

EXPERIMENTAL RESEARCH ON LOW TEMPERATURE THERMAL DESORPTION OF SOILS POLLUTED WITH CRUDE OIL

Pop Dorina, *PhD Candidate, Eng. Technical University of Cluj-Napoca,
Romania*

Micle Valer, *Prof. PhD. Eng. Technical University of Cluj-Napoca, Romania*

Sur Ioana – Monica, *PhD. Eng. Technical University of Cluj-Napoca,
Romania*

ABSTRACT: This paper presents the results of the experimental researches on the parameters of thermal desorption applied for the decontamination of soils polluted with crude oil. The main parameters tested in this process were the temperature (200 and 300 degrees Celsius) and the time for keeping the sample in the oven (10, 15 and 20 minutes). After the analyses, we can observe that by using the two established parameters, the thermal desorption is efficient. The highest thermal desorption efficiency was obtained when keeping the sample for 20 minutes at a temperature of 300 degrees Celsius.

Key words: thermal desorption, decontamination, pollution, crude oil.

1. INTRODUCTION

The problems of environmental pollution due to oil exploitation require precise knowledge, in order to implement the most adequate measures for preventing and fighting against harmfulness.

The technology of thermal desorption is part of the thermal decontamination procedures. It can be used for various types of soils and contaminants, especially being used for oil products and organic pollutants, having a series of advantages [4]:

- Easy and rapid implementation;
- Destruction of polluting compounds;
- Allows the re-use of the de-polluted soils.

2. MATERIAL AND METHOD

In order to research the basic parameters of the thermal desorption technology, we elaborated the scheme of the research plan, presented in figure 1:

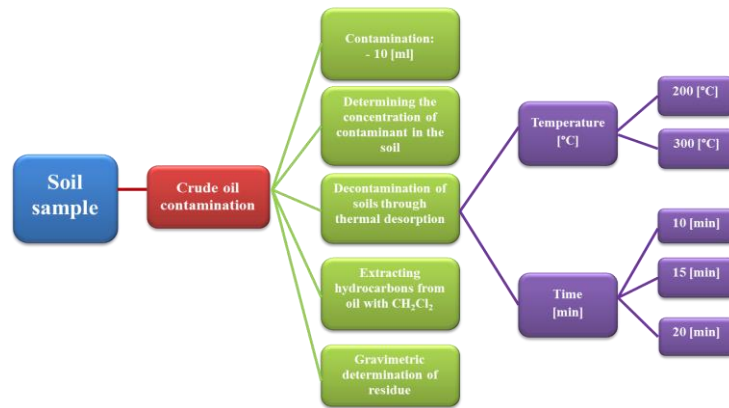


Figure.1. The scheme of the research plan

The soil sample was taken from the depth interval of 0 – 20 cm according to STAS 7184/1-75, in the commune of Bonțida, Cluj County (Fig. 2).

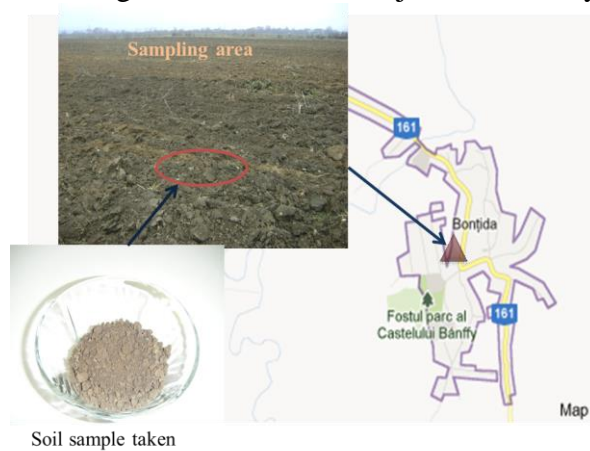


Figure 2. The overall image of the sampling area

The experimental researches on the thermal desorption were carried out at the Technical University of Cluj-Napoca. The experimental determination of the crude oil content in the control sample was performed using the Soxhlet method, as per STAS SR 13511/2007 [2].

This method is the most well-known technique of solid-liquid extraction, which, in practical analyses is performed in an extraction device having the same name, presented in figure 3.

