500lei

The savings made for the 15,000 liters of regenerated oil

90000 - 37500 = 52500 lei

This oil can be regenerated 3 times.

Thus: If the 15000 liters of H46 oil were regenerated 3 times, the price would be

37500 X 3 = 112500 lei

If it did not regenerate it would be 15000 x 6 x 3 = 270000 lei

The achieved economy would be:

270000 - 112500 = 157500 lei

Also these 15000 liters of oil resulted after the 3rd regeneration, which could be turned into grease:

The price of a kg of grease bought from the market is 4.60 euros (1 euro = 4.8 lei)

4.6 Euro / kg x 4.8 RON / Euro = 22.08 lei / kg. new grease

The same type of grease with a drip point of 140oC made from waste oil that can no longer be regenerated

1 kg of used oil grease = 9 lei

Economy achieved at 1 kg. of grease 22.08 - 9 = 13.08 lei

13.08 x 15000 = 196200 lei

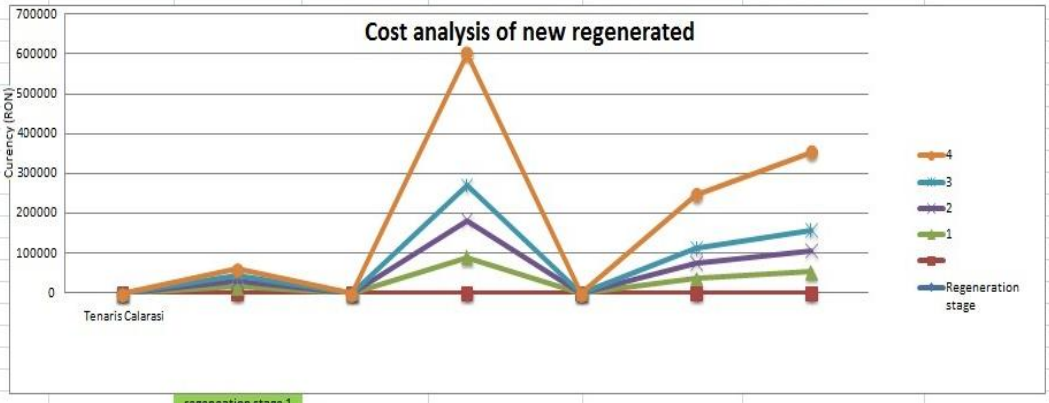
The economic efficiency registered at the Electric Steelworks where 15000 liters of H46 oil were regenerated, oil that had to be neutralized, given to the collector and replaced with new oil and according to those presented in figure 8, a total saving of 353700 lei was achieved composed of:

157500 lei from oil regeneration;

196,200 lei from the transformation of used oil into lubrication.

Tenaris Calarasi

Regeneration stage	Nomenclature	Quantity (liters)	Price of new oil (RON)	Total price new oil (RON)	Regenerated oil price (RON)	Total price regenerated oil (RON)	Difference between new oil and used oil (RON)
	Hydraulic Oil (H46)	1	6	6	2.5	2.5	3.5
1	Hydraulic Oil (H46)	15000	6	90000	2.5	37500	52500
2	Hydraulic Oil (H46)	15000	6	90000	2.5	37500	52500
3	Hydraulic Oil (H46)	15000	6	90000	2.5	37500	52500
4	Neutralization / transformation into greases	15000	22.08	331200	9	135000	196200
						Total difference used new oil, regenerated oil + transformation in grease (RON)	353700



regeneation stage 1
regeneration stage 2
regeneration stage 3

Fig.8 - Tenaris Calarasi economic efficiency diagram

1.4. Conclusions on Chapter 1:

In the above, you can see the efficiency of the applicability of the proposed solution from an economic point of view as well as from the point of view of the qualitative yield it can have.

Both the economic calculations and the experimental determinations were presented in order to see the efficiency of the regeneration installation.

It can be seen that the degree of purity of an oil is as follows:

-Used oil: according to the picture taken with the microscope you can see the level of impurities and after the laboratory tests you can see that this product can only be used and must be neutralized

-Regenerated oil: according to the picture taken with the help of the microscope it can be seen that there are no impurities and from a technical point of view the product can be used in optimal conditions

-New oil: according to the picture taken with the help of the microscope it can be seen that there are impurities with a low level for an unused product, so the more the problem of regeneration arises.

As a conclusion it can be said that after using the used oil regeneration plant it can be seen that as a result of this process, the oil is of a quality (maybe) better than a new type of oil.

From the above, we managed to implement the integrated management of industrial lubricants through regeneration.

So any industrial processing unit that has in the technological flow industrial oils, greases or industrial emulsions can reduce its costs by regeneration thus protecting nature, practically

applying the concepts of Circular Economy, which involves sharing, reusing, repairing, renovating and recycling existing materials and products. as much as possible

Through this innovative concept of integrated management of industrial lubricants it is possible to achieve a long-term protection of the environment, saving resources, economic efficiency.

