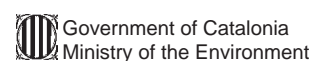
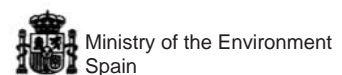


MEDITERRANEAN

Recycling Possibilities and
Potential Uses of
Used Oils

CLEANER
production

Regional Activity Centre for Cleaner Production (RAC/CP)
Mediterranean Action Plan



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If you consider that some part of this study could be improved or there is any lack of precision, we would appreciate if you could notify it to us.

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INTRODUCTION

Within the context of the Mediterranean Action Plan (MAP), the Regional Activity Centre for Cleaner Production (RAC/CP) has made this study of the possibilities for Recycling and Reusing Used Oils with the purpose of presenting the main processes and technologies that enable the use and application of both used mineral oils from industry and vegetable oils from catering companies and restaurants.

The study focuses on the countries covered by the Mediterranean Action Plan where there is a wide diversity of features and characteristics concerning the management of used mineral and vegetable oil.

Even so, from the list of countries in the Mediterranean Action Plan and the consultations carried out in each one of them, there are clearly two groups according to the actions that each country is carrying out in connection with the use and recycling of used oils.

The countries in the first group are those that are aware and concerned about environmental matters and at the initial stages with regard to environmental legislation and, in consequence, everything else to do with the regulation and control of environmental actions that are also carried out in the country.

The countries in the second group are those that are aware and concerned about environmental matters and that have a legal framework that regulates actions with regard to the environment.

Broadly speaking, the first group is made up of countries that do not belong to the European Community or its sphere of influence, whereas the second group is formed of countries within the sphere of influence of the European Community where actions are oriented by supranational regulations such as the Community's Directives, which results in the efforts of each country being directed towards a final objective that is common to all of the other countries.

Contents of the study

The study is structured according to the differences in relation to the source of origin, treatment and application of both used mineral and vegetable oils.

Starting with two sections that give an introduction and background information, the study goes on to present all of the aspects connected firstly with mineral oils and then vegetable oils.

With regard to mineral oils, the contents of the study are as follows:

- *Introduction*: a definition of used mineral oil is given. The effects of used oils on man and the environment appear in this section, together with the treatment processes that can be applied to used mineral oils. A characterisation of used mineral oils is also given.
- *Collection logistics*: a description is given of the systems of collection, from the source where the used oil is produced to the delivery of the product to the processing company.

The various existing treatment processes that fall into three groups are then explained:

- *Minimisation processes at source / reprocessing* of used mineral oils.
- *Re-refining processes / regeneration* of used mineral oils.
- *Energy recovery* systems.

After the description of the processes and technologies, the following sections are organised as follows:

- Possible ways of *exploiting* used oils and separated products.
- *Economic aspects* deriving from the transportation, treatment and reuse of used mineral oils.
- *Practical case examples of companies* that are carrying out industrial processes with used mineral oils.

- The final section deals with *proposals and conclusions* that cover different aspects connected with the used mineral oil subsector.

The structure of the study with regard to **used vegetable oils** is as follows:

- *Introduction*: a definition of used vegetable oil is given, together with a description of the harmful effects that are associated with them. A characterisation of used vegetable oils is also given.
- *Collection logistics*: a description is given of recovery and transportation systems.
- The following section deals with the *pretreatment* process to which used oils are subjected.
- A description is then given of the *possible uses* for used vegetable oils.
- The final section deals with *proposals and conclusions* that cover different aspects connected with used vegetable oils.

Methodology

The following aspects have been taken into account during the carrying out of this study. Given that the study focuses on the countries that come under the Mediterranean Action Plan, the different official authorities and/or National Focal Points of the RAC/CP in each country have been consulted in order to incorporate the information provided by each one and thus obtain a better understanding of the situation in each country.

National and international companies and institutions that are directly involved with all areas concerning used oils have also been consulted. For example, those in charge of the current commissioning by the European Community of a study on the available bibliography on the subject of used oils have been contacted.

Lastly, consideration has been given to the currently available bibliography on the matters dealt with in this study.

BACKGROUND

From the information provided by the countries within the sphere of influence of the European Community, it can be said that both mineral and vegetable used oils are considered to be hazardous waste in accordance with the current prevailing regulations and Community Directive 91/689/EEC. The hazards of these types of waste arise as a result of the effects that they can have both on health and the environment.

In view of these effects, guidance is given in Directive 91/156/CEE on actions to be carried out concerning the management of this type of waste. Consideration is given to the following basic lines of action:

- Reprocessing, minimisation at source: via the application of clean technologies that enable greater savings of natural resources to be made.
- Re-refining / regeneration: re-use through application in new uses.
- Combustion: valorisation as a source of energy, either for producing heat or for obtaining energy through cogeneration.

According to the information made available by each country, however, it comes to light that all countries in both group one and group two are directing their efforts towards reducing uncontrolled dumping and treating used oils as products capable of being exploited in different processes.

1.1 Introduction

Used mineral oil is any industrial oil that has become unsuitable for the use to which it was initially assigned. These especially include used oils from combustion engines, transmission systems, turbines and hydraulic systems, the different sectors of the car industry and industrial shipping activities.

Used mineral oils are classified according to current European regulations as hazardous waste due to the effects that they can have both on health and the environment.

The direct effects that these types of oil can have on health include the following:

- Irritation of lung tissue due to the presence of gases that contain aldehydes, ketone, aromatic compounds, etc.
- The presence of chemical elements such as Cl (chlorine), NO₂ (nitrogen dioxide), H₂S (hydrogen sulphide), Sb (antimony), Cr (chrome), Ni (nickel), Cd (cadmium), and Cu (copper²) that affect the upper respiratory tract and lung tissue.
- They produce asphyxiating effects that prevent oxygen transportation due to their content of carbon monoxide, halide solvents, hydrogen sulphide, etc.
- Carcinogenic effects on the prostate and lungs due to the presence of metals such as lead, cadmium, manganese, etc.

Direct effects on the environment that stand out include the following:

- The pollution of soils, rivers and the sea due to their low biodegradability.
- On coming into contact with water, they produce a film that prevents oxygen circulation.

- Uncontrolled combustion can lead to the emission of chlorine, lead and other gas elements into the atmosphere, with the corresponding effects.

In view of these effects, guidance is given on actions to be carried out regarding the management of this type of waste. Consideration is given to the following basic lines of action:

- **Reprocessing, minimisation at source:** industrial oils used in hydraulic systems can be reused by both the company itself and the same hydraulic system after the oil has been put through a cleaning process. This treatment prolongs the life cycle of the oil in question.
- **Re-refining / Regeneration:** the objective of the different regeneration processes that exist is to obtain base oils that can be used in the production of new oils. Other by-products resulting from the processes are used in other applications.
- **Combustion:** the end objective of combustion is energy recovery for obtaining heat, which is carried out in cement works, power stations and refineries, or energy recovery by means of cogeneration installations for obtaining electricity. The combustion process is acceptable provided that the parameters of emission into the atmosphere, especially those of heavy metal emissions contained in used oils, are respected. The process can be carried out in installations with a power capacity of over 3MW.

According to the information provided by the different countries, the uncontrolled dumping of used oils is totally prohibited.

The different systems for treating oils once they have been rejected from the activity for which they were produced are given in the diagram.

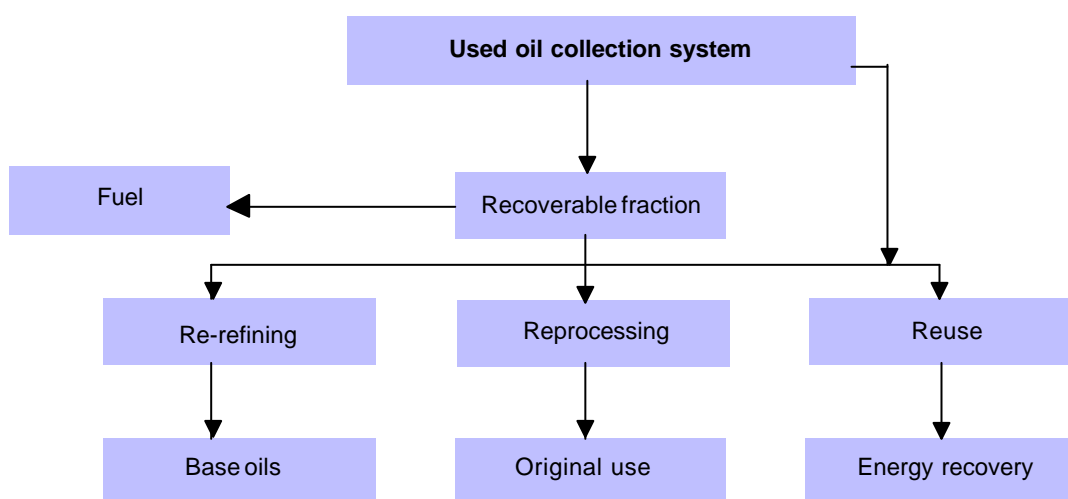


Figure 1. Management systems for used oils

The end management process that has been used the most up until the present time is combustion. This is due to one of the main characteristics of used mineral oils in that they can be physically burned (leaving aside the fact of uncontrolled dumping which is logically no longer considered as a system of management).

On this point, the fact that energy recovery clearly competes with other sources of energy generation, especially petrol and gas oil, needs to be taken into account. At industrial level, economic factors are still top priority with regard to environmental issues, so that when energy is required for industrial processes that are being developed, the source of energy that is used is the one with the lowest economic cost; the cost of used oil is normally lower than that of both gas oil and petrol. It has thus been the industrial sector itself that has stimulated energy recovery from used oils.

Mineral oils that are used and then become spent or used oils are made up of base lubricants and additives that have been developed specifically for lubrication that give special characteristics to the oil. Base lubricants are mostly hydrocarbons whereas the additives, that make up between 15% to 20% of the oil, contain organic compounds derived from sulphur and nitrogen that contain metals.

After the oil has been used and has turned into used oil, the constituent pollutants that can be found are as follows:

POLLUTANTS	EXAMPLES	ORIGIN
Polynuclear aromatic hydrocarbons		Petroleum - Base Lubricant
Mononuclear aromatic hydrocarbons	Alkyl benzene	Petroleum - Base Lubricant
Dinuclear aromatic hydrocarbons	Naphthalene	Petroleum - Base Lubricant
Chlorinated Hydrocarbons	Trichlorethane	Use of contaminated oil
Metals	Barium	In Additives
	Aluminium	In engines
	Lead	In fuel
	Zinc, Chrome	
Inorganic acids derived from chlorine, sulphur and nitrogen		
Organic compounds such as aldehydes, acids, etc.		

Table 1. Pollutant compounds in used oils

The process by which used oil is generated appears in the diagram, together with the possible applications for used oils

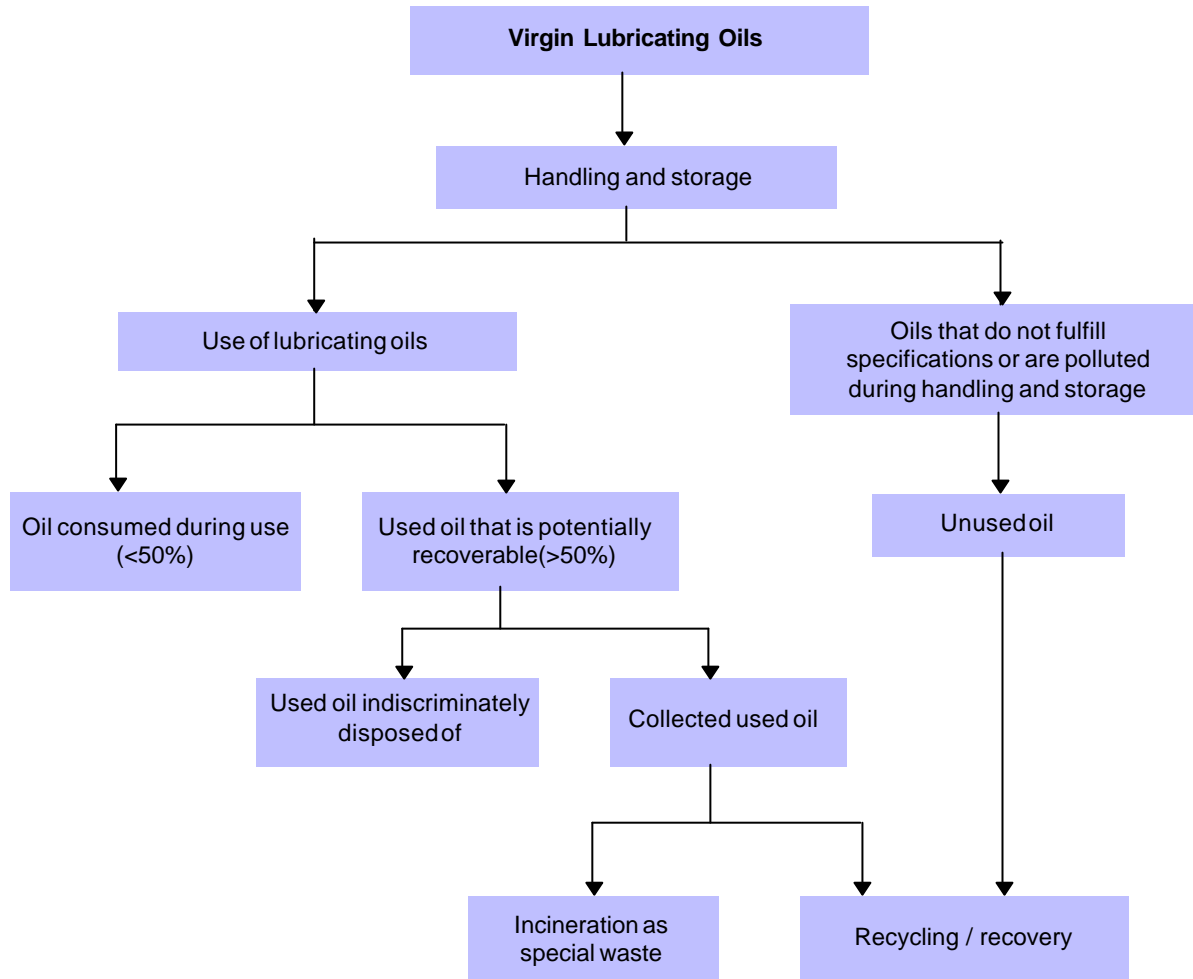


Figure 2. Process of generation of used oils

The percentage of used oil collected in relation to the total amount of oil that is potentially recoverable varies a lot and depends on the countries and how well the collection logistics are organised. The values are around 90 % in the best of cases, although on average, and for countries with efficient collection logistics, these values can reach around 80%.

