



Variability of the components of oil drilling waste according to the treatment temperature

Variabilidad de los componentes de los residuos de perforación petrolera según la temperatura de tratamiento

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Abstract

In Mexico's hydrocarbon sector, drilling waste represents a problem because the techniques to treat it increase production costs. In this regard, thermal desorption represents a good option, even though it presents variations in hydrocarbon removals, which causes added costs since some residues need to be treated again. Thus, the objective of this study was to identify these variations according to the treatment temperature (< 320 °C) on a laboratory scale. The results showed that the samples had minerals in common (quartz, baryte and anhydrite) that did not vary after the treatments. However, other minerals were found in some samples, which were not present in others. The hydrocarbons decreased with respect to the temperature fluctuations. However, when comparing the removed percentages and the fractions, they were different from each other. Moreover, Cd, Cr, Pb, Ni, Zn, V, Ba, Fe, and Mn were detected, but their concentration in the leachates was low. It was concluded that the variation in the constituents of the drilling cuttings could depend on the source of generation and that this factor could influence the final properties of the treated solid. When the removal of hydrocarbons was achieved, the fractions there, and the concentration of metals were different between the samples.

Keywords: Metal, Hydrocarbon, Mineralogy, Waste.

Resumen

En el sector de hidrocarburos de México, los residuos de perforación representan un problema porque las técnicas para tratarlos aumentan los costos de producción. Al respecto, la desorción térmica representa una buena opción, aunque presente variaciones en las remociones de hidrocarburos, lo cual causa costos agregados ya que algunos residuos necesitan ser tratados nuevamente. Es por esto, que el objetivo del presente trabajo fue identificar estas variaciones según la temperatura de tratamiento (< 320 °C) a escala de laboratorio. Los resultados mostraron que las muestras tenían minerales en común (cuarzo, barita y anhídrita) que no variaron después de los tratamientos. Sin embargo, se encontraron otros minerales en algunas muestras, que no estaban presentes en otras. Los hidrocarburos disminuyeron con respecto a los cambios de temperatura. Sin embargo, al comparar los porcentajes removidos y las fracciones, fueron diferentes entre sí. Además, se detectó Cd, Cr, Pb, Ni, Zn, V, Ba, Fe y Mn, pero su concentración en los lixiviados fue baja. Se concluyó que la variación en los constituyentes de los recortes de perforación podría depender de la fuente de generación y que este factor podría influir en las propiedades finales del sólido tratado ya que, si bien se logra la remoción de hidrocarburos, las fracciones presentes y la concentración de metales fueron diferentes entre las muestras.

Palabras clave: Hidrocarburo, Metales, Mineralogía, Residuo.

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1 Introduction

Concerning the environment, the existing guidelines for the final disposal of industrial waste require special attention (Xu *et al.*, 2018). It is a problem, especially, because in many cases of industrial waste disposal, there are no studies to establish the danger level represented by one or various of the components of this waste (De Titto & Savino, 2019). In this regard, the hydrocarbons sector is the topic of interest since waste vary according to the phase it is found. For example, during drilling, waste, such as water production (congenital water) and impregnated solids with hydrocarbons, are generated, while in the petrochemical process, residual polymer waste such as tank sludge and boiler ash are generated. In all these cases, the chemical nature of the waste is usually different and, therefore, also its effects (Iryna & Leonid, 2016).

Due to the volumes generated, the drilling cuttings require special attention, since they are treated to give them final disposition. The concentration of metals and hydrocarbons in the treated solids is variable. Vidonish *et al.* (2016) reported that this phenomenon may be related to the properties of the residue. Other reports, highlight the importance of this behavior, especially because the leaching of metals and hydrocarbons represents a risk to the health of the population and ecosystems (Ossai *et al.*, 2020). For example, long-term studies have been conducted in various ecosystems bordering the Gulf of Mexico. They found bioaccumulation of heavy metals (V, Cr, Cd, and Pb) and hydrocarbons (Kennish & Paerl, 2010). Also, in similar regions, these same compounds have been found as components of petroleum in soils, sediments, and effluents (Fayiga *et al.*, 2018).

In recent years, the hydrocarbon sector in Mexico has had an increase in oil waste derived from the 2013 Energy Reforms. These reforms initially were considered for exploration and drilling as well as for the reactivation of some mature oil fields. From 2018 to date, these activities have increased due to the construction of the Dos Bocas refinery and the reactivation of other mature fields in order to revitalize the economy after the health emergency due to Covid-19. From then on, various environmental problems have been found (Alpizar-Castro & Rodríguez-Monroy, 2016; Moreno-Brid & Gallagher, 2020). The first problem is that large volumes of congenital water are generated, to the point that the existing latrine pits are not sufficient. Because of this, treated water through diffusers are discharged into the Gulf of Mexico. These discharges increase the concentration of metals, mainly in sediments (Kennish & Paerl, 2010; Gómez-Mellado *et al.*, 2020; Canul-Chan *et al.*, 2023).

