



## STUDIES ON SORPTION AND REGENERATION OF GRANULATED CORK FOR OIL AND GREASE REMOVAL

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**ABSTRACT** – The aim of this work is to develop a method to recover sorbed oil and grease from granulated cork, in order to promote biomass regeneration. Experimental procedure involved biomass saturation with oil, followed by chemical regeneration. Oil removal by elution was tested using HNO<sub>3</sub> and NaOH solutions, cationic and anionic surfactants and organic solvents, namely carbon tetrachloride and n-hexane. Chemical desorption efficiency using organic solvents achieved values of  $90 \pm 6$  % and  $72 \pm 6$  % for CCl<sub>4</sub> and n-hexane, respectively. Although the use of organic solvents proved to be an efficient process, it involves high costs and a negative environmental impact. Physical regeneration could be an alternative to the use of solvents for oil removal from granulated cork.

**KEY WORDS:** granulated cork; oily wastewaters; biosorbent regeneration.



## 1. INTRODUCTION

Oil and food processing industries are two main sources of water contamination. Development of new technologies for oil and grease removal from wastewaters is of great importance concerning the environmental impact. These contaminants are mostly water-soluble and dispersed oils. When present in high concentrations, they are efficiently removed by physical and chemical processes, such as gravity separators, flocculation and flotation processes (Patterson, 1985); however for low concentrations the existing methods are expensive and inefficient (Peng et al., 2008). The proposed new approach is based on oil and hydrocarbons' sorption in cork granules (Silva et al., 2005).

If the sorption process is to be used as an alternative in oily wastewater treatment, sorbent regeneration may be crucially important to keep low processing costs and open the possibility of recovering oil and grease. The regeneration process should yield oil and grease in a concentrated form, which facilitates disposal and restores sorbent for effective reuse.

Chemical and mechanical processes have been applied to removal of oil from silica gel, neusilin and agricultural wastes (cob powder, walnut shell), such as chemical flooding, supercritical fluid extraction (Skerget et al., 2007) and compression (Srinivasan et al., 2008). It was reported that sorption of oil on regenerated biomass decreased in successive cycles (Srivastava et al., 2011).

The aim of this work is the development of a chemical regeneration process to separate oil from cork granules, allowing its reuse and keeping the same performance.

## 2. MATERIAL AND METHODS

### 2.1. Biosorbents

Granulated cork is a by-product of the cork industry and it is available in a wide range of granulometries with different densities. Expanded cork with an average diameter between 2 and 4 mm was used in this work.

### 2.2. Solutions preparation

A synthetic oily wastewater was prepared by ultrasonic emulsification of mineral oil in water. For chemical desorption, the following eluant solutions were prepared: HNO<sub>3</sub> 0.1 M, NaOH 0.1 M, ethylenediamine tetraacetic acid (EDTA) 1 mM, hexadecyltrimethylammonium (HDTMA) bromide 10 mM and sodium dodecyl sulfate (SDS) 0.1 mM.

### 2.3. Sorption and regeneration studies

Batch sorption experiments were carried out at constant temperature of 25 °C for a period of 18 hours until equilibrium by contacting 150 mL of synthetic emulsion, containing approximately 2 g L<sup>-1</sup> of mineral oil, with 0.04 g of dark granulated cork. The experiments were conducted in duplicate and the homogeneous distribution of cork in the emulsion medium was achieved by magnetic stirring. Biomass was accurately separated from the liquid phase and then stored for regeneration tests. Quantification of oil and grease that remained in the solution was performed by the partition-infrared method according to methods 5520-C and 5520-F of *Standard Methods* (APHA et al).

Batch desorption experiments with H<sub>2</sub>O, HNO<sub>3</sub> 0.1 M, NaOH 0.1 M and surfactants solutions were conducted in duplicate, by contacting 150 mL of eluant, with 0.04 g of oil-loaded cork. The suspension was stirred at a constant temperature of 25 °C for a period of 18 hours. Regeneration tests using solvents, such as CCl<sub>4</sub>, n-hexane and methanol, were carried out

by adding a fraction of the oil-loaded cork to a flask containing 10 mL of each eluting solution, and stirring vigorously for 5 minutes at a constant temperature of 25 °C.

The regeneration efficiency can be defined as

$$\text{Efficiency (\%)} = \frac{\text{Amount of oil recovered}}{\text{Amount of oil loaded}} \times 100 \quad (1)$$

### 3. RESULTS AND DISCUSSION

Elution efficiency using the selected eluants was calculated using Eq. 1 and results are presented in Figure 1. It can be observed that elution efficiency is higher using organic solvents:  $90 \pm 6\%$  and  $72 \pm 6\%$  for  $\text{CCl}_4$  and n-hexane, respectively. On the other hand, elution efficiency using NaOH seems to be reasonable,  $58 \pm 18\%$ ; however, partial destruction of cork was observed when in contact with alkaline solutions. Water and cationic surfactants exhibited low desorption efficiencies.

