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Research Paper

Study on the Most Efficient Method, for Chemistry Laboratories, on the Recovery of Heptane Emulsed to Crude Petroleum

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ABSTRACT: *Waste management and treatment programs are increasingly relevant, taking into account the global scenario. However, the use of the concept of 3R (Reduction, Reuse and Recycling) still needs to be implemented in university labs. The use of this concept can avoid environmental contamination and thus promote operator and community safety. In this perspective, the present work intends to define an adequate methodology for the reuse of N-heptane used in petroleum analyzes. Methodologies will be compared using rotary evaporator and fractional distillation. The treated residues contained crude oil and n-Heptane, being recovered by fractional distillation under two conditions. In condition 1 a column 80.0 cm long and 6.0 cm in diameter was filled with glass rings and in condition 2 with Raschig rings in stainless steel. The second methodology used a conventional rotary evaporator. The analyzed responses were: the total time of the process, the recovered percentage of n-Heptane and its degree of purity, evaluated by gas chromatography. This study allowed to demonstrate the most appropriate methodology for the treatment of this residue and the economic advantages resulting from the effluent minimization.*
Keywords: *Concept of 3R's, waste management, distillation, n-Heptane recovery and separation processes*

I. INTRODUCTION

The waste generation stream has always been part of any and every activity performed by man over time. According to Levada (2008), the final treatment and / or disposal of waste have been the subject of several surveys throughout history. However, with the concepts of green chemistry being rigidly charged, this topic has been the subject of several research and techniques to update procedures.According to Júnior (2012) with the increasing world population, a vision of issues such as poverty, pollution and scarcity of natural resources. Making interest in green chemistry more and more common, where processes are being exchanged and / or optimized, through environmentally correct techniques.Nolasco et al (2006) points out that the generation of waste resulted in a problem we need solved, especially in industries and analysis laboratories. It is known that the professionals who work in their segment did not receive adequate training to deal with these problems, also because the teaching and research institutions did not treat their chemical residues from laboratory activities, historically speaking.

However, according to the Constitution of the Federative Republic of Brazil Updated 2010, Article 225, of the Environment:

Everyone has the right to an ecologically balanced environment, a common good used by the people and essential to a healthy quality of life, imposing on the public power and the community the duty to defend and preserve it for present and future generations.

According to Nolasco et al (2006), many of the most respected institutions in the country, concerned with the current situations of accumulation of waste, opted for the "3R Concept" (SILVA et al 2014). The act of recycling, reusing and reusing as much waste as possible, aiming to provide stability for the environment to breathe, that is, to return to its equilibrium.

The research on the subject of waste recovery and management has come either now being carried conducted (NOLASCO et al 2006; JÚNIOR, 2012; LEVADA, 2008; OLIVEIRA, 2010; JARDIM, 1998) for liquids and solids. According to Nolasco et al (2006), some of the largest and most respected universities and institutions in the country are participating in the creation and implementation of this theme.

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According to Júnior (2012), in order to proceed with waste management, it becomes essential to establish operational routines of waste stream, or, collect input and output data. Because due treatment and analysis of such results, it allows the creation of a logistics without waste management in order to facilitate its recovery process.

Therefore, an effluent recovery can present a high efficiency, through its reuse or financial viability, adding savings with disposal and / or recovered product.

II. OBJECTIVE

In view of the above, the objective of this article is to create a methodology for the recovery of n-Heptane mixed with petroleum, using two known techniques and to put into practice the more efficient, evaluating the results. The proposal is not only a compilation of the international literature, but also the experience and improvement at the Federal University of Rio de Janeiro (UFRJ), and more specifically, within the Laboratory of Development and Optimization of Organic Processes (DOPOLAB).

III. METHODOLOGY

For an efficient waste generation program certain changes and attitudes were maintained during the procedure. Therefore, it was stipulated that at the end of the analysis the wastes were segregated, performing steps to be followed from the beginning (segregation of the tailings) until the analysis of the purity of the recovered solvent.

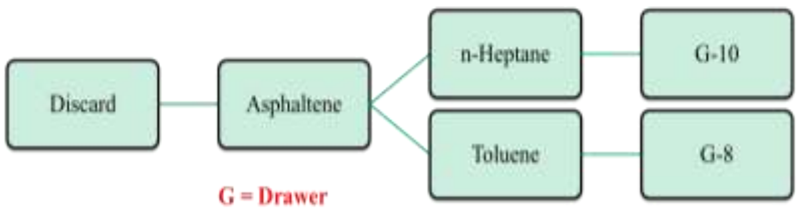

Figure 1 – Disposal flowchart.

In order to have effectiveness segregation, a flowchart for laboratory was elaborated, according to the example of Figure 1, presenting to the technicians the final destination of each type of residue, from a given analysis, to be deposited in the cylinders or bottles which are properly prepared, labeled and separated by drawers.