Environmental Base Lines (LBA) were established and environmental liabilities were declared where oil activities were reactivated. Thus, the second problem is that on these sites, the level of contaminant risk and remediation strategies

are not yet established (Alpizar-Castro & Rodríguez-Monroy, 2016; Gómez-Mellado *et al.*, 2020). The third problem, that requires special attention in Mexico, is the generation of drilling cuttings, even though the chemical stabilization technique may be more effective, the thermal desorption technique is more used in Mexico because it requires less space and shorter periods.

There are also a few desorption units in this region that receive residues from various sources, with different components that can influence the variations in hydrocarbon removals (Lui *et al.*, 2019; Falciglia *et al.*, 2020). Likewise, one of the main challenges facing thermal desorption is the high operating costs, mainly at high temperatures in the treatment processes. Besides, the removal of hydrocarbons varies according to the source of generation of these residues. This phenomenon has limited its usefulness in Mexico since hydrocarbon exploration and production costs are increasing due to the complexity of offshore oil extraction (Zhao *et al.*, 2019; Ahumada *et al.*, 2022).

In this context, some studies have reported that solids contaminated with hydrocarbons have been treated by desorption. This procedure can be limited by the mineralogy of the waste (Khanpour *et al.*, 2014). In the same way, other studies have shown that the interaction between the contaminant and the solid phase varies according to the conditions to which they are exposed (Yi *et al.*, 2016). Similarly, although some reports have indicated that the mineralogy of each oil well may vary according to the geological basin, this indicator has not been considered in the pre-treatment, nor has it been considered during drilling. Besides, components are added based on the characteristics found in the deposit (Zhang *et al.*, 2022). Therefore, each cutting will have a different component.

The removal of hydrocarbons and the concentration of metals in the leachate can be a function of the conditions of the thermally treated solids. Consequently, in this study, the changes in the rate of the removal of hydrocarbons and metals present in the leachate of drilling cuttings treated by thermal desorption (at different temperatures) are analyzed (Snedden & Galloway, 2019).

2 Materials and methods

In this study, three samples were selected from different oil sites in southeastern Mexico (Macuspana and Comalcalco). The first sample is a recovered mud (LO) from an exploratory well located in Comalcalco, Tabasco (UTM 15 Q 478818,93 m E, 2018244,47 m N). The second sample was taken from the drilling waste tank of an oil well (RC1) located in Paraiso, Tabasco (UTM 15 Q 471040,40 m E, 2038786,70 m N). The third sample (RC2) was obtained from an offshore well in the Gulf of Mexico (UTM 15 Q 603067,80 m E, 2144044,51 m N) (Snedden & Galloway, 2019). At each site, waste vat samples (quartering) were

taken, and a composite sample was constructed, for which each sample was deposited in a metal tray and divided into four parts, and random aliquots were taken until 1 kg was obtained. These were labeled and stored in a refrigerator with ice (4 °C) and transported to the laboratory for analysis (SEMARNAT, 2013).

From each one (waste vat sample), a composite sample was constructed. For this, each sample was deposited in a metal tray and divided into four parts, and random aliquots were taken until obtaining 1 kg. These samples were named control samples. Afterwards, they were homogenized by mechanical stirring for 1 h. Finally, the pH and the Electrical Conductivity (EC in $\text{dS}\cdot\text{m}^{-1}$) were measured (wet base).

2.1 Thermal treatments

It has been shown that the behavior of organic compounds on solid particles involves diffusion, adsorption, and absorption phenomena. This is probably because the adsorption of water molecules moves in the hydrophobic spaces of the mineral, even though this depends on the density of hydrocarbon (Zhao *et al.*, 2019). In addition, the problems of diffusion of hydrocarbons in mineral fissures, due to the presence of congenital water (for oil-based muds), are reduced with the incorporation of additives which composition and chemical nature is highly variable (Abdou *et al.*, 2018; Zhang *et al.*, 2022). Likewise, in some studies, it is recommended to evaporate the residual water and to work with low and slow temperature ramps (Li *et al.*, 2019).

For these reasons, the thermal treatments (T) consisted of taking 50 g of each homogenized control sample (dry weight). Afterwards, they were distributed in three batches each ($n = 3$). Thus, they were passed to a rotary oven. This oven was placed in an extraction hood of gas equipped with filters to avoid the emission of pollutants into the atmosphere. Also, during the study, it was decided to work with three thermal treatments: T1: 90-167 °C, T2: 167-245 °C, and T3: 245-320 °C, 10 °C $\cdot\text{min}^{-1}$, and 60 rpm (Li *et al.*, 2019). After each ramp, a batch was removed from the furnace to obtain a representative sample of each heat treatment. It should be noted that after each test, the oven was preheated again before starting the next thermal treatment. These samples were named treated samples.