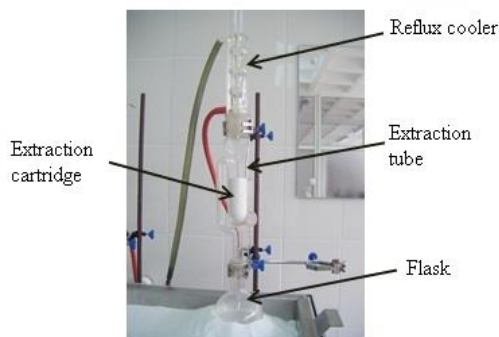


Figure 3. The Soxhlet device

The equipment necessary for applying the thermal desorption technology was the oven – Memmert VO 500 used for the

temperature of 200 degrees Celsius and the silicon carbide bars oven for the temperature of 300 degrees Celsius. The

contamination of the soil samples was performed in laboratory. The quantity of contaminant used was of 10 ml of crude oil per 100 g of soil, of which a quantity of 10 g of soil was subject to decontamination. The depollution was performed by repeated heating of the samples, at various temperatures (200 and 300 degrees Celsius) and different amounts of time for keeping in the proposed installations (10, 15 and 20 minutes), aiming at eliminating the crude oil from the soil.

3 RESULTS AND DISCUSSIONS

3.1. Determining the quantities of crude oil in the control sample

The value of the initial concentration determined for the control sample was of

79000 mg/kg dried substance. As compared to the alert threshold (1000 mg/kg dried substance) and intervention threshold (2000 mg/kg dried substance) according to Order no. 756 of 11/03/1997 [1], it results that the determined values exceed the legal thresholds.

3.2. Variation of the quantity of crude oil after thermal desorption

After applying the depolluting technology, we have determined the content of crude oil according to STAS SR 13511/2007. The results obtained are shown in figures 4 and 5. By examining figure 4 we can observe that the level of crude oil contained in the samples used for decontamination at a temperature of 200 °C, decreases as the time for keeping the sample in the oven increases.

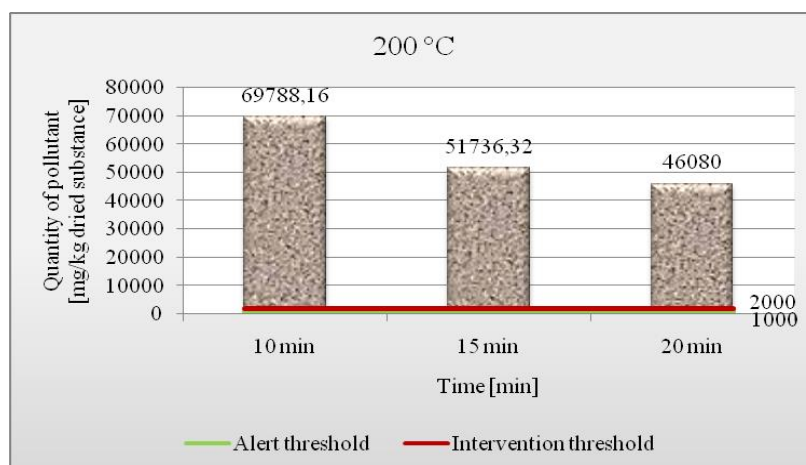


Figure 4. Variation of the quantity of crude oil in the soil after thermal desorption at a temperature of 200 °C

Increasing the temperature to 300 °C (Fig. 5), and keeping soil samples for 10, 15 and 20 minutes in the silicon carbide bars oven, has led to the decrease of the

quantity of crude oil below the intervention threshold (2000 mg/kg), for the sample kept in the oven for 20 minutes.

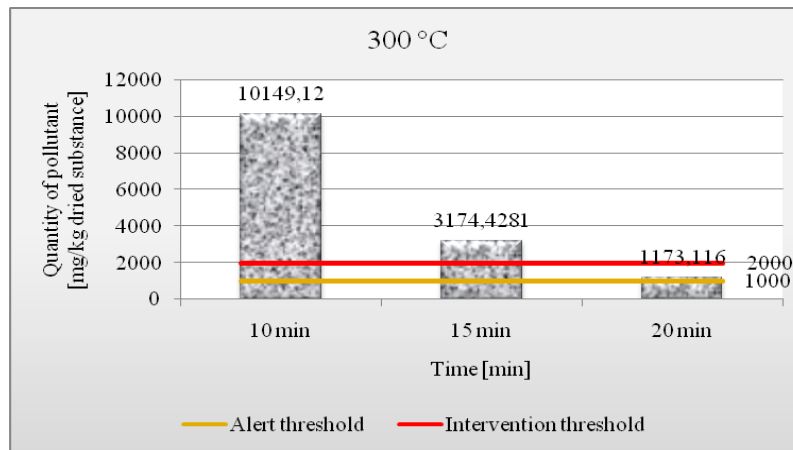


Figure 5. Variation of the quantity of crude oil in the soil after thermal desorption at a temperature of 300 °C

3.3. Variation of the quantity of crude oil extracted after thermal desorption

The quantity of pollutant extracted from the soil using the thermal desorption technology was determined by calculating the difference from the quantity of crude oil existent in the control sample (before the desorption) and that accomplished after the desorption process. Thus, this can be shown using equation 1:

$$C_{\text{extracted}} = C_i - C_f \quad [\text{mg/kg}] \quad (1)$$

where: C_i – initial quantity of pollutant existing in the soil samples (control sample), in mg/kg;

C_f – final quantity of pollutant existing in the soil, determined using the Soxhlet method, after the desorption process, in mg/kg;

$C_{\text{extracted}}$ – quantity of crude oil extracted, in mg/kg.

