

Analysis of the Hexane Loss in a Vegetable Oil Extraction Unit

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ABSTRACT

The aim of this thesis is the study and analysis of the solvent losses in the oil extraction process of IBEROL, S.A. to achieve processes optimization and costs reduction. The operation of the equipment and the values of the operational variables were analyzed in detail and the comparison with literature values was performed whenever possible. In this way, it was possible to identify potential problems and anomalies and make proposals for improvement in order to reduce the hexane's losses and the associated costs. The steam network of the oil extraction unit was also analyzed. This work allowed to conclude that it is important to organize operating manuals for the equipment, defining the control points and the reference values for the different variables. Furthermore, improvements in the equipment's maintenance procedure and frequency should be implemented to prevent leakages, breakdowns and unplanned stops of the process. In fact, the analysis carried out indicates that when the process works continuously and stably the hexane emissions into the atmosphere remain below the maximum values allowed by legislation.

Key-Words: vegetable oils extraction, cost reduction, process optimization, hexane losses.

INTRODUCTION

With the markets' development it is becoming more and more important for businesses to adapt to more competitive environments and to stand out from competitors. Iberol is currently focused on the implementation of the *Kaizen* methodology, which is set out to achieve certain goals such as processes optimization and costs reduction. In this context, this work is focused on the study and analysis of the solvent losses in the oil extraction process.

Solvent extraction is a separation process in which the oil is transferred from the solid to the solvent. According to Dunford (2012) this is the most efficient process for oil extraction and, therefore, the most widely used in the industry. Due to its properties hexane is the most efficient and the most commonly used solvent.

n-Hexane

n-Hexane is used as a solvent to extract edible oils from seed and vegetable crops. Commercial grades of hexane are used as solvents for glues, varnishes, and inks, cleaning agent (degreaser) in the printing industry, liquid in low temperature thermometers. Other uses are as fuels and fuel additives, intermediates and laboratory chemicals.



JunYuanPetroleum Group is one of the leading n-Hexane manufacturers in the world.

CAS Number: 110-54-3
Molecular Weight: 86.18 g/mol
Appearance: Colorless liquid
Melting Point: -95 C
Boiling Point: 69 C
Density: 0.659 g/mL at 25 C

The hexane that are commonly used in organic chemistry are a mixture of various six carbon alkanes, not just n-hexane. The lower toxicity and flammability of heptane has led to a slow shift towards heptane in favor of hexane.



Our highest quality n-Hexane is delivered to our customers directly from our plants based in Shandong and Xinjiang, China.



Quality control is done using the the very latest testing techniques and procedures as provided by internationally updated databases around the globe.



We can deliver our n-Hexane solvents via logistics services using drums, Isotank or tanker vessels.

If the oil content of the seeds is greater than 30%, to optimize and increase the efficiency of solvent use, a mechanical extraction before solvent extraction is usually carried out. Regardless of the preparation steps used, the extraction process is quite effective allowing a high percentage of oil recovery and residual rates in the order of 0.7 to 0.5% of oil in the solids.

Seeds Preparation

The process begins with the removal of raw materials' impurities by sieving. Then, for soybeans, the seeds proceed to a cracking machine to break them into smaller dimensions, whereas for rapeseed the seeds are pre-heated up to about 40°C. Rapeseeds don't pass through the cracking process because the seeds are already extremely small. After the pre-heating, the rapeseeds are forwarded to the flaking stage to increase the specific surface area. Both soy and rapeseed pass through a thermal treatment consisting of heating the solids with indirect steam to 95 - 105°C.

Next, for soybeans, flaking occurs to increase the specific surface area which increase the efficiency of solvent extraction.

Finally, the flakes previously formed suffer an expansion after passing the expander (with live steam injection) to decrease the density of the solid material and increase the density of the medium (bulk), which facilitates solvent extraction and decrease the volume of the equipment's of the extraction respectively. In the case of rapeseed, due to high oil content, instead of expansion there is a pressing stage where 60 - 70% of the oil is removed.