1.2 Collection logistics

Systems for collecting oils are conditioned according to the types of production centre. There are normally a large number of collection points, and the quantities being collected are usually small. On the other hand, the number of industries specialising in oil recovery and treatment is small so that each one has to cover an extensive geographical area.

For the purposes of collection logistics, a distinction must be made between:

- The collection and initial storage of used oil, which must be carried out at the points where it is generated, i.e. industry, garages, etc.
- Collection and transportation from the points where used oil is generated to the treatment centres or industries.

As far as the conditions, systems of collection and initial storage at the points where used oil is generated are concerned, the countries within the sphere of influence of the EEC follow the guidelines laid down in the European Union Directives. Used oil is normally stored in containers prepared especially for this purpose. The principle upheld is that it is the generator of the used oil who is responsible for collecting and storing it in acceptable environmental conditions.

It is regarded that there can be no substantial variations in the collection logistics used by countries that are outside of the sphere of influence of the EEC, given that the conditions, in terms of centres of production and treatment centres, must be approximately the same.

An important criterion that needs to be underlined when organising the systems of collection is that the separation at source of different types of used oils makes subsequent treatment processes easier and thus increases the value of the used oil because the levels of polluting substances in the end products are also reduced at the same time. Unstructured collection systems (where different types of used oils are collected) lead to more difficulties and involve a higher economic cost in later treatment and recovery processes.

Collection systems therefore need to be organised according to the different sources where used oil is produced, with separation being carried out at source to make subsequent treatment processes easier and to reduce their cost.

Generally speaking and according to the stipulations of the regulations in each country, it should also be pointed out that the uncontrolled dumping of used oils is illegal. However, this is clearly still customary practice in many countries, either because no collection and treatment system exists or because the population is not receptive to issues connected with the environment.

The transportation of stored oils from the production centres to the management companies must be carried out via companies that are authorised to carry out the service and that form part of nation-wide organisations that, in the final analysis, organise and control all actions.

These carriers must keep a record of the collection points, quantities and classification of the oil that is collected. Thus, when the collection is made, two samples are taken and correctly coded. The producer keeps one of the samples and the carrier the other. The two samples are essential for resolving differences should any anomalies be detected in the composition of the used oil.

The carrier delivers the oil that has been collected and transported to the treatment centres by vehicles adapted especially for this purpose, together with the samples that have been taken and that will be used in the corresponding analyses. Depending on the components in the oil, authorisation is then given or refused for the following treatment of the oil in the recycling plant.

The analytical tests that are normally carried out include density determination, water content, sediments, PCBs, metals, sulphur, chlorine, etc.

When the carrier delivers the used oil to the company for treatment later on, the oil is put into storage during the period prior to the results of the analyses being given. Once the results are given, the oil is either subjected to treatment in the plant or not, depending on the actual results.

As organisations and carriers gain more experience with the passage of time with regard to the collection system for used oils, they come up with their particular norms and conditions of collection, principally in relation to the level of pollutants in the different consignments that are collected.

The different types of used oils have different applications and therefore different commercial values. The higher the level of segregation at source, the higher the value of the used oils, and the volume of oils that are collected will therefore increase.

A diagram showing collection and transportation is given below.

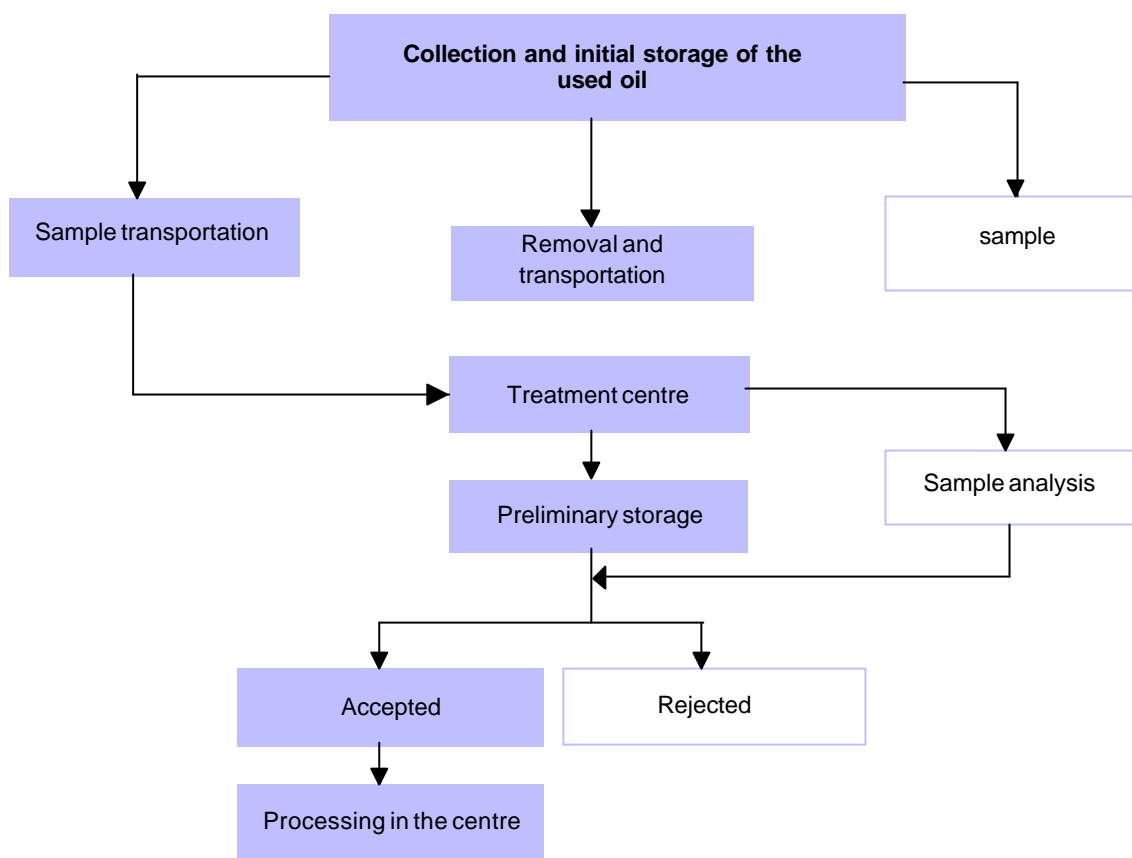


Figure 3. Diagram of the collection and transportation of used oil

The following data is from consultations with the corresponding authorities and/or the National Focal Points on used mineral oils in Italy.

The “Consorzio Obbligatorio degli Oli Usati (COOU)” is the entity responsible for the collection and treatment of used mineral oils in Italy.

According to the data given by the COOU, around 189,000 t of used oil were collected in 1999, which represents 90% of all of the used oil that is generated in Italy.

The quantities collected according to each activity were as follows:

Activity	Quantity of collection (t.)
Service stations	7,200
Garages	74,294
Vehicle breaker's yards	2,443
Road transport	8,509
Industry	38,042
Navy	2,566
Agriculture	2,385
Aviation	274
Electrical companies	2,725
Rail transport	1,947
Public administration	2,062
Army	1,066
Others	41,259
Sub-collectors	4,186
Supermarkets	7
TOTAL	188,971

Table 2. Quantities collected in Italy in 1999 by economic sectors

Collection is carried out through 70 companies that service the production centres. Small-sized containers are used for collection and special containers prepared in the same companies that make the collection are used for storage. The collected oil is then sent to the COOU storage locations. During unloading at the COOU storage locations, samples are taken and analyses made, with checks made of the parameters of density, water content, sediments, PCBs, inflammability, metals, lead, chlorine, fluoride, sulphur and ash.

Following the analysis, and if all of the quantified parameters are within the authorised limits, the used oil is mainly subjected to re-refining or controlled combustion, which are the only two ways that treatment is authorised in accordance with Directive 87/101/EC which the Italian government follows.

In Spain, used mineral oils are regulated by Law 10/1998 of 22 April on Waste. Classification as hazardous waste appears in Royal Decree 952/1997.

From data available for Spain, 500,000 t of lubricating oils are sold every year, 60% of which is used in the car industry and the rest in industrial activities. Around 50%, or 250,000 t, is consumed in process activities and the remaining 50% is used oil that is potentially recoverable.

Given the characteristics of the actions that are carried out in Spain, the waste management of used oils from collection through to end treatment has been subsidised every year. Different modules are established for the amount of subsidy received for end treatment according to the oil that is to be regenerated, recycled or used for energy recovery.

The trend of development in the collection and use of oil is given in the following table.

Year	Oil collected (t)	Regeneration ¹	Recycling ²	ENERGY	RECOVERY
				Electricity	Heat
1994	73.823	4%	-	-	96%
1995	106.528	13%	-	2%	85%
1996	119.831	23%	-	4%	73%
1997	134.646	21%	2%	12%	65%
1998	173.490	18%	2%	24%	56%

¹Regeneration: Base oil production for producing new oils.
²Recycling: Manufacturing of asphalt products from de paraffin in used oils

Table 3. Collection and use of used oil in Spain per year

From the data given above, the following is clear:

- There has been an increase in the quantity of used oil collected since 1994, with an overall increase of 250% in 5 years.
- There has been an increase in treatment involving regeneration and electricity production and a decrease in processes for obtaining heat. It is envisaged that regeneration will continue to increase in the coming years due to the fact that there are various projects under way at the present time.
- The final destination of between 75,000 and 80,000 t. of used oil is unknown. This could very well be due to uncontrolled dumping.

The French authority responsible for used oils sector is the ADEME (Agence de l'Environnement et de la Maîtrise de l'Energie).

According to data provided by this entity, 247,700 T of used oils were collected in 1999, which represents 82 %. The quantities that were collected according to the different sources of production are given below.

Activity	Quantity collection
Garages	113,950
Vehicle breaker's yards	2,450
Road Transport	24,800
Industry	37,150
Agriculture	7,400
Public Administration	29,750
Army	2,450
Sub-collectors	29,750
Total	247,700

Table 4. Quantities collected in France in 1999 by economic sectors

Collection is made through companies that are responsible for collecting and transporting used oils. Collection and transportation concessions are given for a 5-year period, and the collector is responsible for the services in the specific area that it has been allocated. In order to reduce the costs of collection, the minimum collection quantity is 600 Kg of used oil. There has been an increase in the unit quantity collected from each production centre in recent years.

As for the subsequent application of used oils collected in France, there is a clear predominance of cement works (20 plants, with a treatment capacity of 228,720 t/year) in relation to regeneration industries (only 1, Ecohuile, with a treatment capacity of 110,000 T/year). In all cases, chemical analyses are carried out prior to the product being accepted in order to determine the PCBs, chlorine, water, heavy metals, net weight, etc.

Grants and subsidies are available for both collection and subsequent transportation and treatment of used oils.

The collection system described in this section is of a general nature and variations exist in each country according to their specific circumstances and the information provided by each one.

Nevertheless, it is believed to be an effective system, as is shown by its efficiency in terms of the quantities collected in countries where this collection logistics is applied. Excluding the particular circumstances and conditions in each country, this system can therefore be adopted and implemented in countries where there is no structured and systematic system of collection at the present time.

1.3 Minimisation processes at source / the reprocessing of used mineral oils

Application of the criteria for preventing and reducing pollution at source, in terms of the actions to be carried out with used oils, points to actions directed towards reprocessing used oils as being the first option. These actions are very incipient, however, and are not generally used as a treatment system in industry.

Reprocessing involves the recovery of used oil and its reconversion into top quality oil that can be used in production processes. Three objectives are achieved with this type of action:

- There is a considerable reduction in the quantity of used oil that is considered waste.
- In business terms, important savings are made in the purchase of new oils.
- The life cycle of the oil is extended with reprocessed oil being re-introduced into the same production cycle.

Processes for achieving minimisation at source are based on the following stages:

1. Vacuum distillation: a dehydration and degassing stage, during which other volatile pollutants are also eliminated.
2. Ultrafiltration: a stage where the used oil is subjected to sedimentation and filtration processes to eliminate metal particles and other solids contained in the oil.
3. Readditivation: additives are added throughout this process in order to obtain clean oil with the desired characteristics.

Chemical analyses are carried out prior to the oil being introduced into the system in order to establish the condition of the used oil and to thus be able to define the intensity of treatment and additivation that will be necessary to obtain the required end characteristics.

1.4 Re-refining processes / the regeneration of used mineral oils

1.4.1 Introduction

The increase in the variety of used oils in recent years has resulted in the fact that traditional oil treatment processes based on acid/earth technologies do not obtain the required end qualities for base oils. Moreover, concern over environmental issues makes it even more difficult to obtain suitable processes.

The aforementioned technologies based on processes using acid and earth are at present obsolete for environmental and economic reasons. Economically speaking, large investments need to be made in installations while environmentally there is the problem of treating the acid earth generated in the process. Resolving these problems has meant resorting to the introduction of technologies that incorporate vacuum distillation and hydrogenation processes.

Procedures that are common to processes based on vacuum distillation and hydrogenation are:

- The distillation of used oils that have been previously dehydrated.
- The hydrogenation of distilled products.

The different processes that exist are described below and are grouped according to the basic technologies being used:

- Technologies based on acid / earth treatment.
- Technologies based on vacuum distillation and hydrogenation.
- Technologies based on vacuum distillation and earth treatment.

