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1 INTRODUCTION

The project MARE (Eco-Innovation ECO/10/277237) aims at the investment and market exploitation of a new eco-innovative unit (MARE unit) for the processing and material recovery from waste oils and petroleum residues (WO&PR), reducing thus the consumption of raw materials and natural resources. The beneficiaries of the Project are CYCLON HELLAS S.A. and the Ecological Recycling Society.

The general goals of MARE project are:

- To promote an innovative, greener technique for the recovery of materials from waste oil and petroleum residues by evaporation.
- To minimize the market obstacles and barriers for a greener treatment and marketing of waste oil and petroleum residues by evaporation.
- To promote the sustainable use of natural resources, specifically lubricant oils and petroleum, with a life-cycle approach, in order to decouple environmental impact from economic growth by regenerating and recycling waste oils and petroleum residues into a "new product" rather than extracting further resources.
- To contribute to the effective implementation of the Thematic Strategy on Waste and Natural Resources; new Waste Framework Directive, the Hazardous Waste Directive and the POP Regulation.

The MARE project targets at the development of an eco-innovative process for material recovery from waste oils and petroleum residues. Those wastes represent a significant portion of the volume of organic liquid/hazardous wastes generated in Europe and therefore the environmental-friendly and cost-effective management of waste oils and petroleum residues may have important benefits for the environment.

The method is based on the separation at source of waste oils form used lube oils, and on the utilisation of novel evaporation techniques to minimise product polymerisation and maximise the quality of recovered secondary resources. To this end, in the framework of MARE project, it will be developed a processing unit for the transformation of Waste Lube Oils (WLO) and Petroleum Residues (PR) into water and useful final products capable to be used in certain applications.

2 THE UNIT

In the framework of MARE Project, it has been developed a unit that was based on a Thin Film Evaporator (TFE) for the processing of Waste Oil and Petroleum Waste (WO&PR) mixtures. These mixtures were contained different fractions of liquid state waste that derived from Waste Lube Oils (WLO), Petroleum Residues and Waste



from Ships (PRWS) and Petroleum Residues and Waste from Industries (PRWI). The TFE unit has been installed inside CYCLON's refinery and has operated at the August 2014 by receiving approximately 295 tonnes of liquid state WO&PR mixture.

MARE project aimed at the evolution of vacuum fractional distillation with hydro treatment through a thin film evaporator in order to process not only WLO but also WO&PR from other sources and in particular, from ships. The new wiped thin film evaporator was erected at CYCLON's regeneration refinery in Aspropyrgos, Greece. Having a designed treating capacity of 2.000 tons/year (300kg/h) of WO&PR, the thin film evaporator aimed at the separation of these streams in petroleum product readily available for further processing at refineries, petroleum sludge as a by-product that can be utilized in asphalt production and water. More specifically, the outputs of the new unit will be:

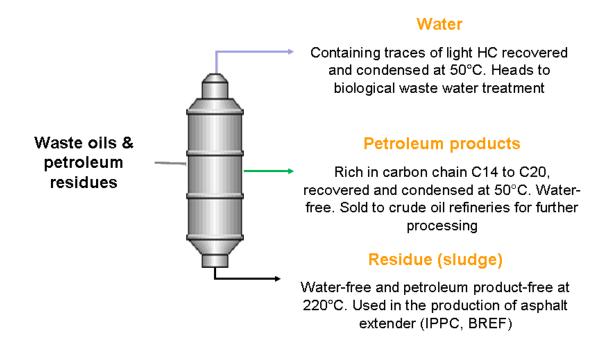
- 20% water containing traces of light hydrocarbons recovered and condensed at 50°C. This output will be adequate for biological waste water treatment and must be treated biologically as they may contain traces of petroleum oils, antifreezes, diluters and emulsions.
- ≥ 20% of petroleum product rich in carbon chain C14 to C20, recovered and condensed at 50°C. This product, free of water, solids and sludge will be supplied to crude oil refinery for further processing. Due to its high quality, no sewer processing is required by crude refiners.
- ▶ 60% water-free and petroleum product-free bottom residue (petroleum sludge) at approximately 220°C. The bottom residue will be mixed with the residue of used lube oil re-refining process for the later production of asphalt extender (IPPC, BREF on best available techniques for the waste treatment industries).