Oil Extraction

The chemical oil extraction takes place in the extractor. In this equipment a solid bed is formed which is continuously immersed in miscella (oil mixed with hexane). The solvent passing through the material bed carries the oil from solids to the miscella. So, the principle of operation is to achieve successive equilibriums between the concentration of oil on the inside and outside of the solid. Hexane begins "pure" at the end of the extractor and during its movement in counter-current with solids, it gets increasingly concentrated in oil. The solids are discharged to be treated in DTD (Desolventizer-Toaster-Dryer) while miscella is treated in the distillation process.

In DTD occurs the removal of solvent by heating the solids, first indirectly with steam (pre-desolventization) and after with indirect and direct steam that passes through solids bed (desolventization). Toasting consists of a thermal treatment to guarantee the elimination of anti-nutritional factors and undesirable flavor substances for animal feed. To finish this step drying and cooling of the solid material occurs (Crown Iron Technologies LTD, 2012).

To remove the hexane from crude oil, the miscella is treated in a vacuum distillation process. The hexane evaporated during the distillation is condensed and separated from water in a decanter. The recovered hexane is then reused in the extractor. Before being sent to atmosphere the gases that do not

condensate pass through a final recovery process, where hexane is absorbed by a mineral oil at low temperature and desorbed at high temperature.

Solvent Use

Currently hexane is the most frequently used solvent at industrial level for oil extraction. However because of its characteristics it can cause industrial safety problems such as explosions and fires.

The excess of hexane in the air develops explosive and suffocating environment. It is also extremely harmful to workers when exposed to high concentrations for a long time. Inhaling it can cause irritation of the respiratory tract and more extreme respiratory problems. The long term exposure of skin to liquid hexane may cause cracks, burns and dermatitis. Leaks into the environment can affect the quality of water, contaminating groundwater and can be damaging to the local fauna and flora (Guarienti, Pine, Godoy, Evangelist & Lovato, 2012).

Because of the described features special care is necessary when using this solvent. Hence there are rules that regulate the solvent consumption. The maximum permissible values according DL No. 127/2013 of 30 August, for hexane released to atmosphere are 0.8 kg solvent/ton of soybean and 1 kg/ton of rapeseed processed.

PROCESS ANALYSIS

To do the study of hexane losses, firstly some research into the equipment and processes was required to develop the good operating practices. During this study the optimal values for the operation variables were established and compared with the real ones. The most important parts of the extraction process studied in this work were the Extractor and DTD.

Hexane Consumption in the Extractor

To monitor the quantity of solvent in circulation per ton of seed processed, i.e., the hexane consumption, the ratio between solvent mass and the mass of solids in the extractor is calculated. A low ratio can reflect an inefficient extraction as the existing hexane is not sufficient to remove the oil from the solids. While a high ratio of solvent results in a larger content in the hexane miscella leaving the extractor. The ratio should be 0.9 or 1.0 to soybean (Smith, 2000).

The calculated the ratio for Iberol's process for soybean turned out to be low. Increasing this ratio to 0.9, the hexane flow increases up to 6 ton/h which will lead to an increase of 1 ton/h of oil extracted. To calculate the increase in profits due to this extra oil recovery, it was necessary to calculate the extra costs in distillation (more hexane circulating, more steam needed for the evaporation) as well as the costs of increasing the hexane losses and of solids flow reduction.

To estimate the extra costs in distillation process, the heat necessary for evaporating the extra amount of hexane was calculated and thereafter the extra amount of steam required (at 10 bar). The extra heat needed is 2.0×10^6 kJ/h, so the steam flow should increase by about 998 kg/h.

The losses were estimated by applying the same proportion with total hexane flow. It was found that with increasing 6 ton/h of hexane, the losses would increase 0.15 kg hexane/ton seed.