1.4.2 Acid / earth treatment processes

These are processes that are nowadays obsolete. These processes are basically as follows:

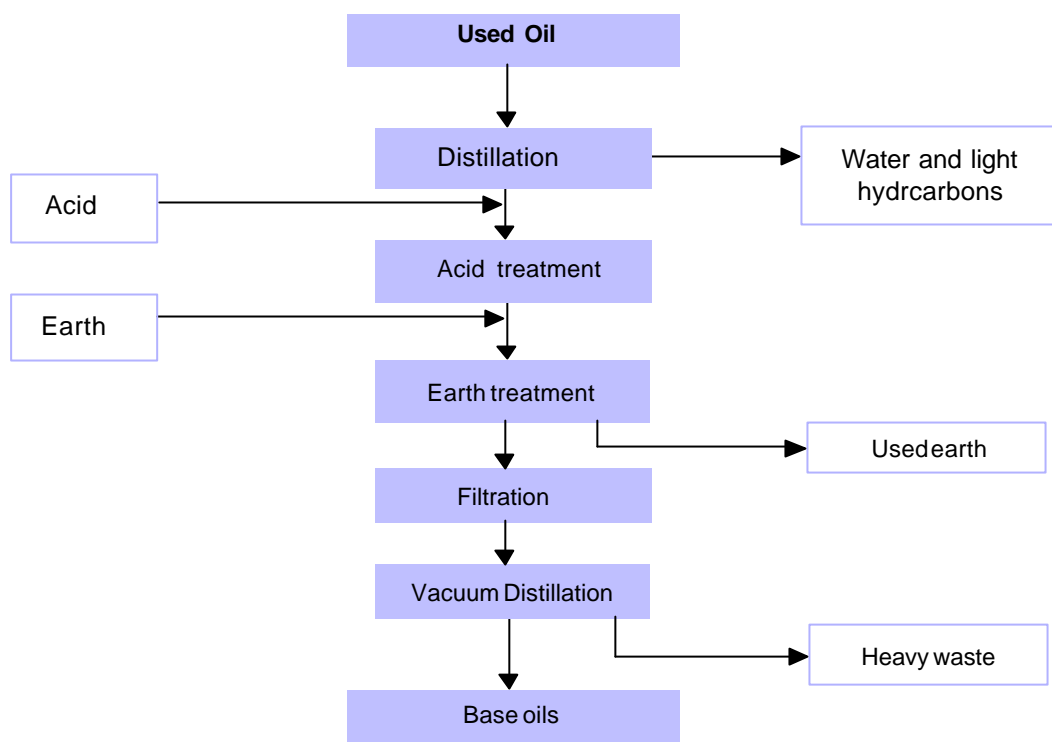


Figure 4. Basic outline of acid/earth treatment technologies

Acid/earth technologies are based on treating the substrate with sulphuric acid to eliminate the polluting substances and then treatment it with earth to neutralise the resultant product. In this way, treatment with earth gives the required colour and odour. It does raise the problem of how the acid waste that is generated is going to be used and applied, which in many cases is toxic and hazardous with the resulting problems of disposal.

Meinken technology

Meinken technology is no longer used for economic reasons and because of the problems that it generates with the treatment of acid earth. It also involves problems of internal corrosion and disposal.

There are some refineries that work with modified Meinken technology at the present time. The inclusion of thin film and contact distillation techniques enables them to reduce the quantity of sulphuric acid to 3% and of earth to 3.5%. Some of them also include hydrogenation.

The main advantages of this process are the low investment and maintenance costs, the possibility of treating low quality used oils, and the flexibility and ease of handling the process itself.

1.4.3 Vacuum distillation and hydrogenation processes

These types of process are the ones that are most used in Italy, where 93% of collected oil is treated by these technologies. This represents 175,700 t of oil per year. 60% of the resultant products are base oils and 8% are light oils. Waste containing additives, asphaltenes, compounds from oxidation and polymerisation, metals and other impurities produced during the refining process is destroyed by combustion processes in special plants.

At the present time, Italy is the number one European country in terms of the quantity of re-refined oil in relation to the total amount of oil produced.

The following diagram gives a general outline of technologies based on vacuum distillation and hydrogenation.

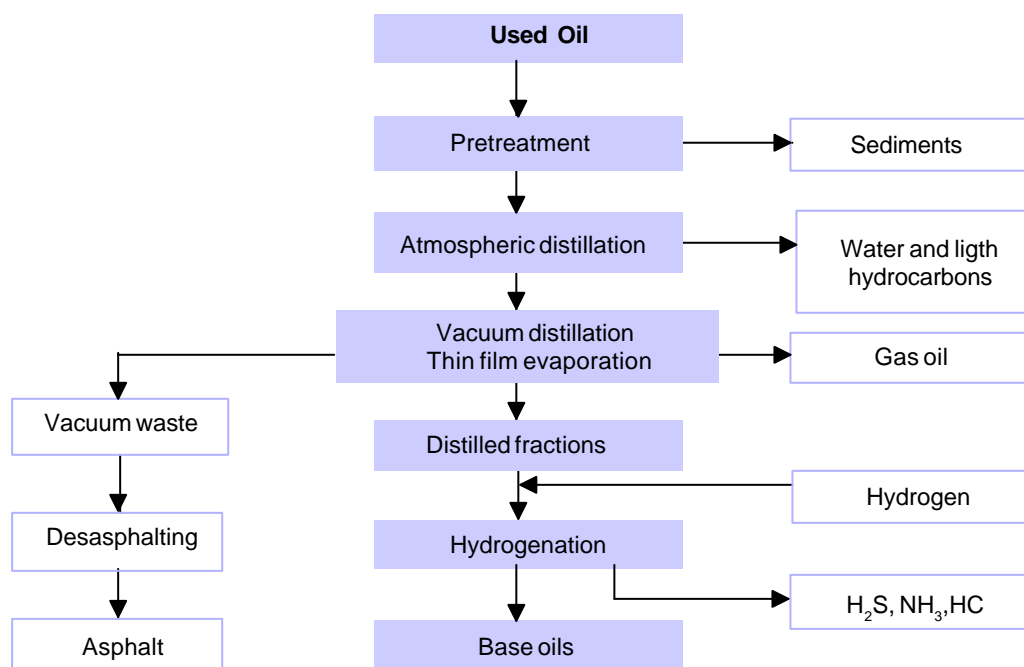


Figure 5. Diagram of vacuum distillation and hydrogenation processes

KTI technology

The KTI process (Kinetics Technology International), also known as KTI Relube Technology, combines vacuum distillation and the hydrogenation treatment to eliminate most of the polluting substances in used oil.

The basic steps of the process are as follows:

1. Atmospheric distillation: this involves eliminating water and light hydrocarbons.
2. Vacuum distillation: the resultant product comes within the range of the lubricating oils. The working temperature should not exceed 250°C.
3. Hydrogenation of the products that have been vacuum-distilled: oils distilled in the previous stage are subjected to a hydrogenation treatment to eliminate the sulphur, nitrogen and oxygen compounds. This stage is also used to improve the colour and odour of the oil.
4. Fractionation: the hydrogenated oil is separated into different base oil fractions according to the specifications and necessary requirements of the product.

This technology accepts PCBs and other hazardous materials, and it gives an efficiency of 82% of high quality base oils (in relation to treated dry used oil).

The waste produced in the vacuum distillation stage contains additives, asphalt by-products, oxidised products and other impurities that have an economic value.

The first re-refinery based on this technology was established in Greece in 1992. Plants of this type also exist in Tunisia and California.

A diagram of the process appears below.

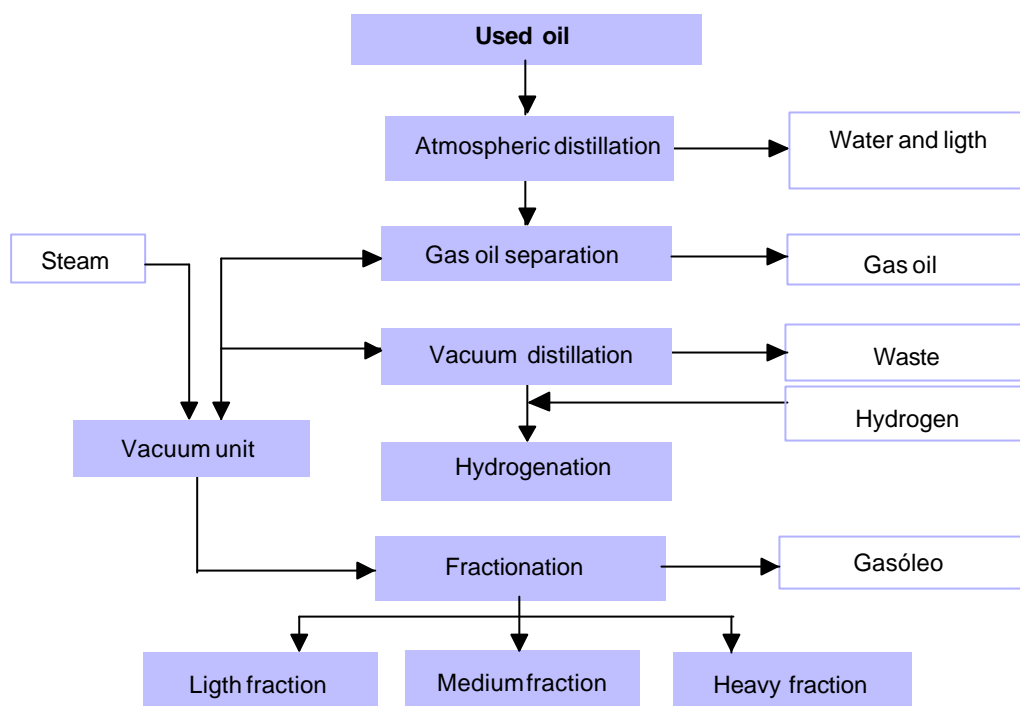


Figure 6. Diagram of KTI technology

Mohawk technology

This technology was developed by the Mohawk Oil Company in Canada and is based on the KTI technology explained above.

The difference between this technology and others in the same group (vacuum distillation and then hydrogenation) is based on knowledge of the chemistry of lubricants and additives that are to be found in the oils in different temperature and process working time conditions. It leads to improvements in the characteristics of the finished products, both in terms of the life of the catalysts that are involved and an increase in the resistance to corrosion.

The basic steps of this process are as follows:

1. Pretreatment: this involves the precipitation of polluting substances and eliminates problems of fouling during the distillation stage. The life of the catalysts is also extended at the same time.

2. Atmospheric distillation: this eliminates water and hydrocarbons.
3. Vacuum and thin film distillation: this involves the recovery of the hydrocarbons from lubricating oils.
4. Hydrogenation: the stage where the oil is purified.
5. Fractionation: different base oil fractions are obtained.

This technology is better than others based on the same process due to the fact that the equipment does not need to be cleaned so frequently. The fact that the vacuum is produced mechanically instead of by using steam leads to a reduction in the quantity of wastewater to be treated as a resultant effluent of the process. The fact that corrosion of the equipment is also reduced enables lower-cost materials to be used in the installation. High quality base oils are obtained with this technology.

Evergreen Oil in Newark (California) and Breslube in Windsor (Canada) hold the licence for this technology.

BERC or NIPER technology

Technology developed by Bartlesville Energy Research Center in the USA, later renamed the National Institute of Petroleum and Energy Research.

This process is similar to KTI that is described above, although it differs in that it incorporates a treatment with solvents.

The basic steps of this process are as follows:

1. Atmospheric distillation: a dehydration stage at atmospheric pressure.
2. Vacuum distillation: the elimination of light hydrocarbons.
3. Pretreatment with solvents: the addition of a 3:1 solvent made up of butyl alcohol, isopropyl alcohol and methyl ethyl ketone in a 1:2:1 proportion that extracts compounds that can potentially pollute the resultant products.

4. Recovery of the solvents: using sedimentation and/or centrifugation processes, the used oil-solvent mixture is separated from the heavy metals, additives and other compounds that pollute the mixture. The solvent is recovered for reuse.
5. Fractionated distillation: different base oil fractions are obtained.
6. Hydrogenation treatment or earth treatment: impurities are eliminated, which improves the colour and odour of the end products.

The efficiency of this technology gives between 75% and 85% base oils. The resultant waste from the process is applied to asphalt, which gives better economic results in relation to technologies based on acid/earth treatments.

The most outstanding drawback is the high cost of extraction in terms of energy when solvents are used.

A diagram of the process appears below.

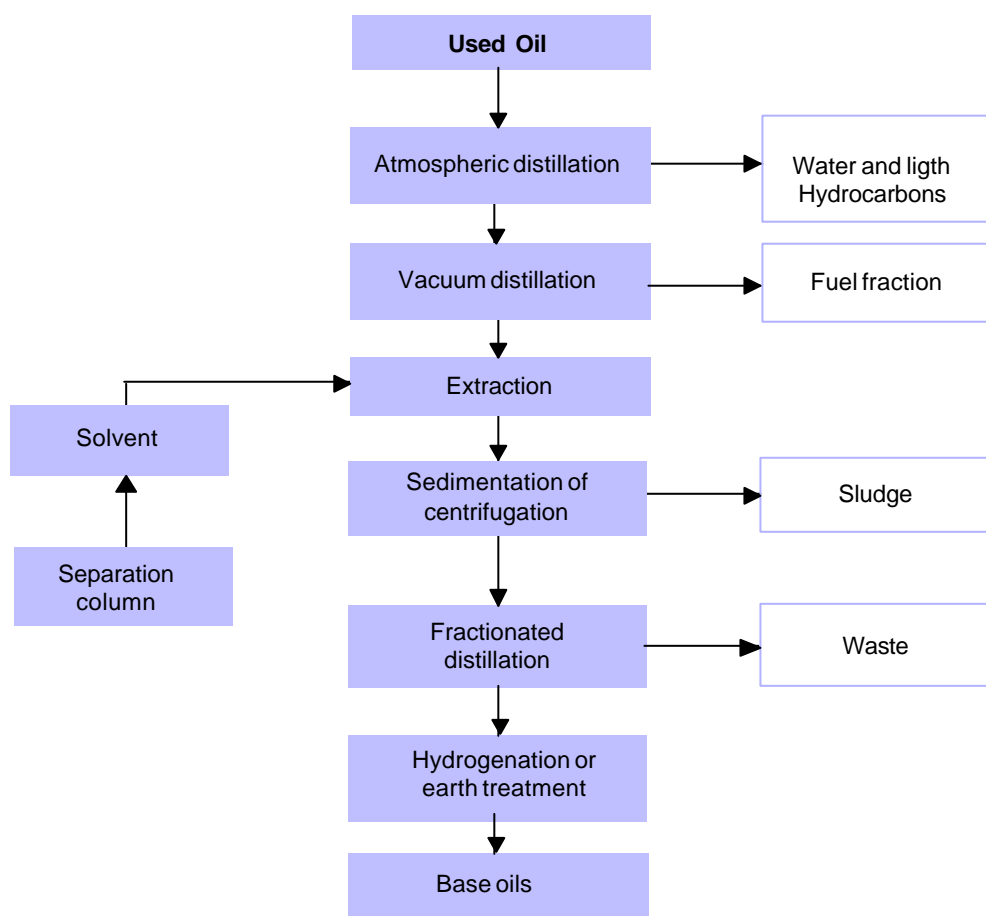


Figure 7. Diagram of BERC or NIPER technology

PROP technology

Developed by the Phillips Petrol Company, this includes a chemical demetalisation stage to eliminate polluting substances in used oils.