The major equipment and infrastructure required for the Processing unit consists of:

- Pre-heater (optional), for the heating demands of the process
- WTFE, for the recovery of volatile fractions (water, petroleum products). The evaporator is the core of the process and must be thin film
- Condensers for condensation of evaporated fractions (water, petroleum products)
- Feed and product pumps, vacuum pumps
- Instrumentation and control valves, process automation
- **Electrical panels**
- Diathermic oil heater for the heating demands of the new processing unit.



Figure 1: Mass Balance of Wiped Thin Film Evaporator



3 PROCESS DESCRIPTION

The thermo-chemical process of evaporation consists of the main unit which is the Thin Film Evaporator, the Hot Oil Boiler which is used for the pre-heating of the process medium and the provision of thermal energy to the main unit and the storage tanks for the amounts of feedstock and the end products (water, light to medium weight fractions and heavy weight residue). Furthermore, the supplementary equipment is consisting of the pumping systems which is operated in vacuum conditions (pressure below the atmospheric) and the collection vessels that are harvesting the liquid state end products prior to their storage at the respective tanks.

Oil recovery by evaporation is more effective than by other chemical or physical methods proposed by other technologies. Product yield is definitively higher and operation is less expensive because of lower utility consumption.

The process consists of the following steps each provided to selectively remove one or more categories of contaminants and recover discrete end products.

Pre-Treatment, including preheating additive injection and dehydration to remove water, gasoline, light contaminants (solvents, glycols, lighter organic) and separate heavy contaminants which remain in suspension in the oil.



- Gas-oil Removal, to separate gas-oil from the base oil for flash point specification and recovered a valuable gas-oil fraction.
- Vacuum distillation and Fractionation by Thin Film Evaporator, to separate light to medium weight petroleum fractions from heavy weight residue and produce two fractions. This operation is achieved by the combination of high vacuum distillation and thin film technology.
- Condensation of Light to Medium Petroleum Fractions where those fractions are condensed and directed to the respective collection vessel by gravity. The non condensed light weight fractions are directed to the CYCLON's refinery torch for immediate burning.

To this en the evaporation procedure is a type of distillation where light, medium and heavy weight fractions are divided and producing different outputs. Distillation is an attractive unit operation for the separation of components. Distillation achieves high selectivity from metals, heavy polymers, carbon, dust, and involves only heating and cooling, without use of chemical agents causing noxious by products.

Due to the high distillation temperature required and the thermally unstable nature of the contaminants this step is carried out in a special thin film evaporator under high vacuum conditions which are lowering the operational temperature levels.

4 THE OPERATION

The quantities of mixed WO&PR, from storage tanks, are pumped by the feed pump through the feed filters. Then they are preheated by the exhaust gases of a hot oil boiler. Finally, they are thrown into the evaporator from the upper part of the rotating drum. The mixture is sent to the outer surface of the rotating drum, where light hydrocarbons and water are vaporized.

Vapours from inner rotating drum overhead are containing mainly water and light weight petroleum fractions (gasoil). From the evaporator, they are routed to the off gas system and are directed to CYCLON's refinery torch for immediate burning.

Vacuum evaporation and fractionation is carried out under high vacuum and by means of thin film evaporator to recover light to medium fractions of petroleum residues, heavy weight fractions and aqueous effluent separately. All the impurities contained in the WO&PR feeding mixtures (i.e. metals, broken down additives, degradation products, etc.) are concentrated in the bottom residue.

In order to obtain maximum recovery rates, the evaporator operated in high vacuum pressure of approximately 30mbar and at an evaporation temperature of 75oC. It must be underlined that the high vacuum enables effective operation at relatively low temperatures. This is one of the main advantages of TFE. Low temperature conditions



are allowing the applications of less demanding materials that are maintaining cost of investment at low levels.

In general, TFE consists of a vertical cylindrical tube, enclosed in a heating jacket, with internal rotor which distributes a thin layer of oil on the heated wall, by means of wiper blades. The upper part of the tube is not jacketed and this acts as a separator. A rotor, driven by an external motor, has blades which extend nearly to the bottom of the tube, mounted so that there is a clearance of only about 1.3 mm between their tips and the inner surface of the tube.

The heating medium for the evaporation process is hot oil that is circulated from a hot oil boiler that utilizes natural gas for its operation.

The generated vapour flows upwards through the large open area within the evaporator. Any entrainment is separated in the bottom section of the evaporation column.

