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Abstract

Coastal areas are of great economic importance, where activities such as fishing, aquaculture, transportation, and tourism can generate billions of dollars. Although tourism is an important source of income, it can negatively impact the environment, especially regarding the generation of liquid and solid waste. Among these, oil and grease residues stand out, polluting millions of liters of water and representing a significant source of coastal environmental contamination. This study aimed to develop a methodology for analyzing oil and grease content based on solid-phase extraction associated with gravimetry, using a 3^2 factorial design. The proposed methodology was tested on sand samples from Copacabana beach, Rio de Janeiro, Brazil, and compared with Soxhlet extraction. The proposed methodology reduced solvent consumption and sample preparation time compared to Soxhlet extraction. The Student's t-test showed no significant differences between the methodologies. The oil content in the samples ranged from 0.50 g to 3.83 g of oil per kilogram of sand.

Keywords: Beach sand, solid waste, residual oil, solid-phase extraction.

Introduction

Coastal areas are of great economic relevance, and the occupation of coastal regions by communities for subsistence purposes dates back over 10,000 years (Rodriguez e Ruiz, 2010). Today, activities such as fishing, aquaculture, mineral extraction, and transportation are among the main interests discussed when it comes to coastal zones, making them strategically important areas from an economic standpoint (Bejarano et al., 2018).

Another significant economic activity in coastal regions is tourism. Beaches are among the top choices for tourists during holidays, vacation seasons, and weekend, especially in the warmer months. In Brazil, tourism has been growing each year. In 2024, the country attracted 6.65 million foreign tourists (a 12.6% increase compared to 2023), generating US\$ 6.62 billion in revenue by November 2024. In addition to foreign visitors, around 126 million Brazilians traveled within the country (EMBRATUR, 2022). Rio de Janeiro is one of Brazil's most visited cities, particularly in the summer months, which include world-famous events such as the Copacabana New Year's Eve celebration, the Carnival samba school parades, and international music concerts that draw large crowds to the city. One of the most popular beaches in Rio is Copacabana Beach, which stretches over 3 km of coastline and is considered a Brazilian landmark. In 2024 alone, the area received 7 million visitors, making tourism a vital source of income for the city (Rio de Janeiro, 2022).

Although tourism is a key revenue stream for coastal cities, it can negatively impact natural resources and the environment. Notable consequences include unplanned urban expansion and the search for new areas to build hotels and resorts, pollution from vehicles, and infrastructure related problems, especially the generation of liquid, solid, and semi-solid waste, which affects both the water and the sand of the beaches (Diaz-Solano, 2011).

The main environmental impacts caused by beach tourism are related to the management of solid and liquid waste produced by visitors and local businesses (Amuquandoh, 2010). These impacts are not limited to a specific area and may spread to nearby regions due to environmental variables and tidal dynamics. Poor management of coastal systems further exacerbates the negative effects of mass tourism (Dadon, 2002).

Among the waste products that severely impact beaches, oil and grease, particularly used cooking oil stand out. Many food establishments are located along the beach strip, providing convenience to visitors. Widely used in bars, restaurants, kiosks, and by street vendors, frying oil is produced in large volumes and, when improperly discarded, causes significant environmental damage (Benevides et al., 2018). One liter of oil has the potential to contaminate approximately one million liters of water. Oil pollution on beaches is a serious issue since these ecosystems serve as habitat for many organisms essential to coastal protection (Huettel, 2022).

Chemically, oils are esters with a long hydrophobic chain linked to a hydrophilic head. These molecules are difficult for bacteria to metabolize and tend to float on water, posing serious risks to marine ecosystems (Valentine et al. 2012). When discarded in sand, oil contributes to impermeability, leading to low oxygen concentrations, and when degraded, releases methane gas (CH_4), a potent greenhouse gas (Baldin e Lopes, 2009). Rainfall can carry oil into the ocean and increase the presence of rodents and disease-carrying insects. Environmental damage can become irreversible depending on the extent of the contamination. Oil hinders plant respiration and nutrient absorption, creating a barrier to sunlight for both flora and fauna. Once it reaches water, it becomes toxic to fish and other marine species (Benevides et al., 2018). In addition to cooking oil, oil spills along the coast can reach beaches through diffusion, combining with coastal sediments and forming a persistent compound that can remain unchanged for years (Milliori et al. 2004). This results in severe damage to beach ecosystems, affecting both wildlife and human visitors.

De Oliveira and Barboza (2023), in a recent systematic review, showed that the permeability of beach sand facilitates oil percolation, expanding the contaminated area and causing greater environmental impacts. Due to this high permeability, oil can accumulate underground, where degradation is more difficult due to limited oxygen availability, which reduces bacterial activity (Rowland et al. 2000). Additionally, there is the potential formation of alternating layers of oil and sand, which may slowly release the contaminant over time (Oliveira e Nicolodi, 2017).