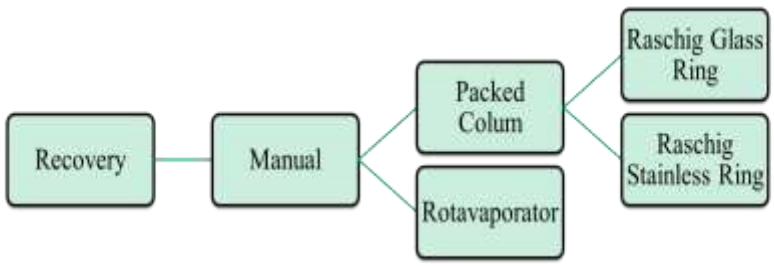

Figure 2 – Methodologies flowchart.

In the elaboration of the methodology of the project, according to the model of Figure 2, the tests were developed through the manual system, using a stuffed column (Fig.3), 31.5 inch long, 2.4 inch in diameter and acting with atmospheric Pressure, configured under two conditions. In condition 1, the test was performed with glass Rraschig rings and condition 2 with Raschig rings in stainless steel. The rotavaporation process (Fig. 4) utilized a conventional rotary evaporator with a vacuum system.

Figure 3 – Packed Distillation Column.

Figure 4 – Rotavaporator.

All the tests carried out used 500 ml of oil-containing waste and n-Heptane and worked with glassware with a volume equivalent to 1 L (bottom flask and collector). The total times, distillate volumes were recorded in form developed for this project and calculated recovery efficiencies.The products obtained in each distillation were suitably tested by gas chromatography and compared with a standard reference solvent for the quantification of their purities.

IV. RESULTS AND DISCUSSION

The Table 1 presents the consolidated results for the evaluated responses in the choice of the best methodology: recovery efficiency, time spent and solvent purity for both techniques employed.

Table 1 – Recovery data of n-Heptane.

Type of Process	Recovery Efficiency (%)	Time Spent (time)	Solvent Purity (%)
Packed Colum (Glass Rings)	88,91	1:00	99,77
Packed Colum (Stainless Rings)	96,95	1:04	98,58
Rotavaporator	70,40	0:59	99,82

Comparing the results obtained by manual distillation techniques, it can be seen in Table 1 that the parameter time spent proved to be insignificant for the choice of the best separation process. The recovery efficiency parameter depends on the process used and presents a higher value when used the column distillation technique with stainless steel filling, in comparison with the other methodologies, explained by the fact of the maintenance of heat coming from these rings. The purity of the recovered solvent has a higher value when the technique is used with the rotavaporator; however, it is concluded by means of the gas chromatographic analysis that in this recovery, the entrainment of higher molecular weight impurities is greater.

In Figure 5, the results of the chromatograms of the N-Heptane samples recovered by each methodology can be observed and compared with the standard 99.9% N-heptane PA sample. It was possible to observe that the distillation in stuffed column with the use of Raschig rings in stainless steel presented a greater drag of impurities to temperatures slightly above the boiling point of n-Heptane, causing in a percentage of Purity lower than other results, demonstrating that a high recovery efficiency may not be as effective for solvent reuse.

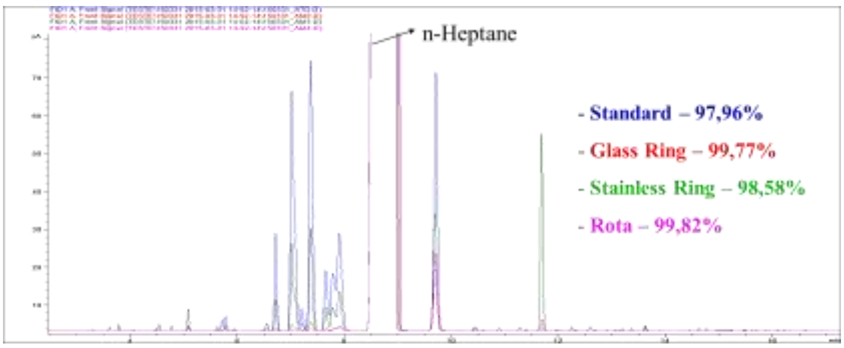

Figure 5: Results of the chromatogram.

The percentage of purity of the distillation method with glass rings and vacuum gives very similar results. However, in situations where oil mixed with the solvent has a high salt content, it was detected that the rotavaporation system does not retain this impurity, unlike Distillation in a packed column, being unsuitable for use in such situations.

V. CONCLUSION

In the methodologies tested considering all the evaluated responses and the specific problems of each, the best recovery option of n-Heptane was using manual packed distillation column with glass Raschig rings.By computing all the results obtained during the project, a reduction of up to 88.91% of the waste volume of the laboratory (petroleum and n-Heptane) was reported, according to the conditions of the methodology chosen to be higher efficiency and with a higher percentage of purity than commercial standard purchased by the laboratory.The use of solvent recovery and reuse methodology for petroleum emulsified n-Heptane proved to be able to provide an estimated saving of $ 5,150 per year for the laboratory. Economics is that it will remain constant, since; the solvent can be treated continuously over time. With the reduction of waste from the laboratory that would be discarded for a final disposal company, we can also see a gain for the environment by reducing material to be incinerated with consequent decrease of CO_2 emission in the atmosphere.

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