<table>
<thead>
<tr>
<th>Code:</th>
<th>ENI45</th>
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<tbody>
<tr>
<td>Format:</td>
<td>Paperback</td>
</tr>
<tr>
<td>Indian Price:</td>
<td>1995</td>
</tr>
<tr>
<td>US Price:</td>
<td>150</td>
</tr>
<tr>
<td>Pages:</td>
<td>624</td>
</tr>
<tr>
<td>ISBN:</td>
<td>9789381039618</td>
</tr>
<tr>
<td>Publisher:</td>
<td>NIIR PROJECT CONSULTANCY SERVICES</td>
</tr>
</tbody>
</table>
Lubricants, greases and petrochemicals are most versatile on the Industrial Plateau now a day. The significance of Lubricants, Greases and specialty products in the day to day functioning of nearly every machine part, instrument, appliance & device cannot be over emphasized lubricants reduce friction & wear between rubbing parts, thereby enhancing their life. A lubricant is a substance introduced to reduce friction between moving surfaces. It may also have the function of transporting foreign particles. The property of reducing friction is known as lubricity.

The broad types of lubricating oils are as under; crankcase oils, gear oils, metal working oils, metal drawing oils, spindle and other textile oils, steam turbine oils. Synthetic lubricants have a higher viscosity index, but are less stable to oxidation. They are suitable for high temperature applications.

In the modern industrial year, greases have been increasingly employed to cope with a variety of difficult lubrication problems, particularly those where the liquid lubricant is not feasible. Greases are essentially solid or semi solid lubricants consisting of gelling or thickening agent in a liquid lubricant. Greases and lubricants are one of the important products derived from crude petroleum. Petroleum is formed by hydrocarbons (a hydrocarbon is a compound made up of carbon and hydrogen) with the addition of certain other substances, primarily sulphur. Petroleum in its natural form when first collected is usually named crude oil, and can be clear, green or black and may be either thin like gasoline or thick like tar. The principal product of petroleum refining are motor gasoline, aviation gasoline, kerosene, jet fuels, diesel fuels, lubricating oils and fuel oils. Considerable quantities of petroleum wax, bitumen, liquid petroleum gases (LPG), industrial naphtha and coke are also produced. Petrochemicals are chemicals made from petroleum (crude oil) and natural gas. Petroleum and natural gas are made up of hydrocarbon molecules, which are comprised of one or more carbon atoms, to which hydrogen atoms are attached. The Indian lubricants industry claims to be the sixth largest in the world. The petrochemical industry in India has been one of the fastest growing industries in the country. This industry also has immense importance in the growth of economy of the country and the growth and development of manufacturing industry as well.

Some of the fundamentals of the book are types of lubricating oils, crankcase oils, gear oils, metal working oils, metal drawing oils, spindle and other textile oils, steam turbine oils, synthetic lubricants, formulations and compounding of lubricants, additives for straight mineral oil gear lubricants, raw materials for lubricants, equipments for lubricants manufacture, reclamation of used lubricating oil, nature of contaminants in used lubricating oil, gravity methods of purification, metal forming and deforming lubricant, cutting oils, heat treatment oils, greases, sodium soap greases, lithium soap greases, aluminium soap greases, mixed soap greases, complex soap greases etc.

The objective of this book is to furnish comprehensive information about nearly all prominent types of lubricants, greases and petrochemicals. This book covers formulae, processes of various petroleum items. This book is an invaluable resource for entrepreneurs, existing units, professionals, institutions etc.

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Sample Chapter:
Raw Material for Lubricants

The basic raw materials for the manufacture of lubricants are: (1) Saponifiable fats and oils, (2) Saponifying agents, and (3) the base lubricating oil. But to improve the quality and grade of the manufactured lubricants, the following are added to make standard: (1) Performed soaps, (2) densifiers, (3) stabilizers, (4) chemical additives, (5) fillers, (6) dyes, and (7) perfumes. Densifiers may be employed in the presence or in the absence of soaps.

Saponifiable fats and oils. Theoretically any fat or oil may be converted into a lubricant or grease, but generally animal fats are used, because they are cheap, next comes vegetable oils, and other synthetic or non-synthetic materials: rosin-oil, naphthenic acids, sulphonic acids, synthetic fatty acids, montan wax, and wool grease. Wool grease is a good substitute for fats. Other substitutes are: tallow; olein, corn oil, repessed oil, cotton seed oil, etc.

While purchasing raw materials, the following points should be kept in mind.

(1) For the manufacture of lubricating greases with good storage stability or long life in service, fatty materials of low iodine value or containing little or no polyunsaturates are recommended. This points to animal fats or chemically or physically modified fats of low iodine value, for this purpose.

(2) For the same type of product, fatty acids are preferable to glycerides, or if the latter are used, they should constitute not over 20% of the total fatty acids.

(3) More translucent calcium base lubricating greases can be made by employing fatty acids rather than fats.

(4) The length of fiber in sodium base lubricating greases can be varied by a change in titer to the fatty ingredients. Thus a very long fibre results if vegetable oils or fats containing a large proportion of unsaturated fatty acids are employed.