2.2 Determination of hydrocarbons and mineralogy

The determination of Total Petroleum Hydrocarbons (TPH in $\text{mg}\cdot\text{kg}^{-1}$) in samples (treated and control) was done by a continuous extraction (Soxhlet) (Yzquierdo-Ruiz *et al.*, 2022). On the one hand, the extract obtained underwent a SARA analysis (fractions saturated, aromatics, resins, and asphaltenes, all in weight (%)) by solvents selectivity in a silica column packed with hexane (Díaz-Ramírez *et al.*, 2008). On the other hand, the remaining solid of each Soxhlet treatment was dried at 450 °C in an electric furnace.

Then sample weight (%) was calculated by gravimetry. Afterwards, 1 g of the obtained dry solid was dealt with 0.5 mL of H_2O_2 (30%), waited 10 min and later dried at 105 °C for 24 h; of the obtained solid, 0.5 g were weighed. This was grounded into a quartz mortar. Finally, the crystal structure was analyzed using the X-ray (XRD).

2.3 Metal content

The metal content was performed on the control and treated samples, as well as their leachates. First, they (each sample) dried at 105 °C, then crushed and sieved. The resulting solid, aliquots of 2 g were taken and treated with H_2O_2 , and HNO_3 (concentrated), allowed to react for a few minutes, and then calcined at 320 °C for 5 h. The obtained sample was treated with HCl (concentrated), filtered. The extract was graduated to 50 mL with deionized water. To determine leachable metals, after each heat treatment, 20 g of each sample was taken and a PECT extract (by its acronym in Spanish that corresponds to the Procedure for Extraction of Toxic Constituents) was made according to the methods established in NOM-053-SEMARNAT-1993 (SEMARNAT, 2003; García-Arreola *et al.*, 2015; Borda *et al.*, 2022). In all samples, the total metals (Cd, Cr, Cu, Pb, Ni, Zn, V, Ba, Fe, and Mn) were determined in an atomic absorption spectrophotometer (Perkin Elmer Analyst Model 800 AA) according to the methods of the NOM-147-SEMARNAT-SSA1-2004 (SEMARNAT, 2007; Hernández-Mendoza *et al.*, 2018).

Finally, an ANOVA test (p-value, $\alpha = 0.05$) and a Tukey analysis (Honestly-significant-difference or HSD and, significance level of 0.05) were performed in Project-R in order to determine significant differences between the three controls (LO, CR1, and CR2) and, the changes that the properties of each one had after the heat treatments (Control, T1, T2, and T3) (Wang & Mi, 2019).

3 Results and discussion

3.1 Hydrocarbons

The ANOVA analysis carried out to the values obtained, showed the physical and chemical properties of the mud (LO) and the drill cutouts (RC1, and RC2) (Table 1). The analysis indicated that at least one control sample was different from the other two ($p < 0.05$) when comparing the results of the Tukey Test. Differences were found between the three means of TPH, solids (% SO), EC (Electric conductivity in $\text{dS}\cdot\text{m}^{-1}$), S, A, and R (Saturates, Aromatics and Resins hydrocarbons, respectively, all in %weight) since all the values were greater than their respective HSD (Table 1).

However, in pH, RC1, and RC2 there were no significant differences ($X_1 - X_2 < \text{HSD}$ calculated and specified in

Table 1). Both samples were different from LO ($X_1 - X_2 > \text{HSD}$ calculated and specified in Table 1). In addition, although the hydrocarbons found in each sample made them different from each other ($p < 0.05$), when comparing the amounts of Asphaltenes fractions (%Asf), no differences were observed between LO and RC2 ($X_1 - X_2 < \text{HSD}$ calculated), nevertheless both were different from RC1 ($X_1 - X_2 > \text{HSD}$ calculated and specified in Table 1).

It should be noted that the values of fractions S, A, and R, as well as solids of the sludge control samples, were different from the values observed in the control cuttings. It may be because the sludge was treated and reused, where the residues generated from these treatments were drilling cuttings. However, when comparing the TPH and solids (SO) values of the cutting control samples, it was observed that these were also different. This coincided with the information found in the literature review which mentioned that the mud suffers wear during the drilling, a process in which crude oil (with a higher content of A, with respect to the diesel used in oil-based mud) and reservoir rocks are added (Yi *et al.*, 2016). Though, the proportions of rocks and hydrocarbons in the samples may vary due to reservoir conditions, mainly geological characteristics (Temraz & Hassanién, 2016; Zhang *et al.*, 2022).