The basic steps of this process are as follows:

1. Demetalisation: this brings about a decrease in the heavy metals contained in the oils by mixing the used oil with a watery solution of diammonium phosphate, which leads to the formation of metallic phosphate compounds.
2. Separation of the metallic phosphates: the metallic phosphate formed in the previous stage is eliminated by filtration.
3. Vacuum distillation: the light hydrocarbons and water are eliminated.
4. Earth treatment and Ni/Mo catalyst: the oil is mixed with hydrogen and passed through an earth bed with the Ni/Mo catalyst.
5. Hydrogenation: sulphur, oxygen, chlorine and nitrogen compounds are eliminated during this stage, which improves the colour of the resultant oil.

Advantages of this technology include the high quality of the base oils obtained, with less than 10 ppm of metals, sulphur and nitrogenous substances. It also produces 90% base oils and the technology is highly friendly to the environment.

The most important drawbacks are the high cost of investment and the fact that the hydrogenation stage requires an earth absorbent treatment.

The only plant with PROP technology in operation at the present time is in Mexico.

A diagram of the process appears below.

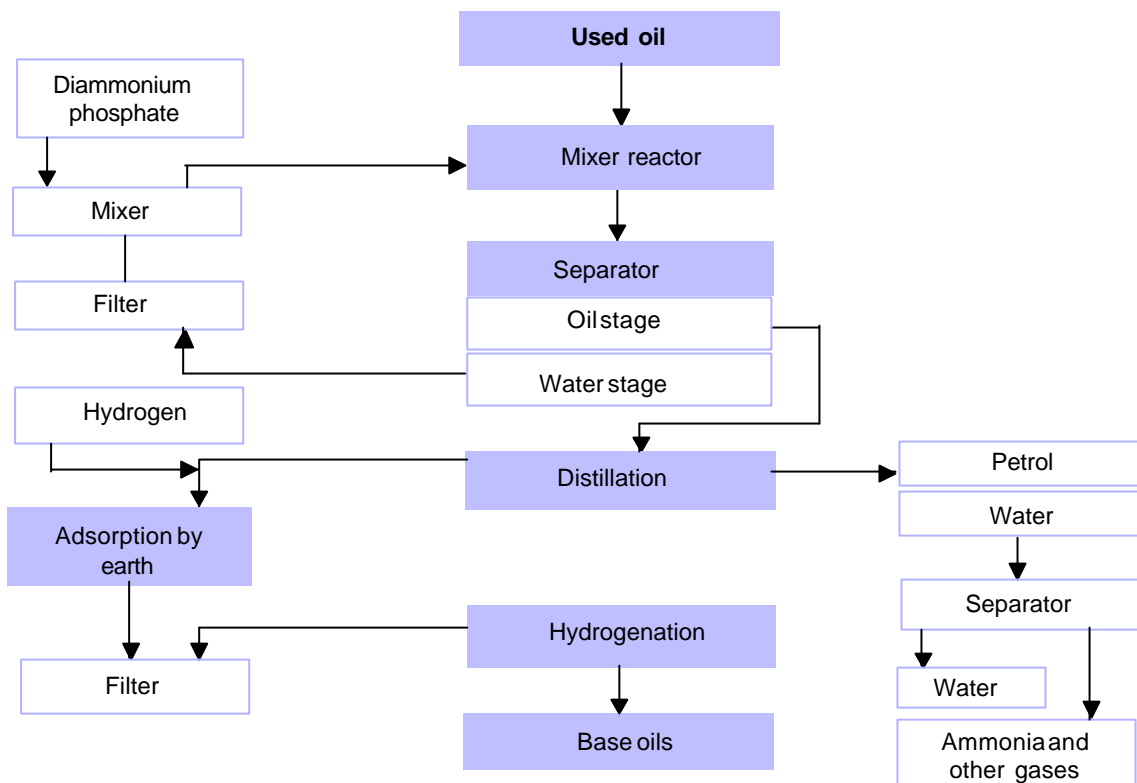


Figure 8. Diagram of PROP technology

Safety Kleen technology

This technology is the result of the combination of thin film vacuum distillation and hydrogenation through a catalytic bed.

The basic steps of this process are as follows:

1. Atmospheric distillation: stage to eliminate water and light solvents.
2. Vacuum distillation with thin film evaporators: lubricating oils are separated from the heavy solvents.
3. Hydrogenation through a Ni/Mo catalytic bed: a stage where thermal stability of the colour and odour occurs by reducing the aromatic compound content (agents that can potentially cause mutations).

4. Kerosene separation.
5. Drying of the base oil: the final stage in obtaining base oils.

The hydrocarbons that result from the atmospheric distillation, together with all of the light compounds obtained in the different steps, are used as fuel in the plant following prior treatment due to the high chlorine content. The catalytic bed is also regenerated and the water that results from distillation is also subjected to an appropriate treatment. It is for this reason that Safety Kleen technology does not generate collateral by-products.

At the present time, there is a plant operating in East Chicago, Indiana (USA), and another in Breslau (Canada).

A diagram of the aforementioned technology appears below.

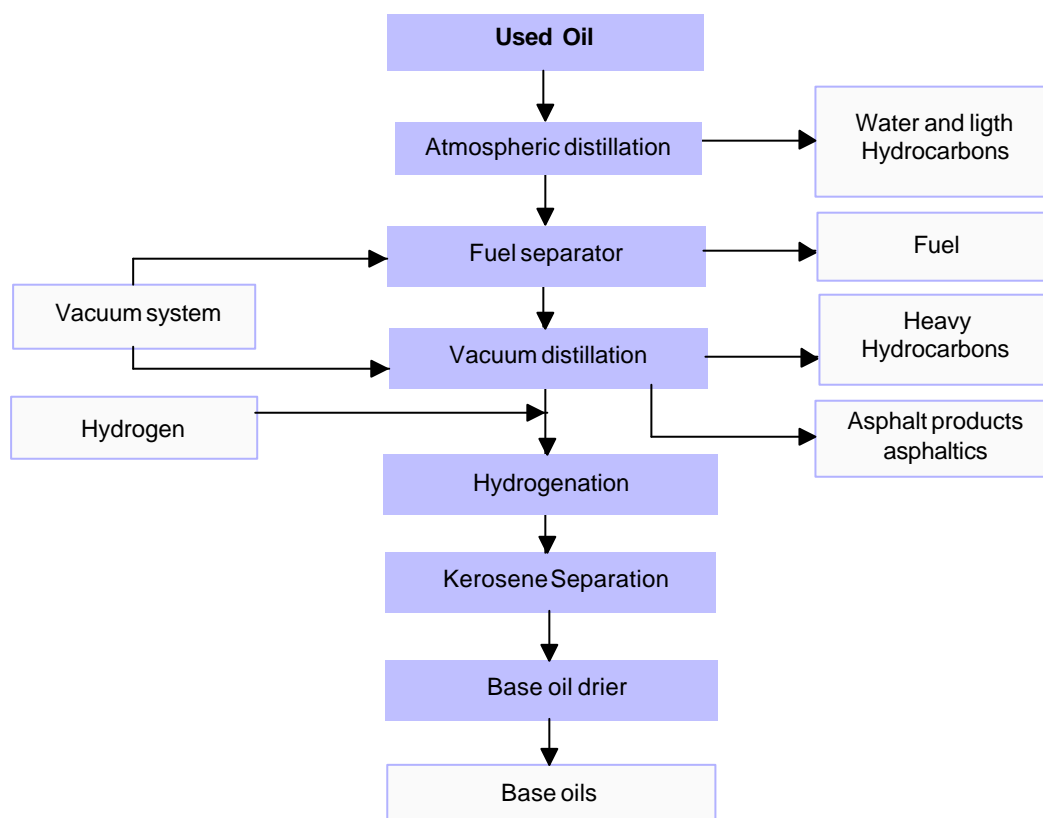


Figure 9. Diagram of Safety Kleen technology

IFP Technology / Snamprogetti Technology

IFP technology was developed by the “Institut Français du Pétrole” and is also known as the Selectopropane Process. This process combines vacuum distillation and hydrogenation but in this case extraction is carried out using liquid propane. This form of extraction is similar to the kind carried out in crude oil refineries to separate out asphaltenes.

The basic steps of this process are as follows:

1. Atmospheric distillation: water and light hydrocarbons are eliminated.
2. Vacuum distillation and extraction of the oil-containing part using propane: oil from atmospheric distillation is subjected to extraction with liquid propane at a temperature of between 75 and 95°C. Light and medium base oils are recovered in this phase.
3. Hydrogenation: stage where the propane is separated from the propane-oil mixture. Asphaltic compounds, oxidised hydrocarbons and solids in suspension are also separated in this stage. The bright stock fraction is recovered from the waste from vacuum distillation.
4. Final stage of hydrogenation of the bright stock fraction.

There is a difference between the IFP and Snamprogetti processes concerning the recovery of the bright stock. In the IFP process, the waste is extracted with the propane remaining from vacuum distillation. The fraction that is obtained is demetalised and hydrogenated through two catalyst beds to obtain the bright stock. In the Snamprogetti process, a second extraction using propane is carried out of the vacuum distillation waste, which is combined together with the vacuum distillate in a hydrogenation process at the end.

Diagrams of each of these two types of technology appear below.

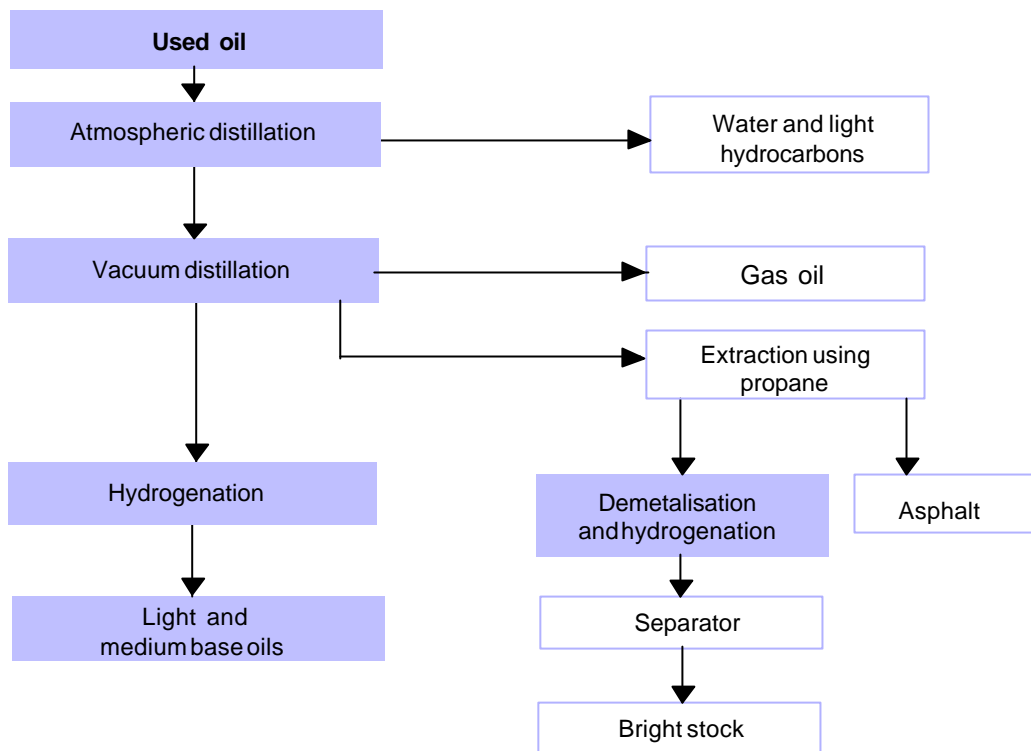


Figure 10. Diagram of IFP technology

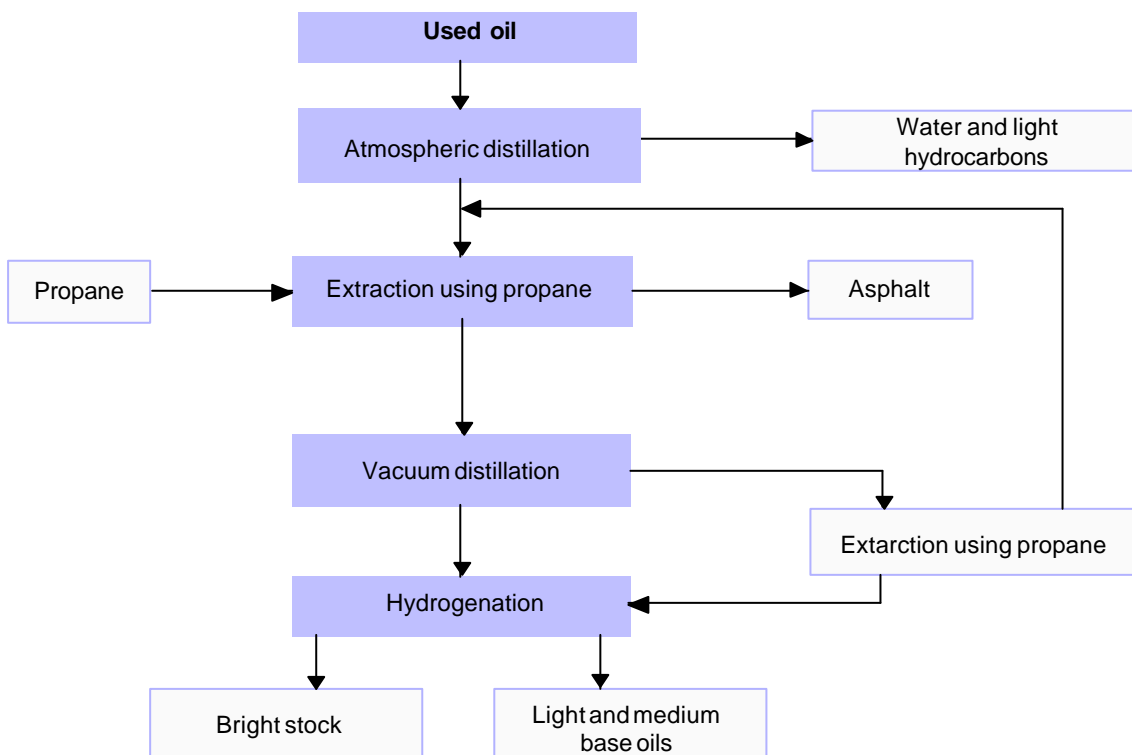


Figure 11. Diagram of Snamprogetti technology

UOP DCH technology

UOP DCH technology involves treating used oil with hydrogen gas at a certain temperature and an appropriate separation process to eliminate solids and metal elements. Halide solvents are directly destroyed during the process and high quality light hydrocarbons are obtained through the use of oxygen. Hydrogenation is carried out in a reactor with a catalytic bed.