Considering the price of steam of 30 €/ton, the price of the meal 350 €/ton, the price of hexane 800 €/ton and the price of oil of 750 €/ton, it was concluded that this extra amount of oil extracted could generate an extra profit of 362 €/h i.e. 8.700 €/day while processing soybeans.

Concentration Profiles in the Extractor

The concentration profiles of oil in miscella were established with the values resulting from analysis carried out on miscella samples taken in each miscella recirculating pump. The analysis of these profiles and their comparison with profiles presented in the literature made possible the establishment of a representative profile for each seed in the Iberol process.

This information is relevant because it allows to compare the results of the samples (made day-by-day) of miscella with the expectable values easily and quickly. With the knowledge gained by employees and factory responsables it is possible to establish actions to solve each type of problem, and thus contribute to procedures' standardization.

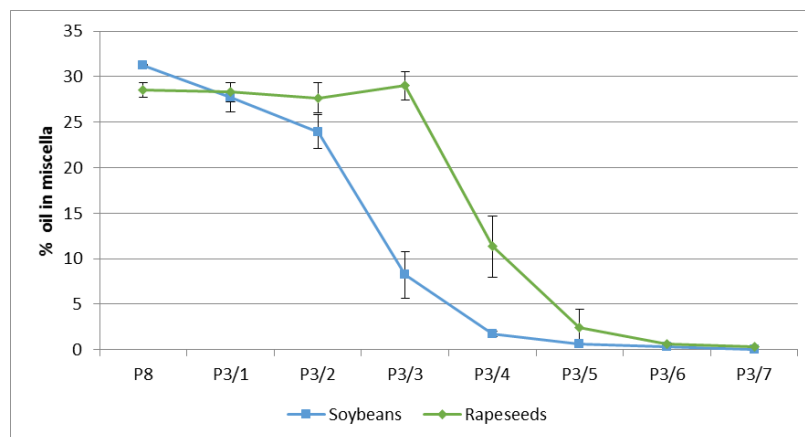


Figure 1 - Concentration profiles in the extractor for soybean and rapeseed oil extraction.

Miscella Recirculating System

The path of the miscella into the extractor is difficult to define, however it is very important to know and understand it in order to ensure the operation is in counter-current. This type of operation allows for greater savings in the amount of hexane that is being recirculated. To study this path, the average time the miscella takes to travel the full height of the bed (h_{bed}) was established by calculating the theoretical drop time. Considering the rotation velocity of the extractor, the distance between the miscella entry point

(represented by the shower) and the output point was assumed to be twice the horizontal distance traveled by this theoretical drop (d). Because, in fact, there is always a certain amount of miscella that is more or less fast with respect to the average time calculated (Rataus, 2000).

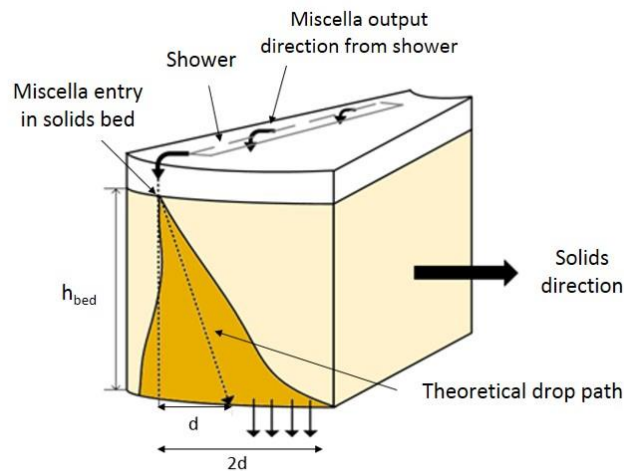


Figure 2 - Schematic representation of miscella path in the bed of solids inside the extractor.

The average speed of the miscella draining in soy bed is twice times faster than in rapeseed bed. Taking into account that the bed height is similar, the mixture of hexane and oil takes longer to pass through the rapeseed bed, delaying the development of the profile. On the other hand, the rotation speed of the extractor working with soybeans, is approximately twice times faster than when processing rapeseed. Therefore, the horizontal distance in both cases turns out to be equal.