In highly populated countries, frying oil waste generation reaches millions of tons annually (Baldin e Lopes, 2009). Managing this waste is essential to mitigate its environmental impacts. Used oil has high reuse potential and can serve as raw material for soap and biofuel production (Florindo et al. 2022). Therefore, monitoring this type of waste is necessary to maintain the quality of coastal regions.

99 Currently, there is no standardized technical procedure for detecting and quantifying oil
100 content in solid samples, such as beach sand. EPA method 9071B is commonly used. In
101 this method, the sample is mixed with a chemical drying agent, such as magnesium
102 sulfate, until a fine powder is formed, which is then refluxed with n-hexane in a Soxhlet
103 apparatus. The solvent is extracted by evaporation, and the oil and grease content is
104 determined gravimetrically. (EPA, 9071). This analysis takes approximately 6 hours. In
105 addition to EPA method 9071B, an adapted version of Method 5520D, also based on
106 extraction and gravimetry, described by the American Water Works Association
107 (AWWA), can be used to determine the oil and grease content in natural Waters
108 (AWWA, 2017). Bejarano et al. (2018) used this method in a study evaluating tourism
109 impacts on Puerto Valero beaches in Colombia, measuring oil and grease levels in both
110 water and sand samples. Method 5520D consists of dissolving oil in a nonpolar solvent
111 (usually hexane) using a Soxhlet extractor, followed by solvent evaporation and
112 weighing of the residue.

113 Other methods recommended by APHA include gravimetric partitioning (Method
114 5520B) and infrared partitioning (Method 5520C), the latter designed for samples that
115 may contain volatile hydrocarbons (AWWA, 2017). Despite the simplicity and low cost
116 of gravimetric-based methods (5520B and 5520D), they have several drawbacks, such
117 as the use of large volumes of solvents, multiple steps, and long extraction times (about
118 5 hours for 5520D), in addition to sometimes yielding imprecise results (Carvalho et al.
119 2019). Therefore, new approaches have been explored in recent years.

120 Kich and Bockel (2017) proposed a method for analyzing oils and greases using UV
121 spectrophotometry at 242 nm, achieving a recovery rate of 86.30%. Although the study
122 showed promise, the authors acknowledged the need for further optimization. Khorshid
123 et al. (2021) developed a spectrophotometric method based on the turbidity of an
124 emulsion composed of oil, water, and CH_2ClF_2 , measured at 293 nm. According to the
125 authors, the method is reliable, simple, and offers high selectivity and sensitivity.

126 Carvalho et al. (2019) proposed a gravimetric partition method (5520B), where water
127 was evaporated from the sample at 85°C in an oven, followed by solvent extraction of
128 the oily residue. Although recovery exceeded 93%, the water evaporation step took
129 approximately four days, an impractical duration.

130 While most studies focus on aqueous matrices, it is equally important to evaluate oily
131 residues in solid samples such as soils and sediments. Rauckyte et al. (2010) assessed
132 oil and grease levels in sediment samples using gas chromatography with flame

ionization detection (GC-FID), with extraction via hexane and ultrasound. Khozanah et al. (2021) determined oil and grease content in Jakarta Bay sediments using Fourier Transform Infrared Spectroscopy (FT-IR), following a prior extraction with a 1:1 dichloromethane/hexane mixture and solvent evaporation at 40°C. Karsi (2025) examined oil and grease levels in surface soil samples using FT-IR, achieving recoveries between 82% and 94% and good sensitivity (LOQ = 7.87 ppm).

These proposed procedures have some limitations, including long extraction times, the use of large solvent volumes, or high analysis costs. An alternative is Solid Phase Extraction (SPE), a technique well-established for isolating lipidic and neutral compounds, which uses less solvent and offers significantly shorter extraction times (Honorio et al., 2019). SPE is a liquid-solid separation technique based on classical chromatography mechanisms, employing a small open column (called an extraction cartridge) as the stationary phase. A solvent, referred to as the mobile phase, passes through the cartridge to elute the analyte (Lanças, 2009).

Due to its versatility, SPE has been widely used for extracting various analytes in environmental matrices, such as pyrethroids in water (Barrionuevo e Lanças, 2001), trace elements like As, Cd, Cu, and Pb in seawater for mass spectrometry quantification (Gireli et al., 2025), and biodiesel compounds like acylglycerols and glycerol (Moreira et al., 2020). Belay et al. (2025) developed a method for extracting 32 PFAS using SPE in human serum using online solid-phase extraction coupled with ultra-high performance liquid chromatography–high resolution mass spectrometry (UHPLC–HRMS).

Given the above context, the objective of this study is to develop a methodology for quantifying oil content in beach sand, based on solid-phase extraction associated with gravimetry.

Experimental

Reagents and apparatus

N-hexane analytical grade were purchased from Neon (Suzano, SP, Brazil).

The SPE was carried in a custom manifold built using a 3D printer with flow controller. Gravimetric analysis were performed using an analytical balance from the brand Shimadzu.