In addition, the difference in SO, pH, and EC between LO, RC1, and RC2 may be related to the fact that different

types of additives were added during drilling depending on the characteristics of the deposit, such as sodium lignosulfonate and $\text{Ca}(\text{OH})_2$, used to control acidity or to inhibit the swelling of clays (which may coincide with the alkaline characteristics of RC1 and RC2) (Zhang *et al.*, 2022). Another important point was that salinity in drill cutting samples had been related to two factors, the first was the production water (with high sodium content), and the second with salt domes, so probably for these reasons drilling cuttings had higher EC than mud (Whitaker *et al.*, 2016).

In terms of removal percentages after each treatment, it was observed that applied temperature allowed the removal of TPH, but this may vary depending on the source of generation. For example, when comparing the values of the controls, T1 removes about ~17% of TPH was observed in all the samples (LO, RC1, and RC2). However, in T2 and, LO achieved ~70% elimination, whereas RC1 showed a reduction of ~45%, and ~58% in RC2. Furthermore, the longest hydrocarbon removals were observed at T3, as a mass loss of ~80% was achieved in LO, and RC1, but only ~70% in RC2. In the case of SO, there were increases in T1 (~17%), T2 (~65%), and T3 (~85%) in LO. On the contrary, for RC1, and RC2, the behavior was similar, and the folds were low in T1 (T1 (~14%), T2 (~16%), and T3 (~16%).

Table 1. Properties, hydrocarbon content, and SARA fractions present in the samples analyzed.

Control								
Sample	TPH ($\text{mg}\cdot\text{kg}^{-1}$)	SO (%)	EC ($\text{dS}\cdot\text{m}^{-1}$)	pH	S (%)	A (%)	R (%)	Asf (%)
LO	36 ± 0.7	48 ± 1.1	0.10 ± 0.01	7 ± 0.1	48 ± 0.5	8 ± 0.1	22 ± 0.1	23 ± 0.2
RC1	11 ± 0.2	80 ± 1.6	3 ± 0.5	8 ± 0.1	37 ± 0.1	21.3 ± 0.1	7 ± 0.1	35 ± 0.2
RC2	24 ± 0.5	68 ± 1.3	3 ± 0.6	8 ± 0.2	14 ± 0.3	55 ± 1.1	9 ± 0.2	22 ± 0.4
HSD	1.29	2.86	0.11	0.36	0.88	3.32	0.33	0.70
Treatments								
LOT1	30 ± 0.2	65 ± 1.11	$20 \pm 0.$	17 ± 0.1	48 ± 0.2	8 ± 0.1	22 ± 0.1	22 ± 0.1
LOT2	15 ± 0.3	84 ± 1.1	ND	ND	33 ± 0.1	7 ± 0.3	21 ± 1.1	45 ± 0.31
LOT3	6 ± 0.1	89 ± 1	ND	ND	ND	2 ± 0.01	6 ± 0.17	91 ± 1
HSD	0.99	2.65		4.53	0.81	2.24	6.38	
RC1T1	9 ± 0.1	90 ± 1.1	312 ± 1	7 ± 0.1	37 ± 0.8	21 ± 0.2	7 ± 0.1	34 ± 0.2
RC1T2	6 ± 0.2	90 ± 1	ND	ND	26 ± 0.1	19 ± 0.9	7 ± 0.3	49 ± 0.3
RC1T3	2 ± 0.1	91 ± 1	ND	ND	ND	6 ± 0.2	2 ± 0.1	92 ± 0.41
HSD	0.40	2.93		3.52	2.15	0.76	6.88	
RC2T1	20 ± 0.1	78 ± 0.3	411 ± 6	8 ± 0.1	13 ± 0.2	55 ± 2	9 ± 0.1	22 ± 0.3
RC2T2	10 ± 0.2	79 ± 1	ND	ND	10 ± 0.4	48 ± 0.3	9 ± 0.4	33 ± 0.2
RC2T3	8 ± 0.2	81 ± 1	ND	ND	ND	14 ± 0.4	3 ± 0.2	83 ± 0.5
HSD	0.68	7.96		1.29	5.56	0.95	5.67	

TPH: Total Petroleum Hydrocarbons. So: solids. EC: Electrical Conductivity. S: saturated; A: aromatics; R: resins; Asf: asphaltenes LO: recovered mud, RC1: drilling waste tank of an oil well, RC2: offshore well. T1: 90-167 °C, T2: 167-245 °C, T3: 245-320 °C.

In addition, as the temperature increased, two effects were observed, one related to the gradual loss of TPH and the other one with the increase to the % of solids (SO). Although it was evident that the temperature favored the elimination of TPH, the desorption of the solid phase fractions was different in each case. This behavior shows that hydrocarbon removal depends not only on temperature but also on the properties of the solid.