The basic steps of the process are as follows:

1. Hydrogenation.
2. Separation of solids and metals.
3. Catalytic reactor.
4. Chemical neutralisation of acid gases.
5. Separation of the aqueous phase.
6. Vacuum distillation and fractionation.

This process enables potentially hazardous used oils to be recycled through an economic system that performs well and good quality products are obtained.

This technology has been tested in pilot plants and has so far not been commercially developed.

A diagram of the process is given below.

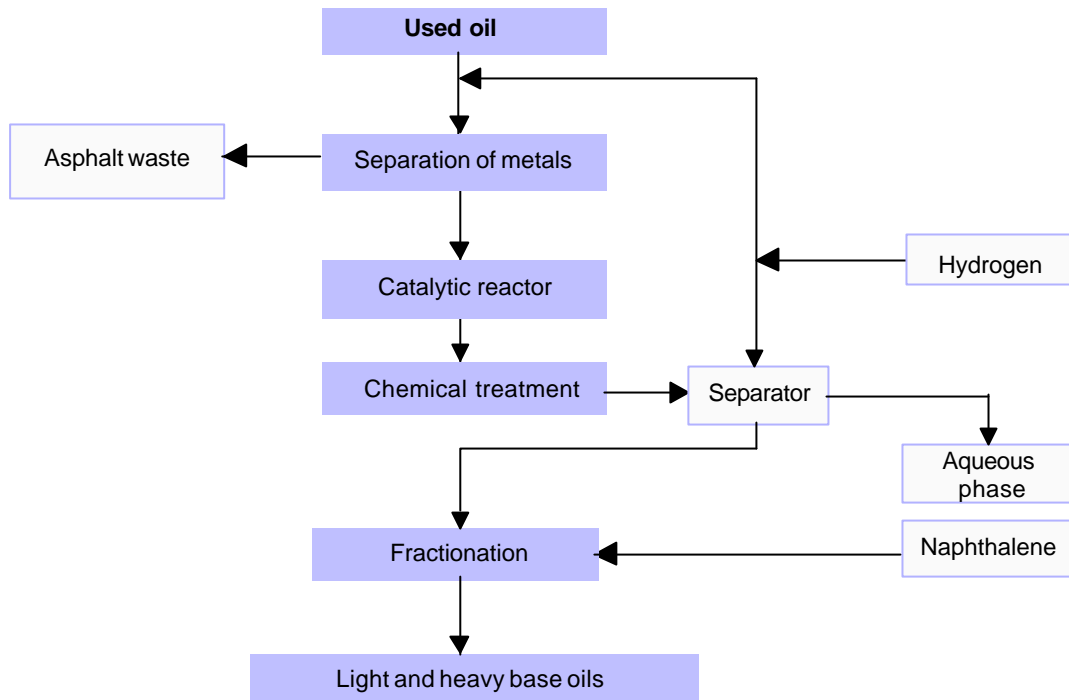


Figure 12. Diagram of UOP DCH technology

1.4.4 Vacuum distillation and earth treatment processes

Viscolube technology

Viscolube technology, also known as TDA (Thermal Deasphalting) is based on the use of propane, followed by vacuum distillation and an end earth treatment.

The basic steps of the process are as follows:

1. Distillation: the separating out of water and light compounds.
2. Vacuum distillation (TDA column) and fractionation: organo-metallic compounds and asphaltic minerals are separated out in this stage, and three base oil cuts are fractionated.
3. TCT (thermal clay treatment): stage where the characteristics of the three base oil fractions separated out in the previous stage are improved.
4. Filtration under pressure.

The advantages of this technology are the low cost of investment; the high quality base oils fractions that are obtained; the low cost of installation maintenance; it is a very clean process environmentally speaking; and the vacuum distillation stage does not require very low pressures. Filtration under pressure allows treatment plants based on acid/earth technologies to be reconverted relatively easily.

The major drawbacks are the 72% efficiency (lower than that obtained using hydrogenation technologies), oils with a PCB higher than 25 ppm are not accepted, and there can be problems afterwards in treating the earth used during the process

The first plant to use this technology was put into operation in Pieve Fissiraga (Milan, Italy) in 1992, and two others have been set up since then in Poland (1994) and Italy (1995).

A diagram of the process appears below.

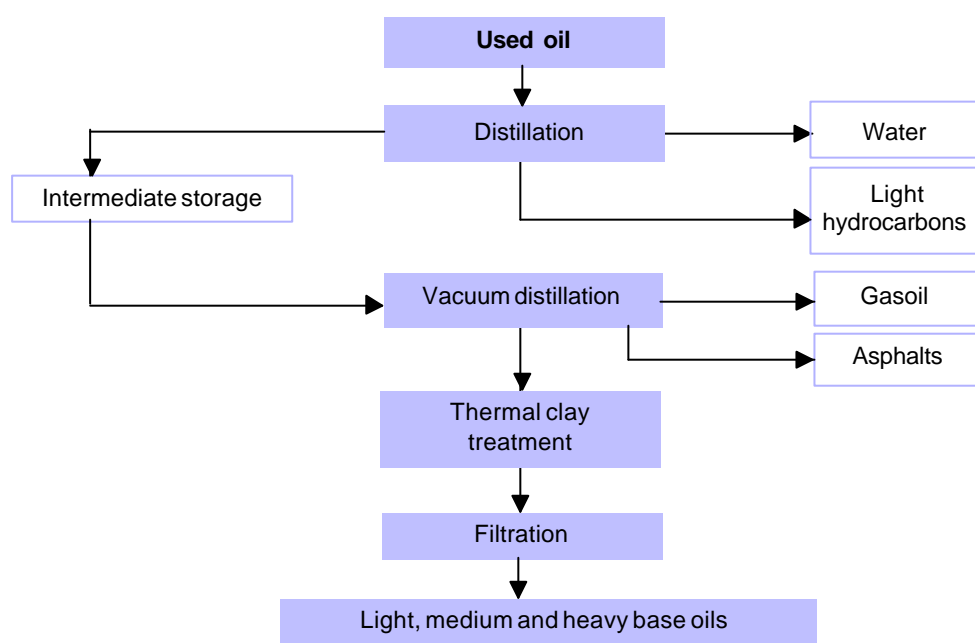


Figure 13. Diagram of Viscoluble technology

RTI technology

RTI technology was the first to use the cyclone-type vacuum distillation column and can work up to 20 mm Hg. Oil is injected at high speed and the oils that are obtained are subjected to a cleaning treatment using earth and then filtered in a filter press.

The basic steps of this process are as follows:

1. Separating the water: stage where the used oil is dehydrated and heated.
2. Atmospheric distillation: stage where aqueous emulsions and combustible fractions are eliminated.
3. Vacuum distillation: stage where medium combustible fractions are vaporised. Distillation occurs at 100 mm Hg.
4. Earth treatment: refining stage where the characteristics of the fractions that have been obtained are improved.
5. Vacuum distillation: stage of distillation in cyclon columns at a pressure of 20 mm Hg. The oil is injected at high speed, which generates a centrifugal force that helps the additives and pollutants that are still in the used oil fraction to separate out.
6. Earth treatment: oil obtained in the previous stage is mixed together with diatomaceous earth and activated clay, and then subjected to filtering.

This process also enables plants that are based on acid/earth technologies to be transformed.

Interline technology

Interline technology is one of the most recent innovations in vacuum distillation and earth treatment technologies. The chemical treatment is here replaced by extraction using propane during the initial stages, and at room temperature.

The basic steps of this process are as follows:

1. Extraction using solvents.
2. Separation of the oil.
3. Atmospheric distillation.
4. Vacuum distillation.
5. Filtering using earth treatment.

This process is interesting from the economic point of view because it eliminates thin film distillation and the need for hydrogenation. Both investment and maintenance costs are low.

The drawbacks include the fact that this technology does not accept oils containing PCBs, and the chlorine content of the used oil cannot exceed 1,000 ppm. Eliminating the earth from the end filtering can also cause problems.

There is one plant that uses this technology in Salt Lake City (USA), and another was put into operation in Stoke-on-Trent (England) in 1996. There is another in Sandy (Utah), and one is in the assembly stage in Seoul (Korea).

Rose - Kellog technology

This technology permits the treatment of used engine oils mixed with lubricating grease, provided that the content of the latter does not exceed 5%. Grease is made up of 85% of oil.

The process consists of extraction with propane in two stages. In the first stage, asphaltenes are removed by subjecting the oil to a specific temperature and pressure. In the second stage, the solution of oil and solvent is subjected to a supercritical temperature and pressure that facilitates the separating out of the oil and solvent, which can then be recovered and reused in the cycle.

The basic steps of this technology are:

1. Extraction using a solvent.
 - Asphaltene separation
 - Oil separation
2. Vacuum distillation.
3. Hydrogenation.
4. Fractionation.

This technology enables important energy savings to be made, due to the recovery of the solvent in supercritical conditions. As with Interline technology, it also allows the oil content in grease to be recovered.

This technology is being used at the present time in Houston (Texas) by the M.W. Kellog Company.

1.4.5 Other technologies

Processes used in other technologies that cannot be included within the aforementioned groups are described below.

Entra technology

The Entra technology can be compared with the technologies described above in the vacuum distillation and earth treatment group because it also uses these procedures. The difference lies in the fact that the vacuum distillation is carried out in tubular reactors where the used oil gets converted into a vapour due to the rapid increase in temperature. The vapour is then subjected to fractionated condensation. The evaporation process is produced by injection of the oil at a constant speed and at a temperature of 400°C. The oil obtained in this stage is free of solid impurities, and metal and other elements.

In the next stage, the distilled oil is purified and decoloured. This is a treatment carried out at high temperature during which sodium is added, which combines with the chlorinated elements to produce sodium chloride.

An earth treatment is carried out afterwards to improve the colour whenever necessary.

The basic steps of this process are as follows:

1. Preliminary stage: a vacuum distillation stage at 130°C and 100 mm Hg. pressure. The stage involves the separating out of water and light elements.
2. Cleaning stage: distillation occurs at 400°C, when the metal compounds and other solid impurities separate out.
3. Decolourisation and purification stage: stage in which the visual appearance of the oils is improved, together with the separating out of the chlorinated compounds through the addition of sodium.

This is a high performance process in which control of the temperature is highly important for obtaining the required results. The process is considered to be clean technology according to the IACT (International Association for Clean Technology). The TÜV analysis (Technische Überwachungsverein) shows the total elimination of PCBs. The main drawback is the handling of sodium, which is complex and hazardous.

A pilot plant has been using this technology in Achern (Germany) since 1988.

Recyclon technology

This technology is based on the aforementioned Entra technology. The treatment is carried out with sodium and vacuum distillation.

The basic steps of these processes are as follows:

1. The mechanical separation of solids and free water.
2. Distillation to eliminate other impurities and water.
3. A chemical treatment carried out with the application of sodium in a reactor.
4. Distillation to separate out compounds with a low boiling point.
5. Separation by evaporation of the base oils from the waste.
6. Final distillation to obtain base oils of a different viscosity.

High quality base oils with good colour and odour characteristics are obtained with this technology. The waste generated during the process has a high calorific value and a low sulphur content, and is recirculated into the process as fuel. Neither earth treatment nor hydrogenation processes are required and it produces few emissions into the air.

Krupp Koppers supercritical technology

This technology uses gaseous hydrocarbons in supercritical conditions to treat used oils.

The process consists of the atmospheric distillation of used oil to eliminate water and light hydrocarbons. It is then combined with ethane and the mixture is subjected to supercritical conditions, which separates out the oil from the polluting substances. The mixture of oil and solvent is subjected to a distillation treatment to bring about their separation. The solvent is then reused in the process.

The basic steps of this process are as follows:

1. Pre-distillation.
2. Extraction.
3. Distillation.

In the case of regenerated oils that contain PCBs, a hydrogenation treatment can be applied. Base oils obtained with this technology have been successfully tested by MWB-B, CEC L-12-A-76, DIN 51361.

Vaxon technology

Vaxon technology, also known as VCFE (Vacuum Cyclon Flash Evaporator, developed in Denmark), uses cyclon evaporators that enable the dirt that accumulates to be easily cleaned. The technology combines the vacuum effect with a specially designed heating system.

The basic steps of this process are as follows:

1. Fractionated vacuum distillation: in this initial stage, water, light hydrocarbons, metal compounds and other bituminous elements are separated out. This stage takes place in four modules under different temperature and vacuum conditions, and base oils that are suitable for the following treatments are obtained in the last two modules.
2. Chemical treatment: the base oils from the previous stage are treated with potassium hydroxide, using temperature control and cleaner oil is obtained. Drying of the oil occurs during this stage.
3. Vacuum distillation: final stage of vacuum distillation to obtain a product that is appropriate for the needs and conditions of the market.

This technology enables base oils to be obtained that are suitable for the manufacturing of new engine oils and industrial lubricants. These are high quality oils that have been approved according to the strictest regulations that exist at the present time. In environmental terms, this is a clean technology because the waste that is generated in the process is recirculated in the same process.

There is one known plant using this technology at the present time. It is run by CATOR (Catalana de Tractament d'Olis Residuals, S.A.) in Catalonia (Spain).

CEA technology

CEA technology (French Atomic Energy Commission), which at the present time is going through its initial phases, consists of mixing used engine oil with carbon dioxide in supercritical conditions at temperatures between 40 and 80°C and a pressure of 150 bars. A ceramic ultrafiltration membrane is used in a later stage to extract the impurities.

1.4.6 Summary of the technologies

The technologies described above and the order in which the steps are carried out in each one appear below.

	Meinken	KTI	Mohawk	Berc-Niper	Prop	Safety Kleen	IFP	Snamprogetti	UOPDCH	Viscoluble	RTI	Interline	Rose Kellog	Entra	Recyclon	Vaxon	CEA
Atmospheric distillation	1	1	2	1		1	1	1		1	2	3					
Chemical pretreatment			1														
Desmetalisation					1												
Separation											1						
Solvent extraction				3			4	2,5				1	1				
Solvent recovery					4			3				2					
Acid-earth treatment	2									4							
Vacuum distillation		2	3	2	2		2	4	3	2	3,4	4	2	1,2	1	1	
Chemical treatment									2					3	2	2	
Hydrogenation		3	4	6	3	3	3,5	6	1	3			3				
Thin film distillation	3					2								4	3	3	
Fractionation		4	5	5	4				4				4				
Earth treatment				7								5					
Autoclave, ultrafiltration																	x

1.5 Energy recovery systems

Energy recovery is one of the possible ways of applying used oils and is the application that has traditionally been used the most.