Comparing the values of the horizontal distances with the distance between showers it is concluded that counter-current flow is not guaranteed. It is also quite probable that an excess of oil in the solids can be carried along the extractor. If, for some reason, the time take by the miscella to cross the bed increases, the oil will fall and can contaminate with an excess of oil the less concentrated miscela.

Residence Time in DTD

The residence time in DTD is an important variable to verify if the solids remain in desolventizing sufficient time for the total evaporation of the solvent until the expected residual concentration. To calculate the total residence time is necessary to know the input flow and the height of solids in each plate. With these values it is possible to adjust the solid levels on the plates for a certain amount of solids at the entrance, so that to obtain a residence time similar to the values presented in the literature. It is worth noting that the amount of hexane in the solids at the entrance of the equipment influence these calculations. In fact, if this amount is too high the time that will be necessary to evaporate the solvent will increase.

The total residence time calculated for the soybean flow is 43 min, which is slightly higher than that given in the literature, between 30 and 40 min (Manual para Extração - Bunge, 2007). With this value it is expected to have a residual content of hexane in the solids between 100 and 500 ppm as indicated in the literature (Brueske & Crown Iron Works Co., 2000).

For rapeseed the calculated residence time was 97 min. According to Vidal (2000), the residual hexane content in the solids will be 195 ppm. This result is very low compared to the values reported in literature, between 500 and 800 ppm.

Hexane Condensation

To reduce the amount of hexane emitted into the atmosphere, one of the options is to install a new condenser. To study the hexane condensing system, the temperatures of the inlet and outlet streams of the condensers were measured during the processing of soy and rapeseed. These temperatures allowed the calculation of the heat transfer driving force given by the log mean temperature difference, ΔT_{in} , which should not be less than 5°C (Baasel, 1990).

The results show that during the rapeseed processing 4 condensers have a ΔT_{in} less than the expected value, whereas in the soybean process 2 condensers do not reach the minimum expected ΔT_{in} value. These results are related to the fact that these condensers are at the end of the network, where the gases temperature is already near the ambient temperature.

From the observations made in the plant, it was found that the number of condensers seems to be adequate, even for the highest flow rate (with soy) as in the last condenser condensation was not observed thus indicating that the content of hexane in the exhaust gases should be very small.

Hexane Recovery with Mineral Oil

To reduce the amount of hexane in the gaseous effluent is also necessary to analyze the ultimate hexane recovery system by absorption in mineral oil. The analyses of the flow rate of gases removed per seeds quantity that enters in the process allowed to conclude that gaseous flow rate is smaller than expected, between 1 to 2.5 m³ air/ton of seeds (Manual para Extração - Bunge, 2007), when the calculated values were 0.8 for soybeans and 0.5 for rapeseeds.

So, either the system is very well isolated with no air intakes, which is not the case, or the system is not working under vacuum, as it is supposed to, because of the removal of a too low amount of air. Actually, it is quite difficult to maintain the system under vacuum.

Concerning the flow of mineral oil, the ratio between volume of mineral oil and the volume of circulating gases was 179 L/m³ for rapeseed and 50 L/m³ for soybeans, which reveals the use of an excess of mineral oil comparing with the values indicated in literature, minimum rate of 25 L/m³ (Wegner, 2000). Thus, a higher gas flow rate can be handled and so higher vacuum values can be attained.

HEXANE LOSSES ANALISYS

Hexane losses can be due to leakages that occur due to malfunction or poor maintenance of equipment and piping or to losses in the various outputs of the process: meal, oil, gaseous effluents and liquid effluent (wastewater) from hexane decantation.