Chapter 2. ANALYSIS OF THE POSSIBILITIES OF TRANSFORMING WASTE INDUSTRIAL OILS INTO GENERAL GRESES

The theme of the research presented in this part of the paper aims to solve a particularly important problem for the oil products regeneration industry, namely: reuse of oil and grease products so that their loss is as small as possible while reducing the costs of beneficiaries by regenerating at least three or oils used both for cooling the machine and for thermochemical treatments. In this way the neutralization of the mentioned petroleum products must be considered as a last resort.
The paper has its methodological and theoretical-scientific support in the literature, both on the current stage of industrial oil regeneration and for obtaining general purpose greases with a high degree of applicability.

2.1.Introducere

Oils used in machinery during the production process end up being contaminated with metallic and non-metallic inclusions.

In these mineral oils are found soaps of light metal soaps called greases or oily liquids. The light metals whose soaps are used to thicken oils or oily liquids are sodium (Na), calcium (Ca), aluminum (Al), barium (Ba), lithium (Li), lead (Pb), etc.

Greases are part of the category of plastic or quasi-plastic fluid media - non-Newtonian.

Greases are mainly used for lubricating low-speed friction torques. The use of greases is limited by the operating temperature of the friction coupling which must be lower than the drip point by 15-25 ° C.

Greases are used not only for lubrication but also for sealing and protection of metal surfaces, reducing the effects of vibration, shock or sudden changes in speed.
The composition and structure of greases determine their general properties, among which we can mention:
- viscosity of the base oil;
- thermal stability;
- penetration;
- ash content,
- drip point: represents the temperature at which the first drop of heated grease falls under certain conditions; this point represents the temperature of the transition of the grease from the semi-solid state to the liquid state
- flash point: the minimum temperature at which the oil vapor mixture becomes flammable
- lubrication point: the minimum temperature at which Vaseline continues to flow

A classification according to the fields of use is presented schematically in the figure below, where in parentheses is given their symbolization according to Romanian standards.

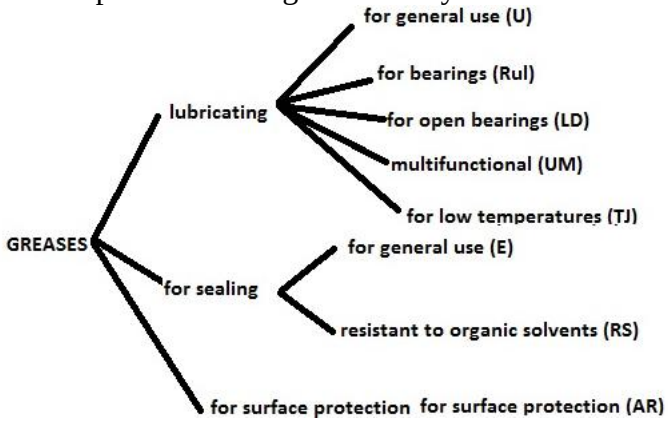


Figure 9: Classification of greases

The notation of greases contains next to the word "Grease" the symbol of the field of use, the drip point, the base of the soap, the consistency, through the symbol (digits) of penetration, in tenths of a mm, of a cone for five seconds in the grease mass at 25 ° C and additives.
Here are some examples of grease scoring.
- Grease Rul 165 Na 4 ⇔ lubricating grease for bearings, with drip point min. 165 ° C, based on sodium soap, consistency 4;
- Grease LD 170 Na 7 ⇔ lubricating grease for open bearings, with drip point min. 170 ° C, based on sodium soap, consistency 7
- Grease LD 170 Na 7 ⇔ lubricating grease for open bearings, with drip point min. 170 ° C, based on sodium soap, consistency 7
- Grease UM 185 Li 2 EP ⇔ multifunctional lubricating grease, with drip point min. 185 ° C, based on lithium soap, with consistency 2 added for extreme pressure.
The classification of oils by consistency, according to NLGI (National Lubricating Grease Institute) is shown in Figure 10.

Class – NLGI	Penetration at 25˚C, mm/10	Structure
000	445 to 475	Fluid
00	400 to 430	Semi-fluid
0	355 to 385	Extremely soft
1	310 to 340	Very soft
2	165 to 395	Soft, ductile
3	220 to 250	Medium
4	175 to 205	Hard
5	130 to 160	Very hard
6	85 to 115	Extremy hard
7	Sub 70	
NGLI class according to DIN 51 818 penetration according to DIN 51 804 part 1		

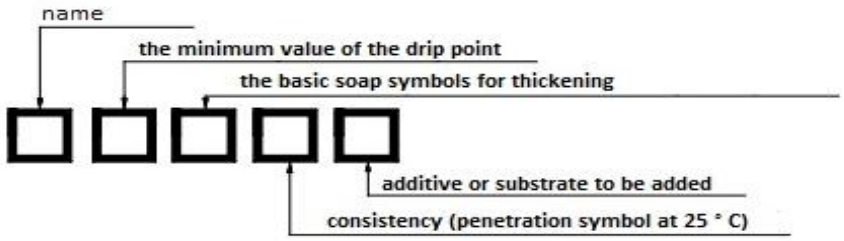


Figure 10: Classification of greases by consistency

Sodium-based greases are water-hungry, and are recommended not to be used in humid environments, and lithium and calcium-based greases repel water and have the potential to work in high temperatures. It is necessary not to exceed the limit temperature of use of the grease because it oxidizes and with it its durability is greatly reduced (an excess of 100 C reduces the durability of the grease by about 50%).