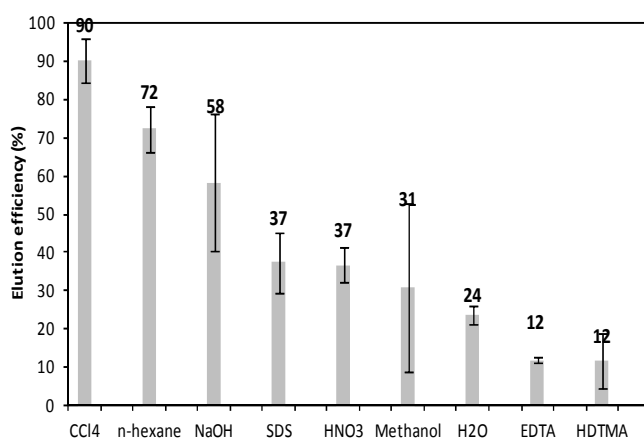


Figure 1. Elution efficiency for each eluant tested.

Fig. 2 shows the elution efficiency using  $\text{CCl}_4$  as function of solid-liquid ratio. It can be seen that elution efficiency decreases considerably with S/L ratio. As oil sorption seems to be a reversible process, high oil and grease concentrations released by the eluant can

decrease elution efficiency, retaining an amount of oil loaded in the cork. Thus, it can be concluded that the influence of this parameter in the elution efficiency is significant.

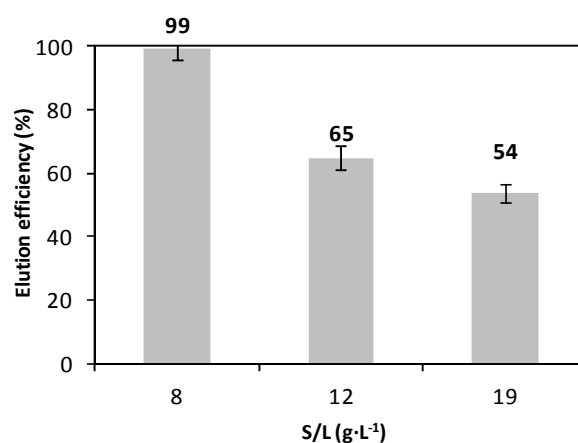


Figure 2. Influence of solid-liquid (S/L) ratio on elution efficiency using  $\text{CCl}_4$  as eluant.

Desorption using  $\text{CCl}_4$  is a rapid process, reaching an efficiency of 100 % in 2 minutes (Fig. 3). In this study,  $\text{CCl}_4$  proved to be an excellent eluant for granulated oil loaded cork; however it is a very expensive and unhealthy solvent.

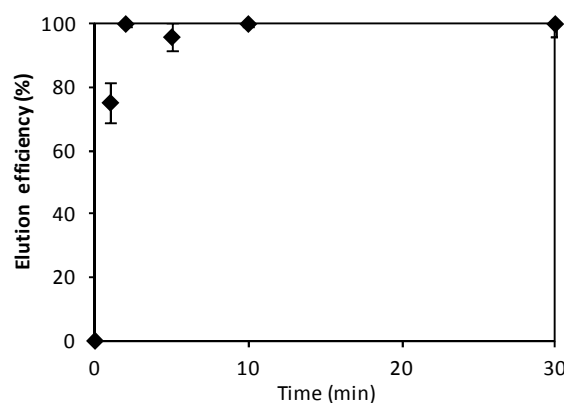


Figure 3. Experimental desorption kinetic data using  $\text{CCl}_4$  for  $\text{S/L} = 8 \text{ g L}^{-1}$ .

Physical regeneration processes, such as centrifugation and compression, will be studied to minimize the high costs and the negative environmental impact.



#### 4. CONCLUSIONS

Regeneration of cork in different consecutive cycles was achieved using chemical processes. The use of organic solvents as eluants resulted in high desorption efficiencies:  $90 \pm 6 \%$  and  $72 \pm 6 \%$  for  $\text{CCl}_4$  and n-hexane, respectively. Desorption using  $\text{CCl}_4$  was very fast, taking less than 2 minutes; however, organic solvents are very expensive and unhealthy. As an alternative, physical regeneration processes will be studied.

#### 5. ACKNOWLEDGMENT

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