The total daily hexane loss is measured by taking into account the daily variations of the hexane accumulated in different equipment. However, to account for the losses in the different output streams it is necessary to collect and analyze samples of the different products and effluents. To identify the leakages it is necessary to go through the facilities to check the status of each leak point and also to detect new leaks points.

Gaseous Effluent Losses

To monitor hexane in the gaseous effluent a portable explosimeter detector was used. This equipment comprises a flammable gas detector, in this case, calibrated to detect hexane. This type of equipment is used to analyze the atmosphere in a confined space, particularly in areas with explosion danger, for detection of equipment's leaks. The concentration is measured in relation to the lower explosivity limit that represents the minimum concentration of a flammable gas in the air which allows the combustion in a presence of an ignition source. This measure only allows the estimation of the hexane concentration knowing the gas flow rate.

Losses Distribution

After calculating the losses in oil and estimating the gaseous and solid losses it is possible to calculate the distribution and impact of all the different losses (Figure 3).

The hexane losses in the crude vegetable oil are measured by the relationship between concentration and the flash point temperature of the oil, which is determined using the Pensky Martens method.

Since it was not possible to analyze the solids, the hexane losses with the solids were estimated considering a concentration of 500 ppm after desolventizing, as reported in the literature (Brueske & Crown Iron Works Co., 2000). Neglecting the losses with the liquid effluent, the losses of hexane due to leakages were calculated by difference, i.e. the total loss hexane minus the sum of the above mentioned values.

As seen in Figure 3, the losses with the oil during rapeseed processing have a greater impact than when processing soybeans. This is due to the fact that losses are calculated in relation to the amount of meal and in rapeseed process more oil per ton is extracted than in soy.

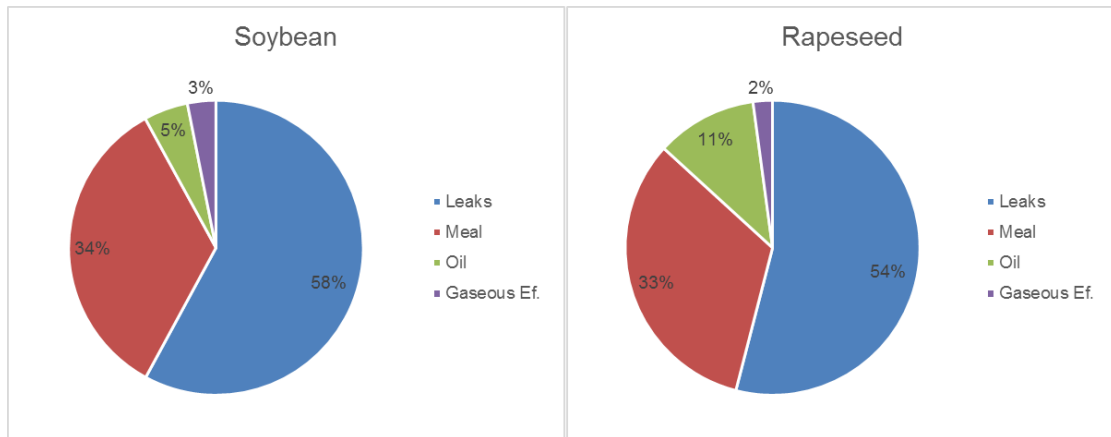


Figure 3—Losses distribution when processing soybean and rapeseed.

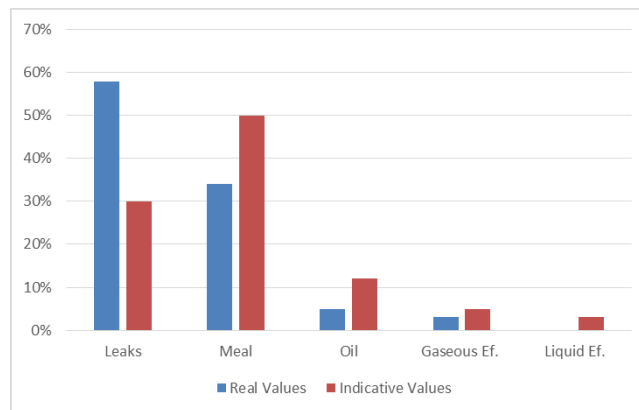


Figure 4 - Comparison between estimated losses and the literature results (Dpto. Técnico A&G, 2000).