Also, significant differences were observed between TPH, SO, EC, and pH in all control samples and T1 (HSD between means is greater than calculated. In these results, it was noted that an increase in salinity in drilling waste, that could be related to the production water content (EC is greater RC1, and RC2 compared to LO). Furthermore, the cuttings contain material added during this process and could also be related to a higher content of solids in the cuttings (Söregård *et al.*, 2020). It is important pointing that pH, and EC, could only be determined in samples of T1 in LO, since for RC1, and RC2 the reading was very variable when using the method of the saturated paste extract, the samples presented water repellence (Adams *et al.*, 2016), therefore decided to place them as not determined (ND).

Although TPH is usually the hydrocarbon indicator, there are differences between the SARA fractions of the samples after each treatment. In this sense, sludge is regularly composed of a fraction of light to medium hydrocarbons, which have a high content of saturated hydrocarbons and, when used, tend to decrease their density due to wear and tear and combine with reservoir components obtained that contain asphaltenes and resins (water repellence) that cause a problem with the solubility of some additives or the stability of the emulsion (Mao *et al.*, 2019). Other studies explain that raising the temperature favors the mobility of the different fractions, regularly the lightest ones evaporate but the heaviest is deposited in the porous sites, although to date it has not been explained how the different conditions in the solids allow agglomeration or availability of the pollutant, it has been observed that aging makes them more refractory and with less mobility (Zhang *et al.*, 2018).

3.2 Mineralogy

Figure 1 displays the XRD analysis of the LO, RC1, and RC2 untreated samples. The LO sample (a) showed reflections at 200, 22.730, 24.80, 25.85, 26.79, 28.89, 31.51, 32.74, 42.54, 42.87, and 43.98 in 2θ , which coincided with the BaSO_4 phase (baryte) according to the ICDD 72-1378 card. Similarly, peaks were observed at 210, 26.6, 39.55, 48.84, and 54.92 in 2θ , which coincided with SiO_2 phase (quartz) according to the ICDD 05-0490 card. These minerals (baryte and quartz) have been reported for drilling sludge (Temraz & Hassanien, 2016; Mahmoud & Elkhatny, 2019). In addition, the most intense reflection of this diffractogram was observed at 25.42 in 2θ coinciding with the CaSO_4 phase (anhydrite) according to the ICDD

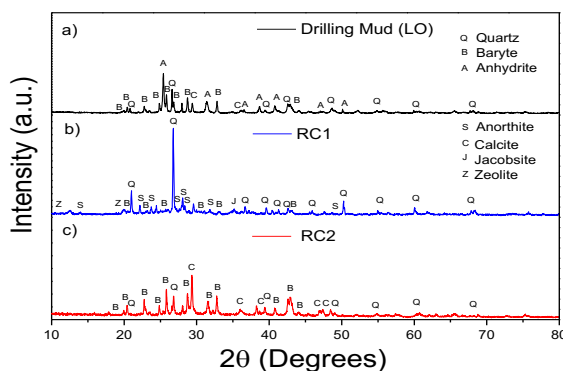


Figure 1. Diffractograms performed on witness samples: LO (a), RC1 (b), and RC2 (c).

37-1496 card. Also, the CaCO_3 phase (calcite) was also detected by its signals at 29.38 and 36.15 in 2θ , according to the ICDD 71-3699 card, in the literature, it is reported that 5% of iron oxides in commercial barite is used in their wells.

However, Barite is also a synthetic ceramic material widely used for its ferromagnetic properties that combined with CaSO_4 contribute to reducing corrosion. Similarly, Barite (BaSO_4) is used to stabilize lutite or similar rocks (Abdou *et al.*, 2018). It is worth mentioning that no aluminum oxides were identified that are contaminants in benthic sludge, but it may be due to the diversity of signals shown in the XRD spectrum in the LO (Temraz & Hassanien, 2016).

Besides, the RC1 sample (Figure 1b), showed the characteristic intensities of the SiO_2 phase (quartz) at 210 and 26.600 in 2θ , as well as, peaks of the BaSO_4 phase (baryte) at 200, 22.73, 25.85, 26.79, and 28.89 in 2θ . Additionally, signals of the anorthite phase (feldspars) (Ca 0.68, Na 0.32) (Al 1.68, Si 0.32), Si_2O_8 were identified at 21.98, 23.59, 27.51, 27.78, 27.84, and 28.02 in 2θ , according to the ICDD 89-1469 card. Some studies report the anorthite phase formation in the southeastern Basin of Mexico (Snedden & Galloway, 2019). In the same way, other couples of signals were observed at 29.62 and 34.92 in 2θ which correspond to the MnFe_2O_4 phase (jacobsite), according to the ICDD 75-3192 card; finally, some peaks at 10.30, 19.54, and 19.72 in 2θ were detected and related to the mineral phase $(\text{SiO}_2)_{64}$ (Silicon dioxide, Zeolite sigma-2, dehydrated) according to the ICDD 77-4190 card (Mahmoud & Elkhatny, 2019).