In the following diagrams (Fig. 6 and 7) you can observe that at temperatures of 200 and 300 °C the concentration of crude oil extracted increases as the variable parameter of the desorption increases – the amount of time.

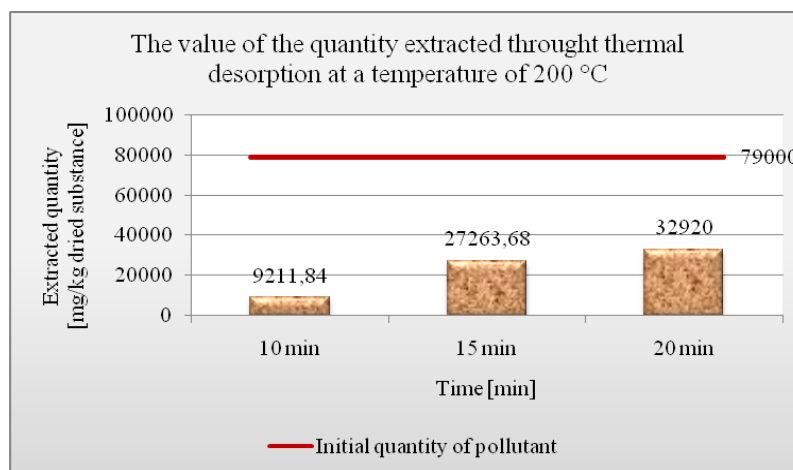


Figure 6. Variation of the quantity of crude oil extracted through thermal desorption at a temperature of 200 °C

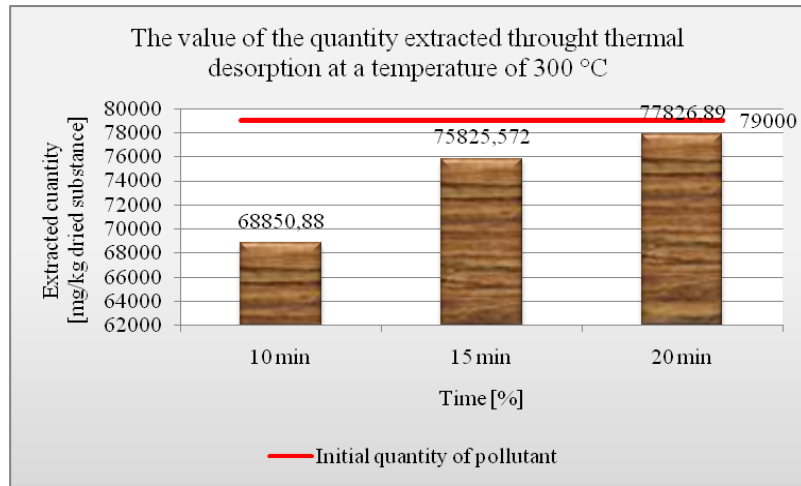


Figure 7. Variation of the quantity of crude oil extracted through thermal desorption at a temperature of 300 °C

In conclusion, we can say that through desorption, the quantity of crude oil was almost entirely extracted at the temperature of 300 °C, while kept in the oven for 20 minutes.

3.4. Efficiency of the extraction process

The evaluation of the efficiency of the extraction technology was performed by determining the final extraction efficiency, it is estimated on synthetic samples or mixes (in this case the soil polluted in the lab), in which the quantity of analyte added is known (control sample), $m_{\text{analyte}}(\text{sample})$. After determining the quantity of analyte in the solvent used in the extraction, $m_{\text{analyte}}(\text{solvent})$, the extraction efficiency is shown in the formula 2 below [4]:

$$\eta = \frac{m_{\text{analyte}}(\text{solvent})}{m_{\text{analyte}}(\text{sample})} \cdot 100 [\%] \quad (2)$$

where: - $m_{\text{analyte}}(\text{solvent})$ – the quantity of pollutant extracted through thermal desorption at different temperatures and amounts of time kept in the oven, in mg/kg;

- $m_{\text{analyte}}(\text{sample})$ – the initial quantity of pollutant existent in the soil (that can be extracted using the Soxhlet method), in mg/kg.

After performing the analysis of the experimental results obtained (table 1), we made the diagrams representing the variation of the efficiency of crude oil desorption, depending on the temperature and the amount of time kept in the oven which were previously established. They are presented in figures 8 and 9.