Comparing the results estimated in this work with the literature values presented in Figure 4 is verified that for soybeans the major differences are in the losses with the meal and in leakages. These differences should be a direct consequence of the values considered in this work for the various terms, since the leaks term is calculated by difference.

To check the influence of each term in the hexane losses a sensitivity analysis was made. In this analysis, first the concentration of solvent in the meal was varied between 500 and 1000 ppm with variations of 50 ppm. The results show that to obtain a percentage of loss relative to the meal' losses similar to the literature value ($\approx 50\%$) its hexane concentration should be 800 ppm. In the case of oil losses variations of more or less 10°C in flash-point temperature were made, since this is the temperature range used in the laboratory measurement. The variation of hexane concentration with temperature is not linear but for higher temperatures 10°C represents approximately 20g hexane/ton of processed soy or 1% of the total losses.

Finally, the variation of the losses in the gaseous effluent was studied by changing the %LEL between 70 and 100 (range of values measured in field). The results show that 2% variation in the explosivity limit represents an increase of only 1 g of hexane loss per ton of processed soybeans.

Hexane Leaks

As expected, the locations where the higher incidence of leakages was detected were the extractor and DTD, as it is where the hexane exists in larger quantities. In DTS, the highest incidence of leakage is at the top because that is where it has the largest amount of hexane to evaporate. The other places where hexane can be lost are mostly the outputs of gases that have been in contact with hexane and also other streams contaminated with hexane emitted into the atmosphere.

For the rapeseed process most of the leaks are located on the extractor. Indeed, leakages in extractor are similar to those of soybeans but it was found that with rapeseed there is a sharp reduction of leakages in DTS when compared to soy, thus increasing the importance of the extractor leaks.

With the measurements made it was found that the leakages are more than two times higher when processing soybeans than with rapeseed, probably because the amount of solids and gases in circulation is also greater. Furthermore, the difficulties in establishing the vacuum in the system while processing soybeans can also explain part of the observed leakages.

It is also important to note that the leakages detected in the extractor and DTD were due to mechanical problems, such as lack of screws in some grooves, lack of isolation/lubrication of equipment fittings and worn-out of moving parts.

It is also important to emphasize that the explosimeter does not disclose concentrations exceeding the lower explosivity limit of 100%. Therefore, a measure of 100% might correspond to a higher amount than that considered these calculations.

Shut down leakages

The start-up and shut-down operations are critical in terms of the hexane losses.

In fact, for the extractor, normal operation existing leakages still occur during a stop but new leakages of liquid hexane were also found. .

This is why that during the extractor stop the leaks detected are almost triple than in normal operation but this value decreases with time as the extractor empties.

For DTS, the leaks also remained during the stop but there was a reasonable reduction of the detected values with the increase of the stopping time.

CONCLUSIONS

The two equipment that suffered more instability derived from wrong or unstable values of the variables are the extractor and DTD. Therefore, it is where that the most significant solvent losses occurred.

The major problems that increase solvent losses are related to the malfunctioning of the equipment and also to the high number of start-ups and shut-downs of operation. Inefficient control of the process and the lack of monitoring losses' points are also a major problem. Providing a better control of the amount of hexane in circulation and/or accumulated in the system is also important.

According to this study, most of the hexane is lost through leakages and in the solids streams. In fact, in normal operation the hexane losses into the atmosphere per quantity of seeds processed are near but below the maximum value allowed by legislation. Thus, the monitoring of the hexane losses should start by its control in the solids produced in DTS. Furthermore, to prevent leakages it is also necessary to improve the maintenance of the extractor and DTD, paying particular attention to their mobile parts.

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