(5) Conversely, short fiber sodium base lubricating greases result from the use of predominately saturated fatty acids such as hydrogenated tallow.

(6) Long chain fatty acids from soap with the greatest solubility. For this reason, if a sodium base lubricating grease of a smooth nature is desired, hydrogenated fish oil fatty acids should be used.

The following are the chief fats fatty acids used in this industry:

**Fatty Acids:** Lauric, Myristic, Myristoleic, Palmitic, Stearic, Oleic, Linoleic, Linolenic, Arachidic, Gadoleic, Arachidonic, Behenic, Clupanodonic.

**Fats:** Lard, Beef tallow, Hydrogenated grease, Herring oil, Menhden oil, Sardine Oil, Hydrogenated Herring, Hydrogenated Sardine.

**Oils:** Com, Castor, Coconut, Cotton seed, Linseed, Olive, Palm, Palm Kernal, Peanut, Repeseed, Soyabean, Tung, Hydrogenated, Castor. Fatty acids are preferable to fats, because they have the following advantages:

- Better control of fat composition.
- Greater ease of saponification.
- Completeness of saponification.
- Greater yield of anhydrous soap, 4.5% increase in soda products from 100 pounds of fatty acids over that from the same weight of fat.
- Greater uniformity of finished product.

**Test for Good Fatty Acid**

To 25 gms. Of liquid fatty acids in a clean beaker add 25 ml. Of alcohol, free, from indicator warming again if necessary to have the mixture liquid. To the above add, while stirring, 15 ml. Of a 50% solution of KOH in distilled water. A good fatty acid will show very slight colour change. A bad acid will be a dark reddish
Preformed Soaps

Nearly all manufacturers of lubricating greases employ preformed soaps to some extent. Since such soaps have a variety of other uses they are manufactured by a number of suppliers and stocked by most chemists. Soaps of almost any metal are available, but the manufacturer of lubricating grease is normally interested only in aluminium, calcium, barium, lithium sodium, lead and zinc soaps.

Advantages and the Use of Preformed Soaps

Compounding of lubricating greases is simplified by employing preformed soaps rather than manufacturing soaps from raw materials. Fewer ingredients need be stocked and weighed or measured. Since the saponification step is eliminated, the time required for the preparation of a batch of lubricating grease should be less when preformed soaps are employed than when fat and alkali are used. Moreover, elimination of the saponification step and the time consumed result in a lower heat demand.

Lubricating Oil

Lubricating oil constitutes the largest proportion of the total raw material required for the compounding of lubricating greases. Selection of the proper lubricating oil is important both from the standpoint of lubrication and of the structure and stability of the finished product.

Gravity of Lubricating Oils

The gravity of lubricating oil is a numerical value with an index of the weight of a measured volume of the product. Readings are made on a hydrometer. Tables are available for the conversion of such readings to specific gravity and weight per gallon.

The gravity of lubricating oils is interest to the manufacturer of lubricating greases because of its influence upon costs. Oils are purchased by the gallon and lubricating greases are sold by the pound. Therefore, if instead of purchasing an oil of 28 gravity, weighing 7.387 pounds per gallon; an oil of 20 gravity, weighing 7.778 pounds per gallon, is purchased, over 5% more weight would be secured for the same volume.

Pour Point of Lubricating Oil

Since the pour point of an oil indicates the temperature below which it is not possible to pour the fluid (Liquid) from a container, it is also indicative to a degree, of the temperature at which lubricating greases, made from such oil, can be forced through pipes or fittings. However, too much dependence cannot be placed upon the correlation of pour point and apparent viscosity at low temperatures.

Dyes for Colour

Most lubricating greases depend upon the natural colour of the mineral oils to provide colour to the finished product. But to make them attractive certain colouring is necessary and dyes are frequently employed for giving vivid colours, which otherwise (naturally) not possible.

Almost all the dyes used for colouring lubricating greases are oil soluble materials. Some colours are sold especially to provide fluorescent green or reds for mineral oils and such colours are also effective in lubricating greases. In selecting a dye, its stability towards light and heat as well as toward free alkali or fatty acids should be checked.

The predominating colours employed in lubricating greases are green, orange, red, and yellow. Less than a pound of dye should be required for 10,000 pounds of finished lubricant. It is well to dissolve the dye in warm oil before adding to the batch of lubricant.

Perfume

A small percentage of lubricating greases have perfumes added particularly in the case where an oil is of low grade and smells badly. Perfumes should be added when the product is as cool as possible. Perhaps less than a quarter of a pound of perfume base is sufficient for over 10,000 pounds of finished lubricant.

Fillers

A great variety of solids of various types are added to lubricating greases to give bulk, provide resistance to
removal of the lubricants, and, according to some workers, to increase the lubricating value. They are:

- Graphite, Asbestos, Mica, Talc, Vermiculite, Metal Oxides, Powdered metals, Metal Sulphides and similar Solids, Carbon Black, etc.

**Synthetic Lubricants**

- **Silicones**, Olefin Polymers, Polyakylene Glycols and Derivatives, Esters, Silicone Fluids, Chlorinated Compounds Halogeno-hydrocarbons, etc.