Table 1. Efficiency obtained after thermal desorption

Crt. No.	Quantity of pollutant [ml]	Concentration of the pollution [mg/kg]	Temperature [°C]	Time [min]	Efficiency of the thermal desorption [%]	Humidity [%]
1.	10	79,000	200	10	11.66	13,2
2.	10			15	34.51	
3.	10			20	41.67	
4.	10	79,000	300	10	87.15	
5.	10			15	95.98	
6.	10			20	98.51	

The efficiency of extracting crude oil through thermal desorption at a temperature of 200 °C (Fig. 8), shows a moderate increase, as the amount of time for keeping the sample in the oven increases. Thus, the highest efficiency is

achieved in the case of the sample kept in the oven for 20 minutes, having an efficiency of 41,67 %, while for the sample kept for 15 minutes the efficiency was slightly lower, namely 34,51 %.

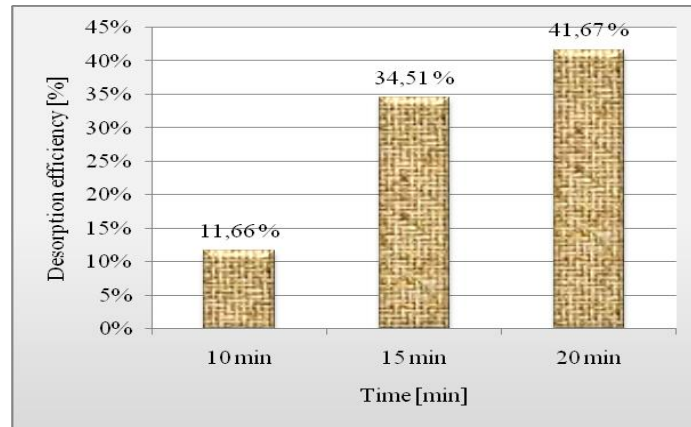


Figure 8. Efficiency of thermal desorption at 200 °C

The highest desorption efficiency of crude oil at the temperature of 300 °C was achieved for the samples kept in the oven for 15 and 20 minutes (Fig. 9), in these cases the extraction efficiency of the two samples exceeding 95 %. In the

case of the sample treated in the oven for 15 minutes, the efficiency is of 95,98 % while for the sample treated for 20 minutes, the efficiency is of 98,51 %.

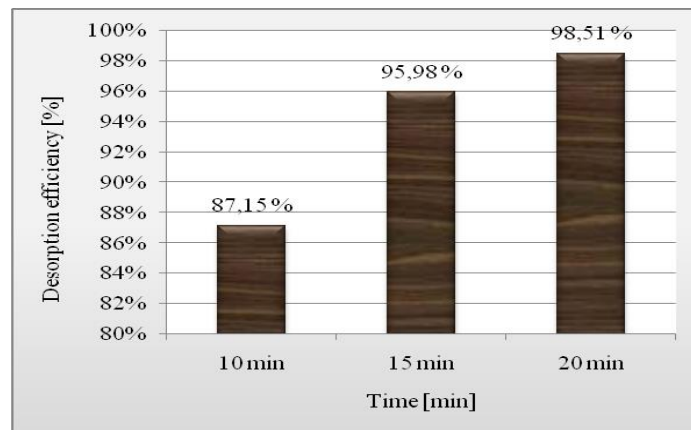


Figure 9. Efficiency of thermal desorption at 300 °C

4. CONCLUSIONS

➤ Experiments performed have shown that, after treating the soils contaminated with crude oil using thermal desorption at the temperature of 200 °C and amounts of time established at 10, 15 and 20 minute in the oven, the efficiency achieved was below 50 %.

➤ The efficiency obtained after by treating soil samples at the temperature of 300 °C is high, having values between 87,15 and 98,51 % depending on the amount of time for keeping the sample in the oven, namely 10, 15 and 20 minutes respectively.

➤ While the treating temperature and the amount of time for keeping the samples in the oven increases, the level

of decontamination increases substantially. Thus, the optimal parameters for obtaining the maximum efficiency are the temperature of 300 °C and the amount of 20 minutes for keeping the sample in the oven.

5. REFERENCES

- [1]. Order no. 756 of 11/03/1997 the approval of the Regulation regarding the evaluation of the environmental pollution, the Official Gazette no. 303 of 11/06/1997.
- [2]. SR ISO 13511, 2007. Soil quality. Determining the total content of hydrocarbons in the soil. The gravimetric method.
- [3]. ***, Chapter 5. Solid – liquid extraction (SLE), http://cachescan.bcub.ro/2008_05_28/cap_5_pagini_82_89.pdf
- [4]. ***, Remediation techniques, Chp. 5, <http://rtpime.files.wordpress.com/2010/03/cap-5.pdf>