4.2. Experimental installation and research method

Obtaining the multifunctional greases from used oils in order not to be transformed into toxic waste is performed with the help of a technological process of transforming them into industrial greases with multiple applicability.

The description of the experimental installation is as follows (figure 11):

Main vacuum emulsifying machine, with the following components:

- Capacity: 5L-100L
- Customized service: to offer and design total solution according to URS (User Requirement Specification) customer specifications
- Slow mixer 0-40 rpm with PTFE scraper
- Homogenizer 0-3500rpm (rotor and stator can adjust the space)
- Heating with steam or electricity
- Pressure: chamber-0.093Mpa up to 0.1Mpa, 0.3Mpa jacket for steam (standard type)
- Lift the upper hydraulic cover
- Safety limiter for safe operation
- Upper cover connection: liquid supply inlet with stainless steel pipe filter, solid material inlet, vacuum inlet, compressed air inlet, vacuum inlet, burglary, CIP cleaning head, discharge port, heavy-duty respirator to dust.
- Vacuum system, containing: water ring vacuum pump or oil vacuum pump and safety valve to protect the ring vacuum pump
- Hydraulic system
- Control system

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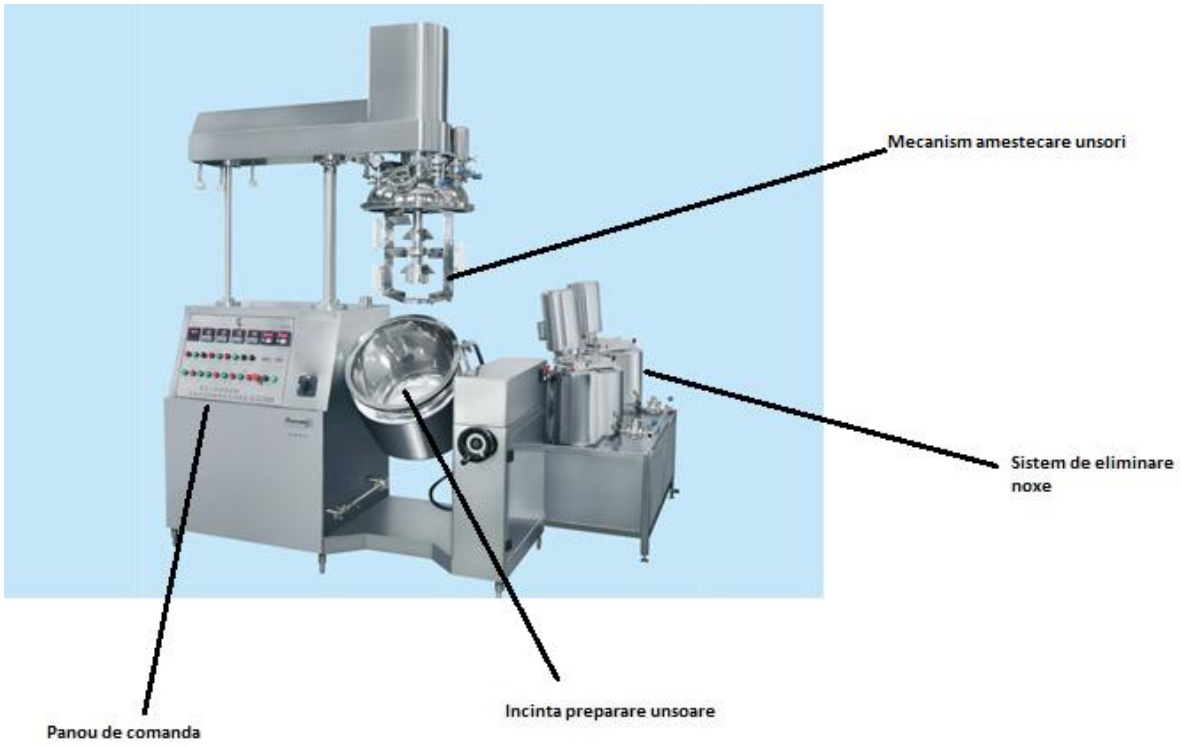

Figure 11 Experimental installation for obtaining greases from waste oils

The technology for transforming used oils into multifunctional greases is the subject of the application for the OSIM Registered Patent published in BOPI no. 2/2019 with no. A201700593133091A2.

This technology follows the principles of the Paris Conference of December 2015 where the foundations of the circular economy were laid, an economy that consists in transforming a waste into raw material for a new product similar with similar or different properties, in order to save the planet's resources by capitalizing on waste. oil and reducing pollution.