In contrast, RC2 sample (c) showed a large content of BaSO_4 phase (baryte) considering the intensity and number of peaks in the diffractogram (22.73, 24.80, 25.850, 26.79, 28.89, 31.51, 32.74, 42.54, 42.87, and 43.98 in 2θ). Also, the CaCO_3 phase (calcite) was the one with the most intense reflection (~ 29.38 in 2θ). In addition, this cut-off identified the presence of SiO_2 (Quartz) (21 and 26.60 in 2θ) (Abdou *et al.*, 2018). Comparing the signals found in the untreated sludge and comparing them to those observed

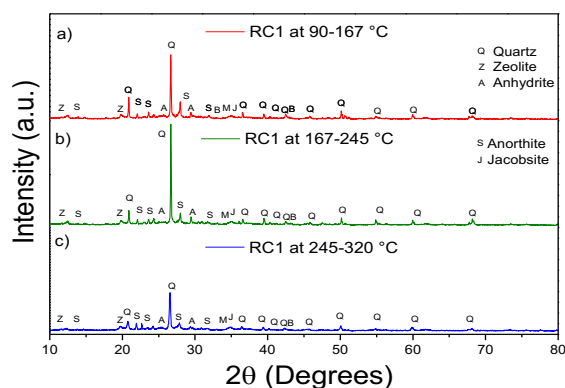


Figure 2. Diffractograms of the RC1 sample using thermal treatments at 90-167 °C (a), 167-245 °C (b) and 245-320 °C (c).

in the drilling cuttings, it can be related that barite, quartz, and anhydrite might come from the components in sludge, although the latter did not appear in the drilling cuttings. It is possible that removal occurred during the oil recovery process, since some reports mention that it is used as an additive (Androvitsaneas *et al.*, 2017). It was also observed that the LO had the greatest similarity of signals related to RC2, although these may be overlapped. In contrast, in RC1, new signals such as zeolite, anorthite, and jacobsite were identified. They could come from the oil deposit. Therefore, these results showed evidence that drilling cuttings can vary their mineralogical components depending on where they are generated. In addition, this evidence contributes to establishing measures to guarantee the greatest removal of hydrocarbons and the reduction of operating expenses (Brooks *et al.*, 2020).

Figure 2 displays the diffractograms of the RC1 sample after being applied to diverse thermal treatments. On these spectrums, a slight increment in the intensity of the peaks was observed after having used T1 (a, and b); Zhang *et al.* (2016) observed that this behavior could be related to a slight increase in the densification or agglomeration of the particles by desorption-deposition of hydrocarbons and not by a phase change (changes to temperatures >500 °C).

Likewise, in the sample of T2 (c), there was a decrement in the intensity of the peaks, as well as a slight widening of the peaks which can be interpreted as the rearrangement of particles and removal of some pores from the sample, especially by the deposition or coating related to heavy fractions (Hosseini-Dastgerdi & Meshkat, 2019). New phases were not observed during the applied heat treatment.

Moreover, Figure 3 exposes to the diffractograms of the RC2 sample after the thermal treatments. The same phases were identified in all the samples indicating that thermal treatment does not cause changes in the mineralogical components. However, the signals reduced their intensity as the treatment temperature was increased (a-c). This could be explained by Álvarez-Coronel *et al.* (2020). The authors reported that at higher temperatures asphaltenes

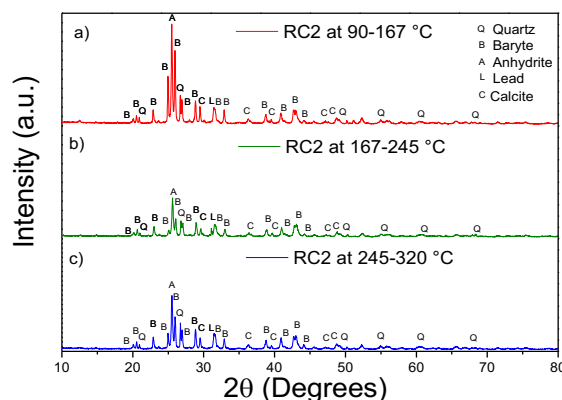


Figure 3. Diffractograms of the RC2 sample, thermal treatments at 90-167 °C (a), 167-245 °C (b) and 245-320 °C (c).

could reduce their density and increase their diffusion and, thus, covered part of the solid phase, causing variations in the DRX readings.

Also, the CaSO_4 phase (anhydrite) showed the highest signal at 25.42 in 2θ , possibly by residual hydrocarbons which were not deposited over this phase [33]. An alarming point is the signal detected at 31.2 in 2θ that corresponds to the Pb phase (Lead) according to ICDD 04-0686 card. This component has been reported in some sludge and congenital waters, as well as, in contaminated sites whose source of generation are hydrocarbons (Yi *et al.*, 2016; Wang *et al.*, 2017; Gómez-Mellado *et al.*, 2020).