**Silicones** are organosilicon oxide polymers. Silicones are available as liquids, semi-liquids, and solids. One of their outstanding properties is that their viscosity is much less sensitive to temperature than that of mineral oils. For example, even a relatively insensitive mineral oil, when lowered in temperature from 120ºC to 25ºC., becomes about a thousand times more viscous, whereas the corresponding figure for a silicone fluid is only about seventeen times. Other properties are: good oxidation stability, very low pour point, and low volatility.

**Fluorosilicone:** By careful control of the polymerization conditions and the use of suitable end-blocking agents e.g. hexamethyl disiloxane, various degrees of polymerization can be gained to yield any desired viscosity of fluorosilicone oil. Fluorosilicone fluids are used mainly for defoaming of solvent based wash solutions or processes and lubrication. The chemical stability and heat stability of fluorosilicone oils, coupled with their good lubricity, accounts for their use as lubricating oils in chemical compressors and in vacuum pumps exposed to chemical fumes. Fluorosilicone fluids are also formulated with various thickeners to make grease-like sealing compounds and lubricating greases.

**Synthetic esters** are now produced in considerable variety. Esters, which are compounds of acids and alcohols, occur widely in nature, for example in fatty oils, but other types of ester, with special properties are now synthesized. One type (of which di-2-ethyl hexyl sebacate is an example) is characterized by good viscosity-temperature characteristics, together with better boundary properties and lower volatility than mineral oils of similar viscosity. Its chief advantage is the very low flammability.

**Polyalkylene glycols** are a group of non-hydrocarbon polymers produced in a wide range of viscosities, and in water-soluble and water-in-soluble qualities. They possess good temperature-viscosity characteristics and find application as hydraulic oils and in special greases.

**Halogeno-hydrocarbons** are hydrocarbons in which hydrogen atoms have been partly or wholly substituted by fluorine or chlorine atoms, or in some cases by a mixture of both. The outstanding merit of the liquid varieties is their high chemical and thermal stability-their demerits are poor viscosity-temperature characteristics and high volatility.

Synthetic lubricants also includes solids materials consisting of carbon and halogen, of which Teflon is the best known. Teflon and Fluon are commercial names for Polytetra fluoroethylene (PTEE), which gives remarkably low coefficients of friction as a metal lubricant, and is effective up to the usefully high temperature of about 320ºC. This is particularly valuable on a small scale when particularly low friction is desired, as for example instrument work. One method of use is as a surface coating in conjunction with a liquid, which may be oil or water.

**Equipments for Lubricants Manufacture**

**Introduction**

There are two chief processing methods: (1) Batch method, (2) Continuous method. These both methods have a number of steps in common. Therefore, the equipments required will be the same in many cases. Steps in processing are as follows, and the equipments are required to fulfill these processing needs:

- (1) Material storage and handling.
(2) Material measuring, either by weighing, or gauging.

(3) Saponification, which involves: (a) Mixing, (b) Heating. This step is eliminated if preformed soaps or bodying agents other than soaps are employed.

(4) Dispersion of bodying agent in the lubricating fluid. If this bodying agent is the soap formed in step 3, the dispersion may take place as the soap is formed. Normally, the dispersion will require additional heat, agitation, and perhaps shear.

(5) Dehydration, which may take place during saponification or at any other point in the processing. In some processes the final removal of water occurs by vacuum treatment of the finished or semi-finished product.

(6) Cooling of the soap dispersions. This may take place during agitation or in a static state. In the case of lubricants in which the bodying agent is dispersed by shear only, this step is not required.

(7) Milling.

(8) Removal of entrained air or of volatile materials. Only a portion of finished lubricant greases are subjected to such a step. Some dehydration may take place during the step.

(9) Handling finished product, which may include packaging and storage: No mention has been made of handling in connection with the equipment by which the various steps are carried on. Most of this handling will be by means or pipelines and pumps, therefore, certain notices may be kept in mind:

(1) Pipelines should preferably be welded and equipped with long radius bends.

(2) Apparatus in which heating is carried out in which rot material is handled should be well insulated.

(3) Individual drives for each piece of equipment should be provided.

(4) Flow of material into finished product and in a package should be downward and toward shipping or storage.
(i) Material Storage and handling
In most plants manufacturing lubricants, oil account for probably 85 to 90% of the total tonnage of raw materials. Fats and Fatty acids are next in volume. Raw materials, other than those above are seldom received or stored in bulk; but rather in the original containers. Raw material storage, therefore, involves both tank storage and warehouse storage. The space for tanks is devoted to fluid lubricants and to some type of fatty products, while the warehouse stock consists of a variety of materials in bags, cartons, and barrels.

(ii) Oil Storage and Handling
Oil handling in a lubricating grease plant is little different from that in a refinery, except that lesser amounts are handled. Most plants prefer vertical storage only, since horizontal tanks require more space for an equal gallonage than vertical tanks. No set correlation between the capacity of a plant and the amount of oil storage is possible. This will depend upon the location of plant and the variety of the products manufactured. Thus a lubricant plants adjacent to or located on a refinery property will probably require less oil storage.
In some cases oils must be blended before use. When this is done in storage tanks, the mixing is generally done by the use of dry air. Proportioning pumps can also be employed for preblending and delivering to storage. In humid climates condensation may occur inside storage tanks and as a preventative, some tanks use calcium chloride drying units on breathers of oil storage tanks. Other plants simply provide a ½ to 1 inch drain, flush with the bottom of the tank, so that water which collects can be drawn off.