The technical problem solved by this technology, which is the object of the aforementioned invention, consists in the transformation of a used oil (hazardous waste) into a new product with different properties: multifunctional grease.

In this sense, in order to confirm the validity and industrial applicability of the process presented in this chapter, some practical examples are presented below.

Option 1:

An amount of oil is taken from the collection and filtered through a cascade filtration system of metallic and non-metallic impurities and also the water is removed, the installation being provided with a heater for water vaporization and oil fluidization. After this process of filtering the impurities and removing the water, the initial oil from the collection was found as purity in purity classes 10-12, (the oil being waste) after the process of filtering and removing the water, reaching the purity classes. 4-6 (purity standard NAS 1638 or ISSO 4406), proceed to the manufacture of grease.

In an enclosure with a volume of 40 liters with a heater, mixing system and thermocouple for measuring the temperature, take 10 liters of filtered oil in which 400 gr. Of soda ash, 600 gr. Of stearin and 1 kg of bentonite are added. . Homogenize the mixture and start the heating and mixing system. The heating is done gradually so that when it reaches the temperature of 140oC the industrial dye 0.026 kg is introduced. stirring continuously until the mixture reaches 1700 C.
Maintain at this temperature by stirring constantly. After that, turn off the heating system and mix for about 10 minutes, then turn off the mixing system.

After the mixture has cooled (after 24 hours), it is emptied, packed and sent to the beneficiary.

Option 2 :

A quantity of oil is taken from the collection and the metallic and non-metallic impurities are filtered through a cascade filtration system and also the water is removed, the installation being provided with a heater for water vaporization and oil fluidization.

After this process of filtering the metallic and non-metallic impurities and removing the water, the grease is manufactured.
Take 10 liters of oil and place in an enclosure provided with a heating, mixing and thermocouple system. Then introduce 1 kg of calcined soda, 1 kg. of stearin, 2 kg. bentonite.
Homogenize the mixture very well, start the heating and mixing installation at a temperature of 1400C, introduce 0.028kg. industrial dye, homogenize the mixture, continue slow heating until the temperature reaches 2100C. At this temperature, the mixture is kept for 10-20 minutes, after which the heating is stopped, the mixer is left for another 5-10 minutes, after which it is stopped.
After 24 hours the mixture was cooled, mixed again, packaged and shipped.

Option 3 :

Take 10 liters of oil which has been filtered through a cascade filtration system from which the water has been removed is introduced into an enclosure provided with a heating, mixing and thermocouple system. 1 kg of calcined soda, 1 kg of stearin and 2 kg of industrial talc are introduced over the 10 liters of oil. Homogenize the mixture formed and start heating.
Gradually heat to 1400 C when 0.028 kg of industrial dye is introduced, homogenize and raise the temperature to 2300 C and keep at this temperature for 10-20 minutes.
Then the installation is stopped, the mixer is left to work for 9-10 minutes, after which it is stopped and the mixture is allowed to cool for 24 hours. After cooling, mix again and pack.

Option 4 :

Take 10 liters of oil which has been filtered through a cascade filtration system from which the water has been removed, place it in a 40 liter enclosure provided with a heating, mixing and thermocouple system. 1 kg is added to the enclosure. calcined soda, 1 kg stearin and 2 kg. colloidal graphite, mix, gradually raise the temperature to 1900C maintaining this temperature for 10-20 minutes, stirring continuously, turn off the heating system, continue mixing for another 10-20 minutes, then stop.

Leave to cool for 24 hours, then mix again and pack.

Option 5 :

Take 10 liters of oil with a viscosity index of 220 to 400C which has been filtered through a cascade filtration system from which the water has been removed, place in a 40 liter enclosure fitted with a heating system, mixture and thermocouple add 0.4 kg. calcined soda, 0.7

kg.stearin and 0.8 kg.industrial talc, homogenize the mixture formed and start heating gradually, at a temperature of 1400 C, add 0.024 kg.industrial pigment.
Heat further and at a temperature of 1700C keep for 10-20 minutes, stirring constantly, turn off the heating system, continue mixing for another 10-20 minutes, then turn off. Leave to cool for 24 hours, then mix again and pack.

Option 6 :

Take 10 liters of oil that has been filtered through a cascade filtration system from which the water has been removed, put it in a 40 liter enclosure provided with a heating, mixing and thermocouple system, add 1 kg. calcined soda, 1 kg.talc, 1 kg. stearin and 0.5 kg. graphite homogenize the mixture and start the heating and mixing system.

At a temperature of 1900C it is kept for 10-20 minutes, stirring continuously, after which the heating is stopped. Continue mixing for another 5-10 minutes, then allow to cool for 24 hours, mix and pack.