3.3 Metals

Evaluating the content of metals present in the solids shows, on the one hand, to know if they can be a source of contamination considering both the MPL (fresh spill) and, for other, previously the solids were disposed in pits that today present environmental liabilities. Also, it has been shown that the disposition of the contaminants in the leachate also depends on the characteristics of the solid (Robles-Martínez *et al.*, 2011; Yzquierdo-Ruiz *et al.*, 2022). In this sense, the ANOVA test of metals it was obtained a $p < 0.05$, regarding Tukey analysis, in all cases, the value of the differences between the means of each value was above the calculated HSD so the samples are different from each other (Table 2), these results together with those of XRD, confirmed the hypothesis that both the mineralogy as the metal content in the samples depended on the generation source.

In the same way, Table 2 shows that although Cd, Cr, Cu, Pb, Zn, and V were not detected in LO, they were found in RC1, and RC2, so it followed that these metals could be contributions from the drill column (Xu *et al.*, 2018; Chen *et al.*, 2022). In environmental terms, only V in RC1 exceeded concentration for industrial use, but Cd, Cr, Cu, Pb, Ni, Zn, Ba, Fe and, Mn do not exceed

Table 2. Metals present in untreated samples.

Sample	Cd	Cr	Cu	Pb	Ni	Zn	V	Ba	Fe	Mn
mg·kg ⁻¹										
LO	ND	ND	ND	ND	0.38 ± 0.02	ND	ND	15 000 ± 6	110 ± 1.4	20 ± 0.1
RC1	21 ± 0.8	3 ± 0.1	400 ± 1.1	43 ± 0.7	26 ± 0.9	8 ± 0.3	79 ± 1	31 000 ± 1	890 ± 4	80 ± 0.3
RC2	12 ± 0.4	12 ± 0.5	320 ± 11	59 ± 2	15 ± 0.3	12 ± 0.4	53 ± 2	43 000 ± 1	362 ± 2	78 ± 0.3
HSD	0.76	0.65	15.15	3.02	1.26	0.70	3.2	8.56	5.92	0.60

ND: Not detected.

the permissible limits of the NOM-021-SEMARNAT-2000 and the NOM-147-SEMARNAT-SSA1-2004 (SEMARNAT, 2002a, 2004b). Likewise, these values also coincided with some standards of EEUU (Da Rosa *et al.*, 2018; Johnston *et al.*, 2019; Méndez-Moreno *et al.*, 2021). Highlights the presence of Ba that coincided with results of XRD (Barite), so they are part of the mineral of the drilling mud or cut (Abdou *et al.*, 2018; Mahmoud & Elkatatny, 2019; Yzquierdo-Ruiz *et al.*, 2022).

In terms of the leachate in each sample according to the heat treatment, the ANOVA test indicates that in all cases $p < 0.05$ and, the results of the Tukey test show that when contrasting the differences of the means between T1, T2, and T3 for each sample in many cases this value is higher than HSD, so there are differences between the treatments (Table 3). For example, the leaching of Cd, and Cr in RC1, and RC2, when comparing the values in T1 is different from T2, and T3 ($X_1 - X_2 > \text{HSD}$ calculated and specified in Table 2), only the T3 samples present values below this indicator for Cd in both samples and in the case of Cr, it is observed that, except for the values in RC2T1, all are below the maximum allowed. In the case of Pb, in RC1 the values of the samples in T1, T2, and T3 do not present significant differences ($X_1 - X_2 < \text{HSD}$ calculated and specified in Table 2), on the other hand, in RC2, the values in T1 are different from T2 and T3 ($X_1 - X_2 > \text{HSD}$ calculated and specified in Table 2), but there are no significant differences between the values of these last treatments ($X_1 - X_2 < \text{HSD}$ calculated and specified in Table 2).

However, since the leaching of metals varied according to the source of generation, the final disposal sites must take measures regarding this, especially, because it has been reported that some of these metals can compete with other nutrients and reduce their availability to plants. Also, it is estimated that metals such as Cd, Cr, Cu, Ni, Zn and, V as potentially toxic due to cumulative effects on ecosystems (Kovacs & Szemmelveisz, 2017; Bakshi *et al.*, 2018). Similarly, it has been found that metals such as V, Pb and, Zn are associated with emissions and spills of products and waste from the oil industry (Wang *et al.*, 2017; Xu *et al.*, 2018; Khudur *et al.*, 2018). These studies, coincide with the results of evaluations carried out in areas close to the study (Kennish & Paerl, 2010; Gómez-Mellado *et al.*, 2020).