(iii) Fat Storage and Handling

Fats, unless of high titer, are handled in either drums or tanks, both such materials are of steel, as a rule. It should also be kept in mind that fat held in storage and subjected to heat and moisture will tend to hydrolyze and if agitated, to oxidize or polymerize to some extent.

Pumps for handling melted fats may be either reciprocating or centrifugal types. Pipelines, valves, and pumps used to handle fatty acids, should be of stainless steel. Where a positive displacement pumps is required for such service, bronze end pumps are satisfactory. Duriron pumps will also serve, but of course consideration most be given to the fact the such metal is quite brittle.

(iv) Storage and Handling of Caustic Soda

Solution of solid caustic soda may be accomplished in an ordinary steel tank by placing the solid cakes on grids which will hold them off the bottom. The drum metal can either be stripped from the solid cake of caustic or number of slits can be made in the drum with an axe before placing in the tank. A centrifugal pump can be connected to the bottom of the tank, and after water has been added, circulation will aid solution.

Flake caustic soda may also be dissolved by suspending it in the upper part of a tank of water. Since the rate of solution of flake is much more rapid than that of solid caustic, a short period of mechanical agitation will suffice for complete solution. Large single additions of flake caustic may heat the water above the boiling point, and caustic solution may be thrown out of the tank by the sudden evolution of steam.

While handling caustic soda or its solutions workmen should be protected by gloves, goggles, and preferably cotton clothing. In case of accidental contact of caustic with any part of the body, the afflicted surface should be flushed with copious quantities of water.

50% liquid caustic soda may be unloaded by pumping, by gravity flow, or by air pressure, but pumping is the most commonly used method. Since the solidification point of this solution is 54% F it should be handled and stored at temperature above this. Ordinarily steel will serve for storage and handling of this grade of caustic soda. If the temperature of the caustic solution does not exceed 140°F, welded tanks will be satisfactory. Above this temperature caustic embrittlement may set in at the welds. For higher temperature it is usually more economical to construct riveted storage tanks.

Since 74% liquid caustic soda freezes at 144°F, special provision must be made for handling this grade. In view of this high solidification temperature, the general practice is to dilute the liquor to 50% strength or less before storing. The matter of dilution is not as simple as might appear, since the heat of dilution may raise the temperature of the solution to the point where caustic embrittlement of the steel storage tank occurs. With nickel-clad equipment such embrittlement does not occur. It is best to cool the diluted caustic solution to 150°F or below before placing it in storage. An alternative is to have a stock of diluted caustic soda from a previous shipment which has cooled naturally, into which the freshly diluted liquid can be mixed. Addition of a calculated amount of water to a tank car of 74% caustic soda may be made by adjusting a steam through a meter or by proportioning equipment. After unloading either grade of liquid caustic the pipelines and pump should either be blown free or washed with dilute caustic.

For handling caustic soda solutions, standard black iron pipe is satisfactory preferably equipped with flanged joints, since coupling tends to leak in service. Asbestos gaskets will resist hot caustic solution. If the lines are to be exposed to temperatures below the freezing point of the solution they should be traced with a steam line inside a common insulation. All iron stopcocks are preferable to valves for such service and if
high temperature are to be encountered, nickel-iron cocks are best. Brass or bronze valves or fittings should not be used for caustic soda solutions. If valves are desired, they should be of all-iron or of iron trimmed with nomel, nickel, or an alloy of this metal. All iron centrifugal pumps with extra deep stuffing glands and graphite-asbestos packings are satisfactory for handling caustic solutions. For high temperatures, nickel or monel shafts will give better service than iron.

Handling Packaged Raw Material

Packaged raw material received by a plant manufacturer of lubricant and greases will include bags, barrels, or cartons in loads. How such packages are stored and handled will of course depend upon the storage facilities of the plant in question. A better plant is to provide a weighing hopper, carried by suspended scales, into which the bag and carton material for a single charged are dumped.

Fillers generally go into the finishing kettles and may be handled by one of the above methods. Since addition of fillers will cause considerable dust, some plants provide a closed room where an oil slurry of materials such as graphite are prepared preliminary to addition to kettles. One supplier of graphite now offers graphites which have been coated with oil so that the dust problem is entirely eliminated.

Preformed soaps, such as aluminium stearate, are also likely to cause considerable dust when handled into vessels. Dust is a source of contamination in other products and increases the work of housekeeping but soap dust is also a fire or explosive hazard. Every effort should therefore be made to reduce such dust. By having all open kettles covered and kept under a slight suction, dusting can be reduced materially.

Equipment for Saponification

The most satisfactory equipment for saponification is that which will permit complete reaction of the ingredients in as short a time as possible. The main factors which influence the time of this reaction are probably temperature, concentration, presence of catalysts, and intimate contact of reacting ingredients.