2.3. Experimental results and their interpretation using laboratory analyzes

The experimental results are further summarized:

Experimental research 1:
-aspect: homogeneous;
-penetration: at 25°C after 60 mixtures (0.1 mm: 360);
-resistance to high pressures on the 4-ball machine (mm: resist);
-drip point: 153°C;
-corrosion on steel blade at 100°C for 24 hours: withstands;
-EMCOR test (grd.): 0;
-resistance to the static action of water (grd.): 1-90;

Experimental research 2:
-aspect: homogeneous;
-penetration: at 250C after 60 mixtures (0.1 mm: 195);
-resistance to high pressures on the 4-ball machine (mm: resist);
-drip point: 193°C;
-corrosion on steel blade at 100°C for 24 hours: withstands;
-EMCOR test (grd.): 0;
-resistance to the static action of water (grd.): 1-90;

Experimental research 3:
Physico-chemical laboratory results
-aspect: homogeneous;
-penetration: at 250C after 60 mixtures (0.1 mm: 180);
-resistance to high pressures on the 4-ball machine (mm: resist);
-drip point: 227ºC;
-corrosion on steel blade at 1000C for 24 hours: withstands;
-EMCOR test (grd.): 0;

-resistance to the static action of water (grd.): 1-90;

Experimental research 4 :
Physico-chemical laboratory results
-aspect: homogeneous;
-penetration: at 25°C after 60 mixtures (0.1 mm: 210);
-resistance to high pressures on the 4-ball machine (mm: resist);
-drip point: 217°C;
-corrosion on steel blade at 100ºC for 24 hours: withstands;
-EMCOR test (grd.): 0;
-resistance to the static action of water (grd.): 1-90;

Experimental research 5:
Physico-chemical laboratory results
-aspect: homogeneous;
-penetration: at 25°C after 60 mixtures (0.1 mm: 380);
-resistance to high pressures on the 4-ball machine (mm: resist);
-drip point: 144ºC;
-corrosion on steel blade at 1000C for 24 hours: withstands;
-EMCOR test (grd.): 0;
-resistance to the static action of water (grd.): 1-90;

Experimental research 6:
Physico-chemical laboratory results:
-aspect: homogeneous;
-penetration: at 25°C after 60 mixtures (0.1 mm: 190);
-resistance to high pressures on the 4-ball machine (mm: resist);
-drip point: 205°C;
-corrosion on steel blade at 100ºC for 24 hours: withstands;
-EMCOR test (grd.): 0;
-resistance to the static action of water (grd.): 1-90;

The morphological aspect of the resulting samples is presented generically in the two figures below:

Figure 12 Experimentally made grease from waste oils (variant 1)

Figure 13. Grease experimentally made from used oils (variant 4)

As can be seen in experimental research 1 and 4 the only thing that differs between the 2 types of practical realization is the drip point and their penetration dating back to the dosage of additives.

2.4. Conclusions in the chapter 2:

Following the use of an oil 4 times (once new and three times regenerated) it was established that the oil can no longer be regenerated, and will be transformed into general grease.
This procedure will help the economic agent in the collection / neutralization process that must be executed by another external economic agent that will demand a fee for the provision of the waste collection and neutralization service.
By manufacturing general purpose greases from waste oils, it has been established that these "wastes" can be reintroduced into the production circuit without the need to neutralize them, applying the concept of circular economy.
The greases that have been created have a wide range of uses (from general coating greases to bearing greases).
The advantages of transforming waste oil that is no longer regenerated into general purpose greases are:

- Reduced costs of supplying a new grease;
- Environment protection;
- Saving natural resources;
- Job stability;
- Financial savings by reducing grease supply costs;
- Decreasing the carbon footprint

C.SUMMARY CONCLUSIONS, PERSONAL CONTRIBUTIONS, DIRECTIONS FOR CONTINUING RESEARCH

C 1. Summary conclusions

C1.1. Regeneration of used industrial oils

- After regeneration, the oils with the filtration system that uses this technology the oils acquire properties as good as the initial ones of the new oil.
- Regenerated oil can be safely reused up to 4 times provided laboratory tests are performed.
- The laboratory analyzes were performed entirely by the economic agent or at his expense as owner of the oil. These analyzes were performed in our own laboratories or at other authorized laboratories.
- Each regeneration procedure was strictly numbered and kept obvious for its recognition.
- The analysis performed for each regeneration provides information for the next regenerations and its number.
- Regenerated oil does not require special supervision.
- A quality certificate can be issued for each regeneration, which will include the date of performance, the location, the equipment from which the regeneration is made, the quantity, the specification of the oil.
- Due to the technology of filtration and addition of regenerating oils, they retain their quality values as new oil with a minor depreciation which is strictly monitored by the analyzes performed
- The term of use of the regenerated oil is the same as that of the new oil with the respective specifications.
- The term of use is given by the manufacturer of the machine / engine with strict observance of the hours of use and the specifications of the oil used.
- Exceeding the operating hours more than the hours specified in the Maintenance Norms of the machine is reflected in the depreciation of the oil and its accelerated degradation leading to increased wear of the machine.
- Thus there are wear of their components and accidental shutdowns.