The vapour that is gathered from the top of the evaporator is washed and condensed in the condenser and then, according to its molecular weight, is fractionated into light and medium weight hydrocarbons. These reclaimed petroleum fractions can either used as gas for heat recovery or they can be directed to crude oil refineries for further processing. The product that is collected from the lower part of the evaporator (bottom) is a heavy weight residual petroleum fraction that can be applied as an asphalt extender. The respective quantities are collected in a separate vessel and thrown into a storage tank. The average temperature of the asphalt product during harvesting is approximately 220oC. Process sour water and condensed light hydrocarbons from vacuum systems are collected and sent to waste water treatment.

Development of this basic design has been devoted mainly to the modification of the blade system. An early alternative was the use of a hinged blade. In this type of unit the blade is forced on to the wall under centrifugal action, the thickness of the film being governed by a balance between this force and the hydrodynamic forces produced in the liquid film on which the blade rides.

5 ADVANTAGES OF THIN FILM EVAPORATOR

The thin film evaporator consists of a vertical cylindrical surface enclosed in a heating jacket and an internal rotor which distributes a thin layer of oil on the heated wall, by means of rotating blades. By the action of rotor (electrically driven) a high turbulence and back mixing occur in the thin layer of the oil film. The main features of the evaporator are:

Short residence time (in the order of magnitude of 10 seconds) by mechanical agitation of oil on the heat transfer surface



- Operation under high vacuum, which helps at reducing the operating temperature and thus the energy demand of the evaporation
- Excellent heat transfer as the process film is continuously agitated reducing thus the energy demand of the process
- Easy handling of viscous fluids, such as WO&PR, as wipers pump liquid down and create a thin film
- Easy handling of dirty and fouling fluids, as wipers continuously agitate the film and prevent the creation of deposits
- Efficient and regenerative cleaning of the contact surface
- Cracking and fouling problems are avoided by keeping low residence time, low wall temperature and high flow turbulence
- Light, medium and heavy weight petroleum fractions are recovered as distillates while heavy components consisting of additives, metals and degradation products are concentrated in the bottom as residue.

One of the main advantages of the TFE unit is its capability of fractioning certain petroleum fractions during evaporation and condensation without using additives such as clay and/or hydrogen. Furthermore, as the evaporation procedure takes place under extremely low pressures in vacuum conditions, the operational temperatures can be very low enabling the selection of less demanding material during construction. It must be noted that during the development of technologies for distillation of petroleum fractions, the major parameter that is affecting the investment cost is the selection of materials for the building up of the evaporator.

6 RESULTS FROM THE OPERATION OF THE UNIT

The output of the TFE unit for that period were 14% of water (41,3tn), 15% of light to medium weight petroleum fractions (44,25tn) and 71% of a heavy weight petroleum residue (209,45tn). The amounts of water were directed to CYCLON's waste water treatment plant while the quantities of light to medium petroleum fractions were sent to a crude oil refinery for further processing. As for the heavy weight petroleum residue, it can be utilized as asphalt extender to road works applications.

Based on the results of the monthly operation, the range of capacity of the TFE unit can reach up to 3.250 tonnes per year of mixtures that are containing WO&PR. Modular realization was the basic construction principle for a pre-defined capacity of TFE unit and consists in the use of prefabricated and pre-assembled skid mounted systems including equipments, instruments, electrical and structural material, predefined according to the basic sequence of the WO&PR processing procedures.



During the trial operation of TFE unit, they were processed mixtures of WO&PR with different concentrations in water content. In particular, when the main originating sources of WO&PR were oily waste containing petroleum fractions from industries, the water content was exceeded 20%. On the contrary, for WO&PR that were referred to a mixture of WLO and PRWS, the water quantities were slightly larger than 10%. In this framework, it was decided that the unit, during its full operational phase (August 2014) shall be use as feedstock WO&PR with water concentration approximately 14%. This decision was taken mainly due to the high energy consumption for processing mixtures with water content above 20% w/w. it must be noted that, for feedstock with relatively low water content, the unit can increase its processing capacity up to 500kgr/h, which meant that TFE can process approximately 200kgr/h above the designed processing capacity. Also, for water content 14% w/w, the recovery rate of light to medium weight hydrocarbons was reached the desired value (15% against 16,4% predicted).



Figure 2: Wiped Thin Film Evaporator during Operation