Equipment for saponification therefore should be selected which will provide the best heat transfer possible and which will provide intimate contact of the ingredients.

Equipment for Batch Saponification at Atmospheric Pressure

Almost any kettle with an agitator and some heat will do some sort of job of saponification. The older kettles, some of which are still in use, were of riveted construction with agitators consisting of straight bars 21/2" or 3" wide, bolted to a shaft at about 18-inch intervals. These agitators were driven through bevel gears, by belts from a line shaft, tight and lose pulleys being provided. In some cases flat bottom vessels used, but modern kettles have either cone or dished bottoms. Fire-heated vessels are single walled.

The source of heat used to promote saponification varies. If direct fire is used, the fuel may be coke, fuel or gas. Steam is the most common source of heat, being used at the pressure available, which is normally 100 to 125 psi. An increasing number of saponification vessels are heated by circulation of hot oil through the jacket. Some installations have been made employing Dowthern as a heating medium. Where temperatures much above 300Â°F are desired, something other team should be employed for heating.

Since practically all open type saponification vessels are vertical, a shaft will extend vertically from the driving gear with probably a step bearing in the bottom of the vessel and occasionally an intermediate bearing between this bottom bearing and the one at the top. The most simple agitators design consists of flat, bars bolted or welded to the shaft at intervals of 15 to 24 inches. Such "paddles" can be made of 3" by 1/3" steel bars twisted so that they will give a down thrust. Since better mixing is accomplished by double motion agitators, many installations stimulate such motion by having a set of stationary blades and another set rotating. Simpler still, breaker bars can be welded to the shell at intervals intermediate to the paddles.

Scrapers to clean the inner shell of saponification vessel not only insure better heat transfer and thus a shorter reaction time, but also improve mixing. When fat, mineral oil, and alkali are first charged to a kettle...
and warmed, the mass is quite fluid, and as long as agitation takes place, the fluid next to the vessel wall will be displaced by turbulence and reasonable heat transfer will result. However, as soon as part of the fat or fatty acids is converted to soap, the mass becomes more viscous, and heat transfer will be retarded if the portion next to the kettle wall is not removed mechanically.

If the same vessel is also used to finish lubricating grease, scrapers have an additional advantage. Without such equipment heavy material would stick to the sides of the kettle and in later stages of the process might become loosened and then break into particles which would remain as lumps and never disperse. Some scrapers consist of a flat bar carried by the ends of the paddles. In some cases the bar is changed to an angle which is so mounted that it is on a swivel and actuated by a spring to hold it against the kettle wall. A saponification vessel can be used with a minimum of instrumentation, but a certain amount is desirable. Of course a gauge to indicate the pressure on the jacket should be installed. In addition a regulating valve is often provided on the steam inlet so that processing can be carried on at different pressures at various stages of manufacture. Most kettles used for saponification are provided with connections to a recording thermometer. The recorder may be used for one or several kettles, and an individual kettle may have thermocouples must be so placed that they will not interfere with the operation of the agitators or the scrapers and yet they should not become coated with lubricating grease or soap which would act as an insulator and thus prevent a true reading. If the bulb is inserted through the jacket of a vessel, clearance or air spacing should be provided so that the jacket temperature does not influence the reading. In some instances paddle blade near the bottom of the vessel will be slotted so that a bulb can be inserted in the space provided by the slot. In kettles which have a beaker bar attached to the inside of the shell, the bulb can be fastened to this bar so that a rigid location is provided. In still other cases the thermocouple is inserted through a bottom opening near to shaft. When a pump and circulating line are provided for a kettle, it is advantageous to have a thermocouple in such as line.

A ammeter for the operator to watch is desirable in connection with processing lubricating greases which pass through a very heavy stage. Ammeter readings can then be made of a part of the compounding instructions so that the operator will know when to shift to a different speed of agitation or to add oil.

Equipment for Dispersion of Thickening Agents

Agents employed for thickening lubricating fluids to produce lubricating grease structure can be divided into two classes, those which undergo phase changes and which require heat or a chemical method of dispersion and those which do not undergo phase changes with heat and which can be dispersed in lubricating fluids mechanically. The first class consists primarily of soaps and it remains to be demonstrated that they can successfully be mechanically dispersed in liquids to form lubrication greases. Perhaps if applied energy were sufficiently concentrated, so as to be converted to heat momentarily, soaps could be dispersed in lubricating fluids by such means.

Milling Equipment

Milling or shearing equipment is often used as an aid in soap dispersion. The simplest arrangement is to provide screens in the circulating line. One plant has such screens progressively smaller, starting with 10 mesh and finishing with 60 mesh. While this aids in soap dispersion, the screens must be screens is rather laborious procedure, modern practice is to use various types of mills for this purpose.

Manufacture of Lubricating Oils

As shown in Fig. 2 the conventional steps in lubricating oil manufacture, are pretreatment of the crude oil charge, as required, followed by distillation of the crude in two steps, deresining or deasphalting, dewaxing, solvent extraction, finishing and blending, including mixing various additives with the final lubricating oil. The recovery and refining of asphalts, resins and waxes are important to the overall economy of lubricating oil manufacture.
The chemical composition of lubricating oils is exceedingly complex the number of carbon atoms varying from approx. 20 to 70. Well refined lubricating oils contain very little olefinic unsaturation but do contain some aromatic unsaturation. The compounds contained in lubricating oils include paraffins, cycloparaffins and aromatics.