Advantages of waste oil regeneration

The following are the main benefits that result from the regeneration of industrial waste oils:
-oils optimally preserve the properties of lubrication, cooling, sealing;
-reduces costs by at least 50%;
-the oil change interval is considerably extended;
-reduction of oil purchases by up to 300-400%;
-costs with the elimination of used oil decrease by 300-400%;
-increases the productivity of the machine by eliminating downtime;
-low maintenance costs due to wear to a minimum;
-the regeneration system removes particles of solid impurities greater than
1 micron;
-the regeneration system eliminates the water content from the oil formed due to the condensation in proportion of 99.9%.
- from the above results the degree of protection of natural resources (oil fields) by saving resources (circular economy);

- the stability of the jobs of the working personnel that serve the equipment;

-decreasing the school dropout of the children of those who deserve the equipment through the stability of the parents' jobs;

- reducing unemployment through job stability;

- protection of the natural environment.

As it was presented in the chapters of the doctoral thesis, at chapter I, point 1.3. the impact of used industrial oil is devastating on the environment in this case on water and soil.

See chapter 4.3. 3 times regeneration of H46 oil in the Anrozi ALRO Slatina section.

- With the money saved from oil regeneration, the production units can be developed by purchasing new equipment and new technologies.

- **Benefits:**

Maximized efficiency

• The oil optimally retains its lubricating, cooling and sealing properties.
• Reduces costs
• The oil change interval is considerably extended
• Reduction of oil purchases by up to 90%
• Costs of disposing of used oils by up to 90%
• Decreased energy consumption by increasing engine efficiency
• Increased efficiency by eliminating downtime
• Minimum maintenance costs reduced to a minimum
• Reimbursement of purchase costs as soon as possible

Environmental burden reduced to a minimum

• Technology reduces the generation of waste oils and therefore reduces the demand for oil.
• Full flow filters last four times longer, less waste.
• Transport, storage and disposal of waste oil are significantly reduced
• The risk of oil leaks and groundwater contamination decreases considerably

The technology presented in the doctoral thesis is the only filtration system that:

• removes solid dirt particles smaller than one micron in size
• removes liquid and gaseous impurities
• remove water
• maintains the chemical balance and permanently reactivates the oil.

C1.2. Transformation of waste oils that can no longer be regenerated into grease

The transformation of waste oils that can no longer be regenerated into greases was the subject of a patent application registered at OSIM published in BOPI no.2 / 2019 with no.A 2017 00593 133091 A2) which refers to the transformation of waste waste oils into multifunctional greases (according to chapter IV of the paper).

I mention that I can manufacture liquid greases for centralized lubrication systems, semi-liquid and viscous greases.

The advantages of transforming used oils into greases

From those shown in the laboratory certificates regarding the analysis of greases, their corresponding quality results:

-drip point between 144-227°C;

-penetration 190-360 (01 mm: 190);

-greases have a homogeneous appearance;

-corrosion resistance on steel blade (at a temperature of 100°C for 24 hours) - withstands;

-resistance to high pressures on the 4-ball machine '-resists.

-resistance to the static action of water - resists

-additives used in the manufacture of waste oils are specific to the materials industry,

-substantial savings of money for the grease user because he will pay smaller amounts of money for the purchase of grease from recycled oils at a quality significantly close to those on the market;

-saving natural resources;

-decreasing the degree of pollution, by recycling oil and turning it into grease;

- for the manufacture of greases from recycled oils, mixed industrial oils can be used, with different viscosity indices, thus eliminating the stage of their neutralization;

-stability of jobs by reducing acquisition costs;

-reduction of waste storage spaces;

-in perspective I am thinking of a solution for recycling used grease that will be: The topic of a patent application.

C1.3. Regeneration of used emulsions

Advantages of used emulsion regeneration:

- the regenerated emulsion optimally preserves the lubrication and cooling properties of the machining tool and the part in contact.
- the costs with the acquisition of emulsifiable concentrate with which the emulsion is formed are substantially reduced,
- the emulsion replacement interval is considerably extended;

-decreases tool consumption;

-decreases the percentage of non-compliant parts resulting from the processing.

- electricity consumption is reduced (by reducing the coefficient of friction between the tool and the part);
- water consumption is substantially reduced (water from the waste emulsion is used).

-substantial savings of money for emulsion users by decreasing the quantities of emulsifiable concentrate purchased;

-reducing the degree of pollution by optimal use of the emulsion and destroying it for a long time [16,17,18].