Although the mechanisms of adsorption-desorption of pollutants in soils is a topic of interest, but it is on the frontier of science. However, Iryna & Leonid, (2016) and Álvarez-Coronel *et al.* (2020) reported that metals and

hydrocarbons were retained in some solids but not in others. This phenomenon is associated with the textural properties of the solid, which can change depending on the conditions to which they are exposed. For example, some barium oxides usually retain Cr (Czop & Łaźniewska-Piekarczyk, 2020; Naveenkumar *et al.*, 2022). This has also been reported for calcium carbonates, and some clay (montmorillonite and zeolite) which retain metals such as Cd, Pb, Ni, and Zn (Manjaiah *et al.*, 2019; Moghal *et al.*, 2020). This phenomenon can explain why after removing some of the hydrocarbons, the retention of metals on the solid was increased. However, this retention varies according to the sample. In general, the least metal leaching was observed in T3 for all samples, so this could be an indicator to determine the temperature at which it is required to treat the solid and, in this way, reduce risks to the environment and health.

It is significant to point out that two of the metals found in the components of the cuttings and their leachates were Pb and Ba. The same special attention should be paid to the control measures in confinement cells or treated solids that are reused in linings since it has been demonstrated that both metals can cause toxicological effects in living beings (Steliga & Kluk, 2021; Omar & Vilcaez, 2022).

Conclusions

Based on the results, drilling muds can have from a similar composition. However, when drilling the well, new composites are added. These can be similar within the same paleontological basin, which shows that there are variations in drilling operations. This set of factors determines the final composition of the solid that becomes waste. These differences can condition the removal of hydrocarbons since the SARA fractions are different in each sample. Besides, the concentration of metals in the leachate was different between the treated solids. Moreover, there are common minerals that coincide with those found in the muds (barite and carbonates). There are some other elements such as Pb that are not found in every sample even though its source cannot be assured since the number of samples was reduced. Also, the ramp from 245-320 °C was the one that presented the best results compared to others with lower temperature values. However, it was found that the residual fraction was mainly composed of asphaltene (> 90%).

Table 3. Metals present in the leachate of treated samples.

Sample	Cd	Cr	Cu	Pb	Ni	Zn	V	Ba	Fe	Mn
Leachates (mg·kg ⁻¹)										
LOT1	ND	ND	ND	ND	0.31 ± 0.01	ND	ND	140 ± 5	103 ± 1.1	19 ± 0.1
LOT2	ND	ND	ND	ND	0.10 ± 0.01	ND	ND	110 ± 4.4	33 ± 0.2	11 ± 0.01
LOT3	ND	ND	ND	ND	0.10 ± 0.03	ND	ND	89 ± 1.1	11 ± 0.4	8 ± 0.03
HSD					0.02			0.54	0.29	0.06
RC1T1	1.3 ± 0.05	0.72 ± 0.028	0.04 ± 0.001	2.7 ± 0.1	0.17 ± 0.006	7 ± 0.21	61.44 ± 1.1	600 ± 1.2	75 ± 3.1	70 ± 0.28
RC1T2	0.9 ± 0.03	0.31 ± 0.012	0.01 ± 0.003	2.6 ± 0.10	0.15 ± 0.006	1 ± 0.03	2.8 ± 0.11	540 ± 2.12	6 ± 0.24	15 ± 0.6
RC1T3	0.12 ± 0.01	0.39 ± 0.015	0.01 ± 0.004	2.7 ± 0.10	0.12 ± 0.004	0.9 ± 0.03	0.33 ± 0.01	34 ± 0.36	6 ± 0.23	15 ± 0.02
HSD	0.02	0.001	0.0005	0.006	0.0003	0.009	0.08	1.09	0.10	0.09
RC2T1	2.11 ± 0.084	5.12 ± 0.202	0.04 ± 0.0016	1.59 ± 0.063	ND	11 ± 0.43	52 ± 1.06	310 ± 1.24	36 ± 0.41	50 ± 0.21
RC2T2	1.70 ± 0.068	1.27 ± 0.050	0.01 ± 0.003	0.8 ± 0.032	ND	2 ± 0.08	44.36 ± 1.32	120 ± 0.48	3 ± 0.01	12 ± 0.48
RC2T3	0.78 ± 0.06	1.16 ± 0.043	0.01 ± 0.001	0.8 ± 0.022	ND	1 ± 0.04	41.09 ± 1.34	89 ± 0.36	2 ± 0.02	12 ± 0.34
HSD	0.004	0.007	0.0005	0.003		0.02	0.11	0.46	0.05	0.07

ND: Not detected

These components are related to water repellence. Therefore, they can influence the interaction of metals and hydrocarbons with the PECT extract and. Consequently, their availability and presence in leachate was reduced. Although this hypothesis coincides with some studies, the content of metals in the extract is different between samples, therefore, it is important to carry out other studies with a greater number of samples to identify if these discrepancies continue, especially, because the treated solids were reused in roads lining or landfills. These considerations are not currently taken into account in the environmental regulations in Mexico, it would be important to include them in the regulations and thus reduce the risks in the sites surrounding these infrastructures.

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