However, given the current trend, there are other possible applications for used oils that in environmental terms are more recommendable that mainly involve less use of natural resources.

Within this group, a distinction can be made between the following:

- Use for obtaining heat: used oils have mostly been used this way, especially in cement works, refineries, etc.
- Use in cogeneration equipment to produce electricity: used as fuel in engines connected to generating equipment.

Uses such as these cannot really be considered technologies, and for this reason this point is not gone into. Nevertheless, these applications are referred to in the following section and one practical case is given of the use in cogeneration equipment.

1.6 Possibilities for exploiting used oils and separated products

The possibilities of reuse are limited to a small group of industrial activities where there must be a safe system of environmental management. Moreover, it is important to take into account that the water from the dehydration processes, the fuel that is recovered, all of the filters in the processes, etc., all need to be treated and eliminated in accordance with criteria that are respectful of the environment.

Details are given below of the possible applications for different products in different processes. In general, the products obtained by the different technologies are base oils, water, light and heavy hydrocarbons, bituminous compounds, and waste containing metals, chlorine, sulphur, lead, etc.

1.6.1 Direct application as fuel

Cement manufacturing works

High temperatures are required in the furnaces at cement manufacturing works in order to convert the raw materials into cement. These raw materials are highly alkaline. Polluting substances such as polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and heavy metals are destroyed in cement production furnaces, so they offer an ideal situation for energy recovery from used oils in conditions that are respectful of the environment.

From trials going on at the present time with spent oils being used as substitutes for conventional fuel, there is apparently no appreciable increase of particle emissions into the atmosphere, particularly of organic, dioxine, furan and other compounds.

The alkalinity of the raw materials neutralises compounds like sulphur oxide, nitrogen oxide and hydrogen chloride, and emissions of these gases into the atmosphere are therefore reduced. Other smaller particles are collected by electrostatic precipitation or filtering and then returned to the furnaces.

The resultant ash of incombustible compounds (heavy metals in the used oils) is subjected to encapsulation processes.

Heating fuel

This form of application is used in garages for vehicle repairs, iron works, etc.

The used oil is burned in heaters that are specially designed to use this type of fuel. This system does produce volatile metal emissions, mainly lead and chloride, into the atmosphere although lead emissions are considered to be minimal compared to other sources of emission.

Owing to the emissions that can occur with this system, it is an application that is not recommended from the environmental point of view.

1.6.2 Application as fuel following a simple treatment

A simple treatment here means a straightforward cleaning of the oil prior to it being used.

Fuel in plants producing bituminous conglomerates

After eliminating the water and sediments from the used oil, the oil can be used as a replacement for industrial gas oil in plants producing bituminous conglomerates.

From information that is available at the present time, metal emissions from these types of plants are higher than those from the aforementioned cement works but in the countries where they are used, they are often lower than the maximum emissions authorised by the regulations. The harmful elements (mainly metals) in these plants are fixed by calcareous rock and then encapsulated with bituminous compounds, which avoids the occurrence of leaching. The working combustion temperature is insufficient to destroy the PCBs.

With regard to acid emissions, the fact that calcareous rocks are used means that the acids are neutralised.

1.6.3 Application as fuel following a severe treatment

Severe treatments involve the extraction of water and vacuum distillation to obtain quality base oils at the end.

Vaxon technology produces good quality gas oil. Distillate waste that contains most of the metals can be used as components for asphaltic mixes.

1.6.4 Re-refining to obtain base oils

The ultimate objective of all of the applications in this group is the obtaining of base oils. These base oils are used in the manufacturing of new lubricating oils for engines, industrial processes, etc.

Using acid/earth technologies

These technologies lead to highly acid sludge being produced that has a very high content of polynuclear aromatic hydrocarbons, sulphuric acid and sulphur compounds, together with metals. Given the physical and chemical characteristics of this sludge, landfill disposal is out of the question. The main application, together with the earth from the process, is as a granulate in cement manufacturing works. This is a high cost application.

Another possible application is incineration although the cost due to the treatment of gases with caustic soda is high. The sludge can thus be treated albeit at a high cost to obtain sulphuric acid or sulphur dioxide.

The earth used in these technologies must be incinerated, with the same problem in as far as the production of gases is concerned.

Using vacuum distillation technologies

The vacuum distillation processes used by modern technologies give distilled products (base oils) with a metal content that is lower than 1 ppm.

All of the metals contained in the used oil are encountered in waste at the vacuum distillation stage. This waste can be combined in suitable proportions with other waste to obtain an applicable product such as natural bitumen.

Using vacuum distillation technologies with chemical or earth treatment

Base oils produced by chemical or earth treatment have a metal content that is lower than 1 ppm.

Spent earth and the resultant chemical compounds from the process can be incinerated in cement works furnaces.

Using vacuum distillation and hydrogenation technologies

Apart from the general emissions that are given off in the oil handling processes, the end treatment of the catalyst must be carried out by specialised companies.

Within this group, mention should be made of UOP DCH technology, where the general effluents have little effect on the environment. The products that are produced are base oils, gas and fuel oil with no chlorine and sulphur compounds, water that has a low chemical oxygen demand and is free of sulphates and organo-chlorate compounds and a heavy waste that is suitable for asphaltic mixtures.

1.6.5 Other applications

Use in refineries

Use in refineries consists of the recycling of used oil through its incorporation into the refining processes that are carried out in the refineries to obtain virgin base oils.

This possible use has been studied in pilot plants in France. By way of a suitable pretreatment that eliminates water and light hydrocarbons and reduces the organo-chlorate compound content, this application can be environmentally correct. All metals are encapsulated with asphalt, which reduces leaching considerably.

Even so, problems with corrosion in the treatment plants restricts the viability of these applications for the time being.

1.7 Economic aspects

Concerning the economic viability of the reuse and recycling of used oils sector, a distinction must be made between the collection and transportation of the oils and their possible applications in line with different technologies.

Experience shows, in general, that the collection and transportation of used oils is economically not very profitable. It should be borne in mind that production is characteristically very scattered and the volumes to be collected are small. For collection and transportation to be efficient¹, there must be a direct or indirect system of grants, subsidies or tax abatement to balance the operating-performance income statements of companies that are committed to collection and transportation. The separating of different qualities of used oil is important for the subsequent treatment of used oil, and this involves a higher cost that must be added to the collection system.

Income from the sale of products and the costs of obtaining these products are points to be taken into account when dealing with the economic viability of the refining plants that use used oil reuse and recycling technologies².

Income from the sale of products from treatment plants varies significantly according to various determining factors, including the price of petrol.

If, for example, the price of crude oil is high, the price of used oil can also rise, because industry that uses the oil to produce heat is willing to pay more for this oil. This price increase means that industries that recycle/re-refine used oil also have to pay more to use it as a raw material, which consequently leads to the increase in cost of the raw materials.

On the other hand, base oils that are produced by the aforementioned technologies in the same situation of a high price for crude oil go up in value due to the fact that the price of base oils from the initial refining process will be higher.

On the contrary, when the price of crude oil is low, economic viability is jeopardised due to the fact that used oil continues to be used to produce heat at a lower cost, and where used oils are used to produce re-refined base oils, the price of the re-refined base oils is low when competing with the base oils from the initial refining process.

The selling price of both untreated and treated used oils is influenced by the following aspects:

- Demand from the cement industry.
- Demand from re-refining and other treatment plants.
- The quality of the oil that is collected and the demand for each quality.
- The price of the other fuels.

¹Although it is not possible to generalise so each case must therefore be studied separately.

²Used oil treatment plants do not in general tend to be profitable either, so for this reason contributions through subsidies, tax abatements, etc. are considered to be necessary.

In terms of cost, the following figures for the treatment of used oils are given for guidance only³:

- The cost of collection and transportation: between 24 and 48€/t.
- The cost of separating out the water and sediments: between 18 and 70€/t.
- Cost of obtaining high quality fuel: between 85 and 150€/t.
- Cost of re-refining for obtaining base oil: between 108 and 168€/t.

From what has been mentioned above, it can be clearly seen that the economic viability of the sector depends a lot on the market conditions although this viability can be complex, even in conditions that are favourable. For this reason, grants and subsidies are given in different countries to stimulate the development of the sector.

1.8 Practical cases of the reuse of used oils

Several practical cases of the collection, transportation, technological application and reuse of used oils are described below.

1.8.1 The Catalan Waste Oil Treatment Company (CATOR, S.A.)

Introduction

The establishment of the CATOR company is a reflection of the Autonomous Government of Catalonia's awareness concerning issues involving the environment. Reduction at source and regeneration were established as being the best options for treating used oils, while other options such as incineration were rejected.

The CATOR treatment plant in Alcover (Tarragona) is a compact, highly efficient plant that does not generate new waste and complies with the strictest regulations with regard to the environment. The treatment capacity is 30,000 t/year, although up to 42.500 t/year can be processed.

³The costs that are given here are approximate and may vary significantly depending on the context.

The regeneration process

The Vaxon Regeneration Process technology is an integrated system for recycling and transforming used oil into base oil of the same quality as straight refined base oils for the manufacturing of all types of lubricant.

The Vaxon regeneration process is specially designed for refining used vehicle and spent industrial oils. It uses cyclon evaporators, vacuum separators with an oil circulation and heating system that have all been specially designed. Plants with these characteristics can be small and designed to be much more compact than traditional refineries.

Consideration also needs to be given in this process to the end treatment of the different distillates from the evaporators. The evaporators extract metals, carbon, ash, sediment and water from the distillates. The end chemical treatment corrects the chlorine, colour, acidity and oxidising compounds, and gives a base oil lubricant that has the same or higher quality characteristics than those of straight refined oil.

This process is highly respectful of the environment. All of the compounds that in the used oils are separated out and reused, some of them in the same process while the remaining ones are valorised on the market.

The CATOR process basically consists of:

1. Fractionated vacuum distillation: in this stage, all of the components in the used oils are totally separated out. This part of the process takes place in four distillation modules with different temperature and vacuum conditions.
 - A. This module works at 200°C and 0.5 bars/pressure. The separation of water and light hydrocarbons (the petrol type) occurs during this first distillation process, and the latter are subsequently used as fuel in the plant itself. The water is diverted to a wastewater treatment plant that has been specially designed to treat water with a high chemical oxygen demand. Water obtained from the wastewater treatment plant is suitable for use in the general services of the refinery and also in the refrigeration circuits. No disposal at all occurs outside of the plant.
 - B. Oil that has not been distilled in the first module passes to the second one, which works at a temperature of 280°C and a pressure of 75 mbars. During this stage, light gas oil and spindle oils are obtained, which are directed to the corresponding storage tanks.
 - C. Work in this stage is carried out at 310°C and the pressure drops to 15mbars. Products that have not been distilled in the previous stage are treated here. Base oils obtained in this module are equivalent to those known on the market as NS 100 and NS 150 (NS are neutral paraffin solvents), that are sent to the corresponding storage tanks.

D. Everything that has not been distilled in the previous modules reaches this stage, where the working temperature is 350°C and at a pressure of 5-10 mbars. Base oils equivalent to NS 150 and NS 330 are obtained and sent to the corresponding storage tanks. The undistilled compounds are bituminous products that contain polymers and heavy metals from the additives and wear of the engines and machines that have used the oil. Analyses that have been made of leaching show that the products are perfectly fixed to the substrates that are used. These bituminous products are stored in the corresponding tanks.

2. Chemical treatment: the different products that have been obtained are cleaned of impurities and metals and are treated one by one in a reactor at temperature and with a solution of potassium hydroxide to clean the oil again. The potassium water is then separated out and dried.
3. Vacuum distillation: the dry oil is subjected to an end distillation process at 340°C and a 10-mbar vacuum. This distillation enables the product to be precisely defined, and a quality to be obtained that is at least the same, and in some cases even higher than that of straight refined oils.

All oils obtained by this process are suitable for the manufacturing of both engine oils and industrial lubricants.

An outline of the process is given below.

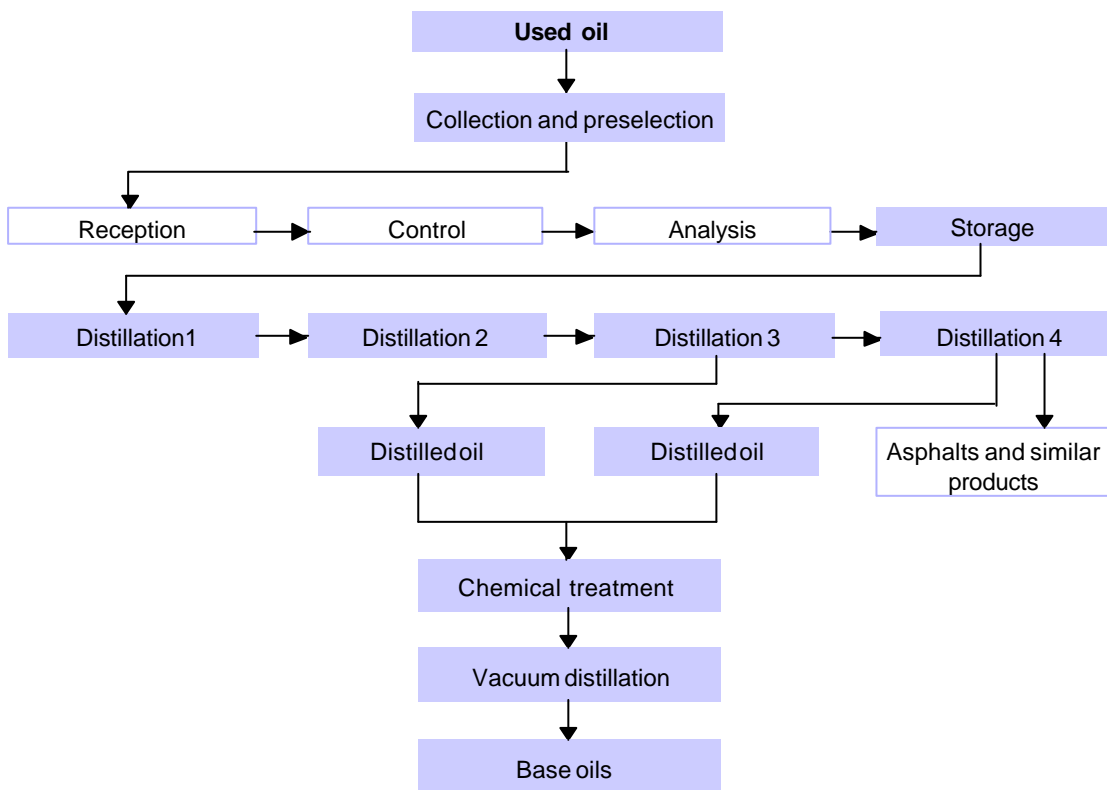


Figure 14. Diagram of the CATOR process

Environmental aspects of the process

The whole process begins after an intensive and thorough check of all of the batches of used oil that have been collected.