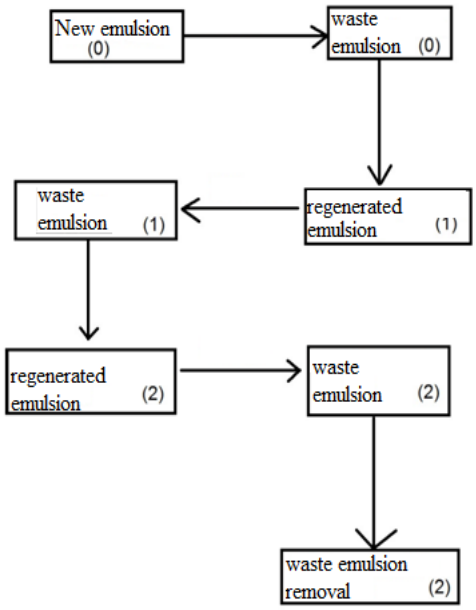

Fig.C.1 – Waste emulsion regeneration scheme - OSIM patent;[39]

From the above, we have succeeded in implementing the integrated management of industrial lubricants through regeneration:
-conservation of natural resources;
-reducing the level of harmful emissions from the air (reducing the degree of pollution by requesting the degree of use of industrial lubricants);
-reduction of waste quantities;
-reducing energy consumption;
-keeping a clean environment for us and the next generations;
-increasing the quality of life and health of children;
-reducing costs, within the degree of affordability, avoiding penalties.

Any manufacturing industrial unit that has in its technological flow industrial oils, industrial greases, industrial emulsions can reduce its costs by regeneration, thus protecting nature, practically applying the concepts of the Circular Economy.

Through this innovative concept of integrated management of industrial lubricants, a long-term protection of the environment and saving of resources in Romania as well as at the level of the European Union can be achieved.

C2. PERSONAL CONTRIBUTIONS

The researches undertaken within the doctoral thesis aimed at establishing a technology for capitalizing on used industrial oils, by regenerating them and reintroducing them into the economic circuit.

At the end of the regeneration cycles from the used oils, industrial greases are made.

In order to fulfill the committed objective of the doctoral thesis, during the doctoral internship I performed scientific documentation activities, laboratory experiments and experiments on pilot installations, I collaborated with researchers and specialists from POLITEHNIC University of Bucharest - Faculty of Materials Science and Engineering, with specialists from the economic, metallurgical and energy sector in which we performed the regeneration of used oils and their reintroduction into the economic circuit.

The original, own contributions made in the doctoral thesis are supported by the following activities:

1. We conducted a documentary study based on the literature on the types of industrial oils and greases and the possibilities of regeneration of waste oils.

2. We performed concrete activities for waste oil regeneration and economic analysis of the implementation of the proposed technology at the economic agent Grupul Feroviar Roman.

3. We performed concrete activities for waste oil regeneration and economic analysis of the implementation of the proposed technology at the economic agent Tenaris Calarasi Group.

4. We performed concrete activities for waste oil regeneration and economic analysis of the implementation of the proposed technology at the economic agent ALRO Slatina - Anozi Section.

5. We performed concrete activities for waste oil regeneration and economic analysis of the implementation of the proposed technology at the economic agent SC Rulmentul Barlad.

6. We performed concrete activities for waste oil regeneration and economic analysis of the implementation of the proposed technology at the economic agent Reloc Craiova.

7. We have carried out our own research for obtaining industrial greases from used oils and we have finalized a technological process for transforming oils into multifunctional greases.

C.3. FUTURE RESEARCH DIRECTIONS

Directions for further research in order to implement the regeneration of waste oils and their transformation when they can no longer be regenerated in grease in as many industrial units as possible so that in Romania to considerably reduce the degree of pollution due to industrial lubricants.

Given the results obtained in the doctoral thesis, it can be considered without reservation that research can be continued both in establishing a technology for regenerating waste oils at the industrial level and obtaining funding to create a technological flow to obtain used oils from grease. multifunctional.

Another direction of research development is that of monitoring the exploitation of oils, in industrial conditions, in order to establish their behavior in operation.

Another research direction in order to achieve the integral management of lubricants consists in the implementation of the OSIM Patent no. 129838/2018 regarding the regeneration of used emulsions, regeneration that can be achieved between 2-6 times as long as the concentrate can emulsify with water.

DISSEMINATION OF THE RESULTS FROM THE DOCTORAL THESIS

Papers published in ISI journals in the field of thesis:

1.Marian Simion Stratescu, Nicolae Panait, Elisa-Florina Plopeanu, Mariana Ciurdaș, Analysis of the possibilities of transformation a used industrial oil in general use grease; U.P.B. Sci. Bull., Series B, Vol. 82, Iss. 3, 2020 ISSN 1454-2331, pag 261-270, indexata ISI, https://www.scientificbulletin.upb.ro/SeriaB_-

2. Marian Simion STRATESCU, Nicolae PANAIT, Cristian PANDELESCU, Experimental research for the purpose of regeneration of used industrial oils and their reintroduction to the technological flow**,** U.P.B. Sci. Bull., Series B, Vol. 82, Iss. 4, 2020 ISSN 1454-2331, pag 235-244, indexata ISI, https://www.scientificbulletin.upb.ro/SeriaB

PATENTS OF INVENTION filed and obtained in the field of thesis:

1. OSIM patent no.129838 - authors Stratescu Simion Marian, Stratescu Sorin

PATENT APPLICATIONS filed in the field of the thesis:

1. Patent application OSIM no.00593 / 2017, Transformation of used oils into greases, authors **Stratescu Simion Marian**, Stratescu Sorin, published in BOPI no.2 / 2019.