Waxes generally are paraffin compounds, both straight and branched and also contain 3-25% cycloparaffins, depending upon the source of crude oil.

Petroleum resins are hydrocarbons of very high molecular weight containing small amounts of oxygen, sulphur and nitrogen compounds, which can be found in bridge compounds or in ring compounds. The hydrocarbons include the paraffin, cycloparaffin and aromatic types in varying amounts and configurations. Asphalt is physically made up of brown solids called asphalters, i.e. asphalatic resins of high molecular weight viscous compounds with a degree of unsaturation and oils. The asphalters are believed to contain condensed aromatic ring compounds with oxygen, sulphur and nitrogen in ring compounds with oxygen, sulphur and nitrogen in ring compounds or in bridge positions.

Where (1) Atmospheric tower
(2) Vacuum tower
(3) Deresining

**Pretreatment of crude:** In order to remove inorganic salts from the crude oil charge to the crude distillation unit, chemical or electrostatic desalting is used. Salts in the crude oil cause fouling of heat exchangers, corrosion in the distillation units and increased coking of the furnaces.

**Distillation:** Crude oil after pretreatment, is charged to the atmospheric tower, where the crude oil is separated into light products. The bottoms of the atmospheric tower, to reduced crude, are charged to the vacuum tower.
The prime object in the manufacture of lubricating oil is the initial separation of the light products and the separation of wax distillate and cylinder stock without any decomposition or cracking of the lubricating fractions; thus a vacuum distillation unit is used to separate the wax distillate and cylinder stock at a lower temperature.

**Derasing or Deasphalting:** Asphalts, which contained in asphalter crudes can be constituted into different properties by further distillation or by air blowing. Resins occur in paraffin or low-asphaltic crudes. Propane is used as a solvent and at different temperatures and ratios causes asphalts or resins to separate from the oil due to a difference in solubility. This process requires a tower for separation of the oil and the asphalt or resin.

**Dewaxing:** Wax is probably the most troublesome product in the manufacture of lubricating oil. Its presence in lubricating oils prevents free movement at lower temperatures.
Usually, methyl ethyl ketone (MEK) and an aromatic solvent, such as toluene are used for dewaxing purposes. The MEK causes the wax in the oil to crystallize, and the toluene is used to dissolve the oil. The solvent mixture, at a carefully controlled temperature, is added in measured amounts at points in the chilling to produce proper crystallization of the wax. Both the wax solution and the oil solution are distilled for removal of solvent (to be reused) and to provide solvent free wax and oil. Thus the two products are a wax-free oil and an oil-free wax.

**Solvent Extraction**: To upgrade or improve the quality of neutral or bright stock, solvent extraction is carried out. It can be performed before or after dewaxing, but most solvent extraction is performed after wax removal in order to prevent any interference from wax in the charge oil. Solvents such as chlorex, nitrobenzene, phenol, benzene, and sulphur dioxide are used. The improved oil (or raffinate) and solvent are taken overhead from the treating tower, and unsaturated material of extract and solvent are removed from the bottom. Solvent is removed from both the raffinate and extract in recovery equipment and reused.

**Filtration**: Bauxite is the common filtering medium or absorbent for the removal of asphaltic and resinous undesirables or simply for light filtration or finishing. The bauxite is placed in a vertical steel tank, and the oil is permitted to gravitate through the bauxite in the filter. A screen in the bottom head of the filter prevents the bauxite from being removed from the filter with oil.

**Blending of additives**: Additive blending is done after bauxite filtration, since filtering will remove the additives. Additives are used according to the severity of the operating conditions for which the lubricating oil is intended and the quality of the base lubricating oil. Naturally, a high quality lubricating oil base requires fewer additives than a lower quality base. Fully formulated lubricants contain additives to boost their properties. Wise choice of additives can increase the biodegradability of the full formation.

**Reclamation of used Lubricating Oil**

Lubricating oil deteriorates, and becomes contaminated with foreign materials, in service. In circulating systems, where a quantity of oil is involved, it is desirable to maintain the oil as clean as possible to provide maximum lubrication efficiency, and to keep wear and damage of lubricated parts to a minimum. When the lubricating oil reaches the end of its life in the engine, what most probably happens is additive depletion. Because of the engine operation, these chemicals, slowly lose their effectiveness. This is the time to change the oil. This used oil can be re-refined for use. Technically speaking, re-refined oil can be used to replace some of the original base stocks. For some applications, in fact, they are better. But the usages depends heavily on the process used to manufacture the re-refined oil and so many other things like the chemical compatibility etc.

Reconditioning of a used oil may be accomplished by full flow, by pass, of batch methods or combination of these. In the full-flow system, the entire flow of oil form the main pressure line is continuously filtered. In the by-pass system, a fraction of the total of flow is continuously filtered and returned to the oil reservoir. In the bath system, as the name implies, all the oil is removed from the lubrication system and is reconditioned as a batch.