This process enables oils with a PCB content lower than 50 ppm and a chlorine content lower than 2,000 ppm to be treated in accordance with the current regulations in Catalonia.

No external disposal is envisaged at the CATOR treatment plant. The plant produces zero waste, and that includes rainwater that is liable to be polluted and which is treated in the plant's wastewater treatment plant.

Gases from the vacuum pumps, which consist mostly of non-condensable hydrocarbons, go to a purifier where they are cleaned and injected as fuel. There is no external flare.

The light hydrocarbons generated during the process and used as fuel in the plant itself receive the same treatment as the base oils to guarantee the permitted emission limits.

Gases from the storage tanks are treated with active carbon filters.

The potassium water from the chemical treatment is neutralised and the potassium salts that are obtained are used as fertiliser.

Valorisation of the products that are obtained

Base mineral oils, natural bitumen, light hydrocarbons, waters and potassium salts are all obtained in the CATOR treatment plant. The different applications for each of these products are as follows:

Valorisable products	Quantity	Application
Base mineral oils	50%-60% of the used oil	Manufacture of industrial and engine lubricants
Natural bitumen	17%-20% of the used oil	Waterproof fabrics and road asphalt
Light hydrocarbons	12%-13% of the used oil	Fuel used at the plant itself
Processing water	7%-15% of the used oil	Wastewater t. plant - refrigeration plant
Potassium salts	5% of the used oil	Fertilisers

Table 5. Products obtained in the Cator process

Together with the F.L. Iberia company (from the Magneti Marelli group, manufacturers of lubricants and a leading brand of engine oils) and Infineum (a leading company all around the world in the production and development of additives), the CATOR company has carried out the ECOROIL project which involves the use of base oils in the manufacture of industrial and engine lubricants.

The ECOROIL project, which consists of the manufacturing of engine oils (100% of the oil) from re-refined oils, has succeeded in obtaining products (Cator's REGENOIL oils) that exceed the tests for obtaining ACEA European and API American approval. It should be mentioned that other re-refined oils have been API approved whereas ACEA approval is much more restrictive and difficult to achieve.

1.8.2 The Aureca process

Introduction

The AURECA process is a system for using used oil that was developed and patented in 1992 by the Befesa Environment SA company. The process was developed jointly with the Department of Special Chemical Technologies at the Industrial Engineers College in Madrid and experimentation was carried out in a pilot plant in Huelva. At the present time, the Huelva plant has a 9 MW electrical production capacity and there are also two 10-MW plants in operation in Madrid and Cartagena and another 8-MW one in Valencia.

The process consists of converting used oil into a fuel that is then used in a diesel engine that powers a generator and eliminates the risks of heavy metal emissions by means of a physico-chemical treatment.

Description of the process

The Aureca process basically consists of three stages.

1. The object of the first stage is to recycle used oil and eliminate water, sediments and all of the pollutants, especially metals, that are harmful to the environment and for their use afterwards in a diesel engine. The used oils are collected, analysed and classified in this stage. The oil is subjected to a physico-chemical treatment in pressure and temperature conditions that are supplied by the flue gases. The process basically consists of adding

different compounds that react with the pollutant elements in the oil which are then precipitated. Water and precipitates are eliminated by decantation and centrifugation.

Three types of compounds are obtained during this stage:

- A. Oil, which is used as engine fuel.
 - B. An aqueous phase that is treated or sent to an authorised treatment plant.
 - C. Sludge from the precipitates that have been obtained, which is sent to an authorised plant for waste management.
2. The second stage consists of using the recycled oil as fuel in a diesel engine adapted to an electric generator. The oil has to comply with certain conditions to ensure that no damage is caused to the engine that it is to be used in.
 3. In the third stage, the heat that is generated in the diesel engine is used to produce a vapour and the combustion gases are purified. The flue gases from the engine combustion are sent to a heat exchanger that generates steam at a pressure of between 2 and 5 bars. The hot water and the steam that are generated are used for oil recycling. Purification of the combustion gases is carried out by washing with alkaline agents such as sodium or calcium hydroxide, which eliminates sulphur and nitrogen oxides, hydrochloric acid and other pollutant impurities until a gas is obtained that complies with emission regulations into the atmosphere.

1.8.3 The Ecolube process

The Ecolube process is a re-refining process based on Interline technology. It is basically an extraction technology that uses propane solvent and then vacuum distillation to obtain base oils.

The process consists of two basic units that are described below.

1. The first unit involves extraction by means of the use of a liquid propane solvent that is mixed together with the used oil and which then selectively and highly efficiently extracts the fundamental components of the oil. Water and asphaltic components are separated out. The propane and oil are separated by distillation. The propane is returned to the process and the oil enters the second unit.

2. In the second unit, the oil is subjected to vacuum distillation and the base oils that have been extracted are purified as they separate out from the combustible gas oil and heavy hydrocarbons that mix together with the asphaltic components produced in the first stage.

Process efficiency is 80% (base oils recovered) and the process does not generate toxic or hazardous waste.

The process also works at room temperature so that the oil-water emulsion breaks down without the need of temperature. This also means that de-emulsion additives that contaminate the extracted water are not needed either. The asphaltic compounds that are obtained contain inert metals and other pollutant compounds that can be used in the manufacturing of asphaltic by-products.

1.9 Proposals and conclusions

The conclusions that are given below are general and cannot be applied in the same way to all countries, given the particular circumstances in each.

It is interesting, however, to assess the general situation so that countries that are in the initial stages concerning the regulation of used oils can take advantage of the experience of countries with more knowledge in order to develop these industries or activities more advantageously.

Details of the conclusions are given below:

- Of the total quantity of used oil that could be collected and treated in line with the technologies mentioned above, around 50 – 60% is actually collected at the present time, which means that the rest (between 40 - 50%) is eliminated in an uncontrolled fashion on landfill sites by or uncontrolled dumping in sewage systems.
- Within the scope of the European Community, legislation on the collection and treatment of used oils is clear in that it encourages minimisation at source (reprocessing) and the re-refining of used oil. It is the responsibility of each State to apply strict policies that comply with the current Regulations.

According to currently available data, the final destination of used oils is as follows:

- Direct combustion: 32%
- Re-refining into base oils: 32%
- Used as industrial fuel: 25%
- Reprocessing in the same process: 11%

Less than 50% of used oil is thus assigned to actions (the re-refining of base oils, and reprocessing in the same process) that lead to smaller waste flows reaching the environment.

- Data are not available for the final destination of used oils in countries that are not in the group that abides by European regulations. It is believed, however, that the trend must be the same as that mentioned above, i.e. a proliferation of uncontrolled dumping and applications mainly in combustion.
- It is important for there to be an official authority that provides the service of overall coordination and monitoring of the actions to be carried out and, if possible, to establish direct connections with the companies in charge of collection and transportation, given that the sources of production are very scattered and can lead to inappropriate actions occurring.
- The quality of the oil is important for optimising later treatment processes. Collection and transportation services must be organised in line with the types of oils for this improves the quality of the used oil to be treated.
- Used oils that are unseparated according to quality are only used as fuel, because they only have a low value. They can cause atmospheric pollution when they are burned as a result of their harmful compounds.
- Consideration must also be given to the fact that the aforementioned technologies and processes can be economically viable or not according to external factors which are stated above. The inclusion of collection and transportation of used oils can jeopardise to a great extent the overall economic viability of the process. For the time being, therefore, grants in the sector are essential for the activity to be attractive to the industrial system in the different countries.
- Likewise, the structure of a subsector that specialises in the treatment of used oils requires modern logistics infrastructures, either by rail or road, because one of the major drawbacks is the large number of production sources that usually produce small quantities.

- Technologies for exploiting used mineral oils are:
 - Re-refining, including hydrogenation, and severe treatment for reducing the polynuclear aromatic hydrocarbons content to an acceptable level.
 - Technologies for returning by-products to refineries as fuel oil mixtures with a low sulphur content.
 - Technologies that enable by-products to be used as fuel in asphalt manufacturing plants, provided that they respect the chlorine emission levels.
 - Direct incineration in cement works.
 - Re-refining with acid / earth technologies.
 - Technologies with applications for by-products like natural bitumen that entail a high risk of soil and air pollution.

Out of all of these, the most recommendable ones are those that permit the regeneration of used oil so that it can be used as base oil (re-refining technologies). It must be borne in mind, however, that some re-refining technologies (for example, the acid/earth treatment) use processes that generate waste flows that must be appropriately treated because of their characteristics.

- Base oils obtained with modern technologies succeed in achieving the levels of quality that the market demands, and they are competitive compared to virgin base oils. There is thus a growing trend as far as their use in engines is concerned⁴.
- Base oils obtained with technologies that involve the reduction of polynuclear aromatic hydrocarbons to acceptable levels have the same effects on health as virgin base oils.
- The use of re-refined base oils means a reduction in the quantity of virgin base oils to be produced, so there is also less need for petrol. There are studies that have shown that energy consumption in the production of virgin base oils is three times higher than that needed to obtain the same amount of re-refined oils.
- As for re-refining, four litres of re-refined engine oil produce the same quantity of lubricating oil as 168 litres of crude oil.

⁴This is the case with Daimler-Benz and Volkswagen, which use oils manufactured from re-refined oils

2.1 Introduction

Used vegetable oils are oils that have been used as raw material for cooking processes in restaurants, large canteen facilities, fried food establishments, catering, food industries, etc.

The lack of specific legislation on oils used for food purposes, together with the fact that the population in general produces used vegetable oils, means that most of these oils end up in the sewage system which causes problems in the purification of urban wastewater.

The mixture of oil and water forms an impermeable film that prevents oxygen from entering, which causes the death by asphyxia of the micro-organisms that purify the wastewaters. The efficiency of treatment installations is consequently reduced because more time and much more energy are needed to break down these oil slicks.

For this reason, it is necessary to set up a system for the selective collection and specific treatment of these oils with the idea of obtaining new products that can be used as raw materials in the manufacture of animal feedstuffs, paint, detergents, humectants, surfactants and biofuel and, at the same time, minimise the uncontrolled dumping of these oils.

Interest in the collection and treatment of used vegetable oils is based on the following points:

- **Protecting the environment:**
 - Improve the purification capacity of sewage systems.
 - Reduce waste production in wastewater treatment plants.
 - Reduce the dumping of oil in the natural environment.
 - Improve the functioning of aeration tanks.

- **The financial economics of community sewer systems:**
 - Functioning of the network.
 - Functioning of the wastewater treatment plant.

The companies that are exploiting the largest quantity of recycled food oils at the present time specialise in the manufacturing of feedstuffs for animals and in the synthesis of esters that are used as biofuel, for obtaining aromas, etc.

Concerning the process of degradation of used vegetable oils, the quality of an edible oil is assessed according to the degree of acidity, its stability, the peroxide concentration and its fatty acid composition.

There is an increase in the polar compounds and in the peroxide and free fatty acid concentration during the frying process, which reduces the product's stability and increases its acidity. There is no variation, however, in the fatty acid composition.

Processes and technologies for regenerating used vegetable oils to be exploited as animal feedstuffs need to direct their efforts towards obtaining regenerated oils with the following characteristics:

- Polar part content: 5-15%
- Stability: comparable to that of an edible oil.
- Acidity: lower than 5% (lower than 1% for refined oils).
- Peroxide concentration: lower than 1 meqO₂/kg.

2.2 Collection logistics

The collection logistics of used vegetable oils is conditioned by the entities that generate them because the largest quantity is produced in the commercial and traditional restaurant industry, hotels, fast-food chains and industrial kitchens, whereas community restaurants produce less due to the fact that the meals in some of them (hospitals, rest homes, etc.) are more restrictive given the people who normally stay in these centres. On the other hand, used vegetable oils that come from domestic use are smaller in quantity than those originating from large commercial restaurants. Moreover, the quality of used oils varies according to the type of centre that they originate from.

As for logistics, the collection process is divided into two stages:

- Collection and storage by the centres that have used the vegetable oils in their activity.
- Collection and transportation of the oils to the companies in charge of treating them.

While regulations do exist in some countries that compel restaurants to renew vegetable oils that have been used, generally speaking it can be said that there are no regulations on the management of these oils, although initiatives are beginning to appear in this respect.

As a general rule, used vegetable oils are stored in metal or plastic containers with a lid to prevent contamination by other particles or compounds.

One of the factors that is of vital importance in the collection process is the separating of the used oils because this contributes to making the tasks of recycling easier and, at the same time, the products that are obtained are of a higher quality.

Companies that specialise in collection must be recognised by government authorities, which must grant the relevant authorisations for carrying out this activity.

In countries where a system of authorisation exists, these companies often sign contracts with the establishments that generate these by-products and where it is the establishment itself that is responsible up until the time of delivery for both the waste and the containers provided for its storage. These containers are only to be used for the storage of vegetable oils and fats originating in the preparation of food products. At the same time, they also formally agree to keep the containers closed and take every precaution to prevent them from being mixed with other waste.

Once the used oils have been collected and taken to the treatment centre, prior to the draining process, the oils go through a preselection process in line with their physico-chemical characteristics (colour, acidity and peroxides). The highest quality products are those that fulfil the following requisites:

- Colour < 37 FAC
- acid index <10 °
- peroxide value < 15 meq/Kg

After these checks, the oils are subjected to the treatment process.

2.3 The preliminary treatment of used vegetable oils

The preliminary treatment process of used vegetable oils involves their recovery through collection from the establishments where they are generated and then transportation to the treatment centres.

This recovery permits:

- 60 % of the oils obtained in the recycling process to be used in the production of feedstuff. This percentage represents the highest quality oils that are recovered.
- The remaining 40 % is used in other industrial processes, such as to obtain biofuels, surfactants, paints, etc. The percentage represents the lower quality oils that are recovered.

The treatment process basically consists of:

- **Filtration:** to eliminate the thicker materials, followed by a hot water treatment to finally clarify the oil being treated.
- **Decantation and filtration:** the oils and fats that have been collected are separated out from the water and other impurities, they are pumped out and they then undergo a second filtration process, followed by decantation again. In each decantation, the oils separate out and become purer each time.
- **Purification:** The dirtiest oil is circulated through a reactor at temperature that is also subjected to continuous agitation at the same time, which causes any water that is mixed with the oil to evaporate. This water is eliminated through a condenser.