2. OSIM Patent Application no.00978 / 2013, Regeneration of industrial waste oils, authors Stratescu Simion Marian, Stratescu Sorin, date of publication in BOPI no.8 / 2015.

PARTICIPATION IN CONFERENCES IN THE FIELD OF THE THESIS:

1. **Stratescu Simion Marian, Stratescu Sorin, CONTRIBUTIONS TO REDUCING POLLUTION IN INDUSTRIAL SPACE, at the Conference organized by the Universitatea Dunarea de Jos Galati, 22.10.2015.**

2. **Stratescu Simion Marian,** Stratescu Sorin, REGENERATION OF INDUSTRIAL WASTE OILS, at the Conference organized by the National Institute of Circular Economy and Calarasi Prefecture, 03.04.2019.

3. **Stratescu Simion Marian,** Stratescu Sorin, POSSIBILITIES OF POLLUTION REDUCTION IN ROMANIA, at the Conference organized by the National Institute of Circular Economy at ROMSIlVA Bucharest, 07.05.2019**.**

R E F E R I N T E SELECTIVE

[1]Sandor Bernard;Daniel Balint; Ioana Balint Teoria curgerii fluidelor, Editura Orizonturi Universitare Timisoara 2003

[2]Constantinescu I;V.N.Danaila;S.Galetusa Dinamica fluidelor in regim turbulent Editura Academiei 2008 pagina 10/290]

[3] Agenţia Nationalã pentru Protectia Mediului., Buletin informativ nr7/2015

[4]Ioan ŞTEFĂNESCU Lorena DELEANU Minodora RÎPĂ, Lubrefiere si lubrifianţi Editura EUROPLUS, Galaţi 2008 pagina 110 / 362]

[5].I.Florescu; D.Florescu; Olaru I. Mecanica fluidelor si masini hidraulice, Editura Tehnică Chisinau 2003

[6] Florescu Iulian , Mecanica fluidelor,Editura Alma Mater Bacau 2007 pagina 105/130]

[7] Elumalai PV, Annamalai K, Subramani L, Arularasu S, Appu Raja S. Experimental investigation on lemongrass oil–water emulsion in low heat rejection direct ignition diesel engine. J TestEval. 2019;47:1–16. https://doi.org/10.1520/JTE20170357

[8].Eugen Constantin; Diana Maria Bucur, Tratat de mecanica a fluidelor Editura AGIR 2011

[9].Ionescu D; D.Gheorghe Introducere in mecanica Fluidelor, Editura Tehnică Bucuresti 2005

[10] Kumar ARP, Annamalai K, Premkartikkumar SR. Performance comparison of Di diesel engine and LHR engine with bio diesel as fuel. Int Conf Adv Eng Sci Manag. 2012;31:312–6.

[11]. Mecanica fluidelor Cristea L; Florin Nl C.Arghirescu Editura AGIR 2013

[12].Rosca Radu Elemente de mecanica fluidelor si actionari hidraulice Editura Ion Ionescu de la Brad Iasi 2015

[13].Radu Tirulescu; Ovidiu Mihai Craciun Elemente de mecanica fluidelor si unele aplicatii, Editura Univesitatii Transilvania Brasov practice 2009

[14] Marian Simion STRATESCU, Nicolae PANAIT, Cristian PANDELESCU, Experimental research for the purpose of regeneration of used industrial oils and their reintroduction to the technological flow, U.P.B. Sci. Bull., Series B, Vol. 82, Iss. 4, 2020 ISSN 1454-2331, pag 235-244, indexata ISI, https://www.scientificbulletin.upb.ro/SeriaB

[15]. Marian Simion Stratescu, Nicolae Panait, Elisa-Florina Plopeanu, Mariana Ciurdaș, Analysis of the possibilities of transformation a used industrial oil in general use grease; U.P.B. Sci. Bull., Series B, Vol. 82, Iss. 3, 2020 ISSN 1454-2331, pag 261-270, indexata ISI, https://www.scientificbulletin.upb.ro/SeriaB_.

[16].].Brevet de inventie OSIM nr.129838, autori Stratescu Simion Marian, Stratescu Sorin.

[17]. Regenerarea uleiurilor uzate industriale , Cerere Brevet inventie OSIM nr.00978/2013, autori Stratescu Simion Marian , Stratescu Sorin, data publicarii in BOPI nr.8/2015.

[18]. Transformarea uleiurilor uzate in unsori , Cerere Brevet inventie OSIM nr.00593/2017, autori Stratescu Simion Marian , Stratescu Sorin, publicata in BOPI nr.2/2019.

[19].Andreev, V.G. and Tolmachev, G.P. (2002). Future directions in recycling of petroleum-containing wastes. Thermalcracking-optimum method for utilization of waste oils, *Chemistry and Technology of Fuels and Oils* 38(6