**Nature of Contaminants in Used Lubricating Oil**
Contaminants in a used oil may be divided into two classes:

1. Products resulting from chemical action within the system, including effects due to fuels; and
2. Foreign material which enter the system.

**Products resulting from chemical action within the system are as follows:**

(a) Carbon, and other products of partial decomposition of oil or of incomplete combustion of fuel;
(b) Oxidation products (which may be either soluble or insoluble in the oil), due to chemical action at high temperatures;
(c) Gummy product, both soluble and insoluble, resulting from polymerization (combining) of unsaturated
components in the oil; and
(d) Sulphur compounds, formed by sulphur in the oil or fuel.
Foreign materials may include some of the following:
(I) Dirt and dust from the air;
(II) Metal particles resulting from wear of operating parts of the machine, or left over from machining
operation during an overhaul;
(III) Foundry core-sand from castings;
(IV) Water condensed from air moisture or products of fuel combustion; and
(V) Fuel dilution.

Purification of Oils
Three basic methods of treating contaminated oils are used, both singly and in combinations:
(1) Gravity purification,
(2) Filteration, and
(3) Reclamation

Gravity Methods of Purification
Gravity methods which are based on the relative weights of clean oil and the contaminants to be separated
out, include the use of settling tanks and of centrifuges.

Settling Tank
In a settling tank method, a batch of dirty oil is allowed to stand for ten or more days in a tank, and insoluble
matter (including water), which is heavier than oil, settles to the bottom under the influence of gravity. The
tank should be free from vibration and the oil is undisturbed. Small particles and dispersed oxidation
products are not removed in this process. Best result are obtained when the oil is heated to the range of
120ºF to 160ºF reduce the oil viscosity, thus facilitating settling.
Drawbacks to the settling tank method of purification are : the time element, the space requirements for
tanks, extra oil charge for the engine during settling process, and the fact that impurities are only removed
periodically.
This method is adaptable to straight mineral oils, but is generally unsatisfactory with heavy duty additive-
type oils.

Centrifuge
A centrifuge works on the principle of separation by centrifugal force. This force, supplied by the high-speed
rotation of the bowl containing the contaminated oil, is several thousand times the force of gravity. As a
result, the centrifuge is much faster and more efficient than the settling tank. Substances having a higher
density than the oil, such as water ad heavy particles, are thrown out against the walls of the centrifuge with
greater force than the oil. As a result, a stable oil water leave by separate outlets, the oil discharge tube
being nearer the center. The sediment is removed from the walls by cleaning at regular intervals. For some
types of centrifuge; special arrangements are provided to prevent mixture of purified oil with incoming
contaminated oil, in the vicinity of the oil water boundary.

"Through-put" capacity is the maximum number of gallons of contaminated oil which can be sent through
the centrifuge each hour, with no regard for the degree of purification. The effective capacity of the
centrifuge, which is much more meaningful, is the rate at which contaminated oil may be processed to give
the desired degree of purification. This rate depends upon prevailing conditions and what is deemed to be a
desirable degree of purification in each case.

In so-called wet centrifuging, water is intentionally added to the entering stream of contaminated oil. This
added water may have a washing effect on the oil and a tendency to carry away more of the lighter solid, as
well as some acids which are more soluble in water than in oil.
A centrifuge is especially well adapted to a by-pass system, in which part of the oil being circulated is by-
passed through the centrifuge each cycle. Centrifuge is also suitable for the batch system of purification.

Filteration

Filters may be applied on full-flow, by pass or batch systems where there is sufficient pressure to overcome the internal resistance of the filter. There are three principal types of filters:

(1) Mechanical, (2) Absorption, (3) Adsorption. Where a filter is installed in the lubrication system of machinery, such as an engine, a relief valve is provided to cut off flow to the filter at a predetermined pressure. This is to protect the engine against loss of oil supply. Filters may be integral units composed of a filter element in a sealed container, in which the entire unit must be replaced. When blocked with filtered material, or they may be replaceable element type, wherein the container can be opened for the replacement of the used element.

Regenerating Process of Used Lubricating Oils

Lubricating oils are discarded after a specific period of use, to give engines longer life and better performance. These are either drained into rivers or burnt in air, causing pollution of air and water. Thus the re-refining of used lubricating oil is another way of tackling the disposal problem through reutilization. The 80-90% of used lubricating oils remain unchanged, these can be economically regenerated.

India depends mostly on the foreign market to meet her requirements of lubricants and greases. The deficit in indigenous availability of lube stock is fulfilled through import. As such, the import of base stock and lubricant technology is making the important product "Lubricant" costlier, with the passing of each day. The economic factor alone is the most critical factor for industry to recognize the conservation of lubricant as an important area.

Regeneration of lubricating oil, a practice in many oil rich countries, should be popularized as this will stop drainage of foreign exchange and conserve our existing stock.

Contaminants Present in Used Lubricating Oil

Contaminants present in used lubricating oils can be classified as:

(1) Volatile, (2) Oils soluble compounds, (3) Oil insoluble compounds. Unburnt fuel and water constitute volatile component while in use in the engines, lubricating oils from primary oxidation products which polymerize and finally converted to asphalts by pyrolysis. These constitute oil soluble compounds. The third category of compounds includes dust, metallic particles, soot, degraded additives; metal soaps, etc.