A diagram of the process appears below.

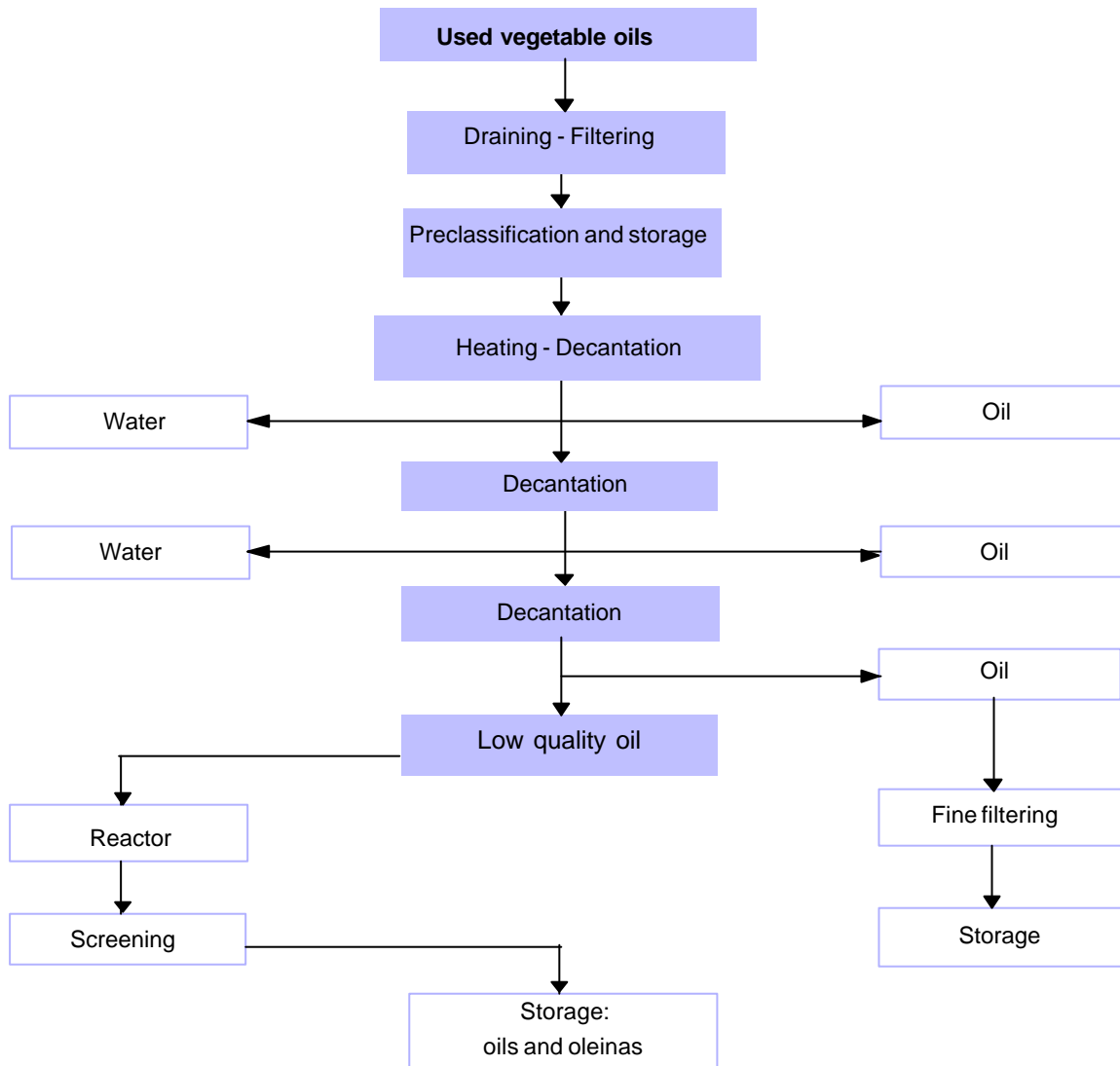


Figure 15. Diagram of the preliminary treatment of used vegetable oils

2.4 The possibilities of using recycled vegetable oils

Ways for using recycled vegetable oils are directed towards obtaining products that are used in the industrial field, with the purpose of minimising the expense of natural resources that are available.

2.4.1 Application in the production of animal feedstuff⁵

Oils subjected to the aforementioned preliminary treatment (point 4.3) are already being used in animal feedstuff. However, the process that is described below enables large quantities of used vegetable oil to be treated, and produces an oil with a higher utility level for use in animal feedstuff. The process is in the experimental stage and has been tested in a pilot plant.

Two fractions are generated in the process:

- One fraction (70% of the used oil) complies with the specifications that correspond to oils for human foodstuff.
- The rest (30%) is the resultant waste from the process and is suitable for use as fuel.

The process allows for the recovery of the part of the oil that has not been subjected to transformation processes after use. This fraction is made up of triglycerides and unsaponifiable compounds and separation is carried out using carbonic acid gas as a solvent at temperatures between 40 – 60°C. The fact that the process takes place at low temperatures prevents chemical reactions that would otherwise lead to the formation of new chemical compounds.

Chemical analyses are carried out to characterise the oil in order to control the product entering and coming out of the process. These analyses establish the composition of the oil and whether contamination has occurred due to mixing with other oils.

Positive aspects of the refining method for used vegetable oils using carbonic acid gas include:

- Elimination of the polar compounds that are responsible for the toxicity of the used vegetable oils.
- Reduction of the possible contamination of used vegetable oils by used mineral oils.

The results in the following table are from trials that have been going on up to the present time:

⁵ At the time of going to press of this study, no regulation has been developed to limit the use of used oils in the production of animal feedstuff. Even so, this study contains other opportunities for reusing used oils that do not involve their introduction in the food chain (see sections 2.4.2. and 2.4.3).

	Acidity ¹	Peroxides ²	% polar parts ³
Used oil	16	0.2	25.7
Regenerated oil	0.5 - 0.7	0.1	10 - 13
Unused oil	< 0.2	0.1	7 - 15
Maximum values for food quality	0.1 - 2	0.1	25 - 27

¹ Acidity in % free fatty acids
² Peroxides in MeqO₂/Kg
³ % polar parts in mass

Table 6. Results obtained

2.4.2 Application in obtaining biodiesel

The obtaining of biodiesel from used vegetable oils is an emerging application that is being rapidly developed and is undergoing trials in different pilot plants in various countries, parallel to the obtaining and expansion of biodiesel from the cultivation of oleaginous plants.

The treatment process for used vegetable oils for use as biodiesel is based on a set of chemical reactions that are described below.

The general outline is as follows:

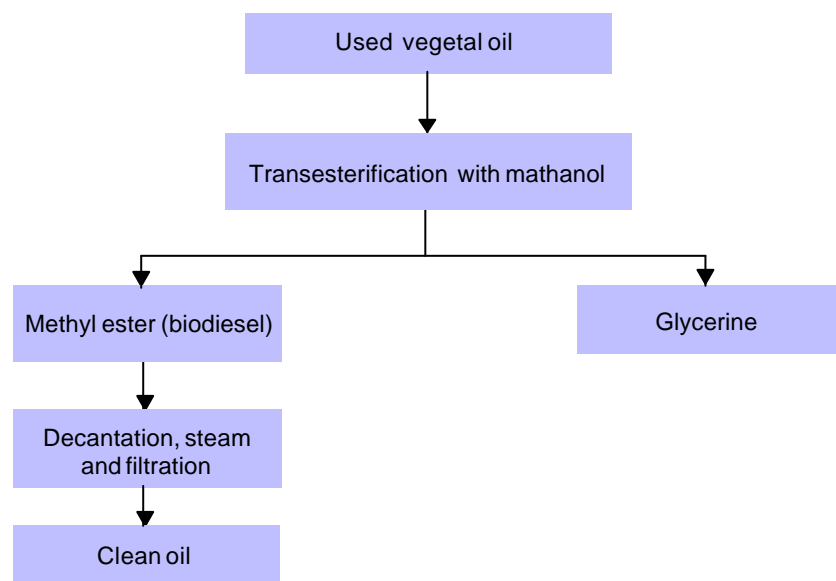


Figure 16. General outline for obtaining biodiesel

The chemical process that must occur for used vegetable oils to be used as biodiesel is transesterification, which consists of transforming the oil or fat with a catalyst (methanol) to release the methyl esters in the fatty acids and glycerine.

The chemical reaction of elements is as follows:



The reaction that takes place can be divided into the following stages:

- The triglycerides that form the constituent acids of the oils are transformed into methyl ester (biodiesel) with a glyceride mixture as a by-product.
- Once the biodiesel and glyceride have been separated by decantation, the residual methanol is removed from both by way of steam.
- The last step consists of separating out an insoluble biodiesel residue by filtration to achieve a clean, homogenous product.

The reaction occurs in an alkaline medium and at a low temperature (between 20 and 50°C). Processes with these characteristics can be carried out continuously or discontinuously and sodium hydroxide, potassium hydroxide or sodium methylate can be used as alkaline catalysts. Transesterification normally occurs in two stages, with the cleaning afterwards also being carried out in two stages.

In continuous processes, settling tanks are used to separate out the glycerine. In discontinuous processes, sedimentation deposits are used. The highly acid glycerine stage is separated out by acid treatment using sulphuric acid, acetic acid or phosphoric acid.



The three stages are separated by using a settling tank and the glycerine is reneutralised and evaporated. The fatty acid that is obtained can be subjected to esterification to optimise the efficiency of the process.

2.4.3 Other applications

Applications of used vegetable oils for obtaining biodiesel and in the production of animal feedstuff have been commented on in the previous sections. These are the two most important applications.

Within the context of the International Olive Oil Trade Fair that was held in Reus (Spain) in May last year and in the paper presented by Mr. Dominique Helaine, a representative of the European Interprofesional Association that brings together the national associations of companies that collect and/or treat oils for animal feedstuff (ERPA), other channels were revealed that permit the valorisation of used vegetable oils once they have been submitted to suitable treatments for obtaining an end product that is stable and that has a classified composition.

The possible uses that were mentioned are as follows:

- Use in the manufacturing of industrial lubricants: mainly in steel mills.
- Use in the manufacturing of surfactants: for the production of soap and detergent.
- Use in direct or mixed combustion: use of the calorific value of the oil provided that this is higher than 8,500 kcal/kg.

In summary, the main applications are those explained in sections 4.4.2 "Application in the production of animal feedstuff" and 4.4.1 "Application in obtaining biodiesel". The three applications defined in this section are used in the case of surpluses and cannot be considered as being applications that are widely used.

2.5 Economic aspects

The economic aspects of the used vegetable oils sector are intimately connected with the structure of the sector.

This is a basically unstructured sector and in most countries there is no legal framework regulating the activities of the agents involved, so that treatments that are carried out are highly variable and the quality of the resultant products is therefore diverse in many cases.

It must also be added that treatments applied to used vegetable oils require little investment in equipment and technology. As a result of this and the lack of any specific regulations, the recovery sector is sometimes made up of small companies that come and go very easily and that assign very few resources to improve the quality of the process and the end product for its use in other applications.

2.6 Proposals and conclusions

The following aspects are important concerning the general characteristics of the treatment and recycling of used vegetable oils sector:

- According to the information received and for the majority of the Mediterranean countries, there is a legal vacuum with regard to the treatment, recycling and use of used vegetable oils.
- This legal vacuum means that, apart from some exceptions, companies specialising in this subsector (mostly recycling for use in animal feedstuff) are technically unprepared, which means that in the majority of cases they could produce better quality products.
- The absence of legislation means that there is no uniformity of criteria between the different countries in terms of the characterisation of the parameters defining the quality or lack of quality of used vegetable oils. For example, whereas in some countries a polar component content of 25% is accepted, in others the maximum accepted quantity is 10%.
- The fact that there is an absence of regulations controlling the sector (there are no frameworks of action for carriers and waste managers of used vegetable oils) means that the sector is not transparent because it is difficult to account for the quantities that are collected and treated, the products that are obtained, the uses that are made, etc.
- There are associations and federations⁶ that draw together and take account of the interests of the entities that specialise in this economic sector. They are relatively new associations, however, and still have a long way to go in the sector being dealt with here.

⁶ ERPA: European association that groups the national associations for the collection and/or treatment of recycled food oil. Although they do not all come from the Mediterranean region, the national associations include Grofor (Germany), Brevo (Belgium) and Covhar (France), amongst others.

- Along the same lines, the drawing up of laws that regulate the sector must be carried out in such a way that takes account of the fact that the centres that generate used vegetable oils are highly fragmented (the same as with used mineral oils) and their production is normally low. This would apparently make it obligatory to provide economic aid for companies that are responsible for collection and transportation in order to offset the expense that this involves.

The following points are important concerning the application of used vegetable oils in the production de biodiesel:

- This is an alternative source of energy that is renewable and ecological. Its characteristics are similar to those of diesel oil although the emanations are more respectful of the environment.
- The physico-chemical characteristics of biodiesel prepared from used vegetable oils are very similar to those obtained with fresh vegetable oils (rapeseed, soya, sunflower, etc.).
- It can be applied as fuel in diesel engines. Its use as fuel in diesel engines and for any other use, either in its pure state or mixed with diesel oil, does not require large changes to be made to engines nor is it harmful to them.
- These products can be used as additives to vehicle diesel oils with the purpose of increasing their ketone value.
- They contain much less sulphur than diesel oil and the quantities are practically negligible, which means that they hardly produce sulphur oxide in combustion. They are therefore less aggressive and more respectful of the environment.
- They have a high inflammation and combustion temperature.
- The practically non-existent corrosion of copper by these products means that use is suitable in internal combustion engines and boilers.

It must be borne in mind that the final destination of the resultant products from the treatment and recycling of used vegetable oils so far has been animal feedstuff and, to a lesser degree, as biodiesel although important efforts have been made in this respect in recent years.

For this reason, the legal regulation of the sector is considered to be essential in order to mark out the possible applications for used vegetable oils in line with their characteristics, as well as taking into account that human health can be seriously affected by irregular actions by the waste managers of these oils as they are incorporated into the food chain via animal feedstuff.

Likewise, and based on the information that has been obtained, it would appear that studies are needed to characterise and provide more information on the sector in order to be able to then define the legal framework within which this activity must operate⁷.

⁷ The European Community has started an initiative along these lines with the purpose of making a complete bibliography available on the subject of used vegetable oils.

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ANNEXES

ANNEX I
Consultations

Annex I: Consultations

Entities and organisations consulted

A list of the different people who were consulted during the preparation of this study appears below.

Country	Contact person	Telephone	Fax	e-mail
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