Principles of Used Lubricating Oil Regeneration

Re-refining and reclamation are the two procedures used in regeneration. Re-refining may involve the following steps:

(1) Settling and Dehydration.
(2) Acid treatment.
(3) Clay treatment of acid treated oil.
(4) Removal of clay by filtration.
(5) Distillation or fractionation.
(6) Blending with bright stock and incorporation of additives.

Re-refined oil compares well with the virgin oil. Reclamation, on the other hand, involves steps like settling, dehydration, removal of asphalts and other solid bodies. It does not remove diluents and certain oil degraded products.

Existing Process for Regeneration of Used Lubricating Oils

Processes employed in United States:

1. In the process followed at Mohawk Refinery Co. Newark N.J. a 10,000 gall. Dish bottomed tank is charged with feed stock which is heated to 82Â°C (dispersant filled oils require higher temp.) with 98% sulphuric acid and settled, leaving mainly saturated naphthenic and paraffinic molecules. After acid and settling treatment 82-84% of the feed remains. This is followed by heating to 316Â°C in the clay contact
step. In a flash chamber, clay and residue drop down while overheads are pulled down to reflux with a product steam joining the second tower. Here the lighter ends are gathered for process fueling, and the bottoms are recycled. Two lubricants products result a 350-SSU at 37.8°C stream and 110/120 SSN at 37.8°C light distillate.

2. S and R oil co. Houston Tex. re-refiner process (i) 300,000 gal./mo. Of crank case drainings. First the waste oil runs through a pipe still for dehydration. Next, acid treatment in air blown agitator is followed by clay containing at 204°C. Then filtration takes place using sweetland filters, rotary vacuum filters or even plate and frame presses. The final step is blotter pressing to remove any remaining clay.

3. Diamond Head Oil Refining Co. Keramey N.J. does not use acid in its re-refining processes but employs more severe clay treatment, coupled with higher temperatures.

4. A new re-refining method for lubricating oils is said to offer favourable economies and eliminate refiner's own water pollutants acid sludge and filter cake. The process has been developed at Villanova University. 

**The new process has five basic steps:**

1. After the moisture content is reduced to less than 0.1 percent by heating to 138°C the acidic contaminants are neutralized by sodium hydroxide or other undisclosed agents.
2. Dilution with light naphtha as a coagulant followed by solids removal.
3. Atmospheric distillation to recover the light naphtha for recycle.
4. Vacuum fractionation to recover product and split out No. 1 heating oil.
5. Clay treatment. The oil is heated with clay to 338°C for about an hour under a nitrogen blanket and with mechanical agitation. The mix is then cooled to 149°C and the clay is removed via a rotary vacuum filter. By products of the process are a carbonaceous material similar to carbon black and a potential rubber extender oil. Out of total lubricating market in U.S. reclaimed oil accounts for about 6%.

In the field or re-refining of used lubricating oils, acids treatment is being replaced by light hydrocarbon treatment (propane and naphtha) resulting in better yields and obviating the problem of the disposal of acid sludge.

Research is being carried out in different countries of the world to evolve further cheaper methods of used oil regeneration.

**Characteristics of a Used and Regenerated Heavy Duty Motor Oil.**

<table>
<thead>
<tr>
<th>Properties</th>
<th>Used Oil</th>
<th>Regenerated Oil</th>
</tr>
</thead>
<tbody>
<tr>
<td>Appearance</td>
<td>Dark</td>
<td>0.890</td>
</tr>
<tr>
<td>Density D420</td>
<td>0.892</td>
<td>0.890</td>
</tr>
<tr>
<td>Flash point, open cup °C</td>
<td>260</td>
<td>236</td>
</tr>
<tr>
<td>Kinematic viscosity, cs at 37.8°C</td>
<td>165.4</td>
<td>162.4</td>
</tr>
<tr>
<td>98.8°C</td>
<td>17.66</td>
<td>17.3</td>
</tr>
<tr>
<td>Viscosity Index</td>
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<td>H6.5</td>
</tr>
<tr>
<td>Acidity, mg KOH/gm.</td>
<td>3.0</td>
<td>----</td>
</tr>
<tr>
<td>Conradson carbon</td>
<td>0.805</td>
<td>0.26</td>
</tr>
<tr>
<td>Residue % wt.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
A flow diagram for a process on regeneration of used lubricating oil is given in figure : below 7.1 which gives complete idea.

Plants are said to be running on this process.

Process developed at the Regional Research Laboratories, Jorhat Assam (Indian Patent No. 127751) consists of the following steps.

1. Clay Treatment
   Used lubricating oil together with activated fuller’s earth and water is heated to suitable temperature in a closed vessel with constant stirring.

2. Filteration
   Oil from (step 1) is cooled and then passed through a filter press.

3. Distillation
   Filtered oil (step 2) is then subjected to vacuum distillation. Some portion of the distillate collected is discarded as heating oil. Distillation is continued upto a still temperature ensuring more or less complete recovery of the oil. Distillation residue comprises most of the spent additive and is discarded.
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