FTIR analysis was performed on a Shimadzu spectrophotometer, model IR-TRACER.

Study Area Selection, Sample Collection and Storage

The selected study area was Copacabana Beach, located in the South Zone of Rio de Janeiro. It is considered one of the city's main tourist attractions and holds significant economic importance for the state.

Sample collection was carried out between lifeguard stations 2 and 4 along Copacabana Beach, covering a total of 2 km. Sampling units were selected at strategic points coinciding with commercial establishments along the beachfront. Each sampling unit consisted of three points arranged perpendicularly toward the sea:

1. Near commercial establishments located on the Copacabana boardwalk,
 2. In the area that is constantly wet (reached by waves), and
 3. In an intermediate zone between the two aforementioned points,
- as shown in **Figure 1**.



Fig 1 Study region, Copacabana Beach, Source: Google Maps, adapted by the author

For sample collection, the surface layer of sand was discarded, as exposure to ultraviolet (UV) light decomposes large hydrocarbon molecules (King et al., 2014). Thus, samples were collected at depths ranging from 5 cm to approximately 20 cm using a sterilized aluminum scoop and stored in hermetically sealed plastic bags. Collections were conducted during the first days of the week to gather data reflecting waste discarded during the high-traffic beach days, aturday and sunday. Sampling was performed in March 2024, at the end of the Brazilian summer.

Sample Processing and Quantification of Oils in Beach Sand

For oil extraction from beach sand samples solid-phase extraction cartridges were prepared by packing sand into polypropylene syringes. Filters were placed at both ends of the cartridge to retain the packed material and prevent particle loss during sample percolation. A solvent (hexane) was passed through the cartridge at a flow rate of 1 mL per minute, and the eluate was collected in a pre-weighed test tube, as shown in the schematic representation in **Figure 2**.

The volume of solvent used for elution and the mass of sand packed into the cartridge were optimized based on an experimental design described in this study. After extraction, the solvent was evaporated in a water bath at 70° C until constant weight was achieved.

All experiments were conducted in triplicate.

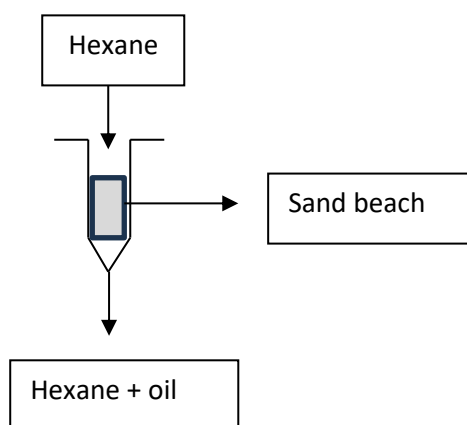


Fig 2 The schematic representation of the solid phase extraction

The quantification of oil in the beach sand samples was performed using gravimetry, and the oil content in the samples was determined using the following calculation:

$$\text{Oil content (mg/g)} = [(B - A)/m] \times 1000 \quad \text{Equation (1)}$$

Where:

B = mass of the test tube + residue (in mg)

A = mass of the empty test tube (in mg)

m = mass of the sample used for extraction (in g)

Experimental Design

A complete 3^2 factorial design was performed to optimize the solid phase extraction procedure. The variables studied were the solvent volume (hexane) during elution (x_1) and mass of beach sand (x_2). The variables and evaluated ranges were selected based on preliminary studies. For the volume of hexane used, the levels tested were 10 mL (-1), 15 mL (0), and 20 mL (+1); while for the mass of sand packed into the cartridge, the levels were 2 g (-1), 3.5 g (0), and 5 g (+1). The experimental data were processed in Microsoft Excel using a full second-order polynomial model:

$$y = b_0 + b_1x_1 + b_2x_2 + b_{12}x_1x_2 + b_{11}x_1^2 + b_{22}x_2^2 \quad \text{Equation (2)}$$

and the coefficients of the model were calculated by linear regression. A response surface was generated from the estimated model and represented as 3D surface. Thereafter, we performed an ANOVA to verify the fit of the model.

For validation of the developed methodology, a standard sample was subjected to Soxhlet extraction, adapted from method 5520D described by AWWA (APHA, 2017) for the determination of oils and greases in natural waters, following the methodology described by Bejarano et al. (2018)^{2,17}. Ten grams of sand were placed in a cellulose cartridge, and oils were extracted at a rate of 20 cycles per hour, totaling 80 cycles. The volume of hexane used was 150 mL. After extraction, the solvent was evaporated at 70°C using a rotary evaporator, and the flask containing the oil residues was cooled in a desiccator. The oil content was determined by gravimetry, according to Equation 1. The process was performed in triplicate.

Recovery tests were conducted at two levels. The sample studied was spiked with a known amount of oil, and after the optimized extraction process, the oil content was determined by gravimetry. Recovery was calculated according to Equation 3:

$$\text{Rec (\%)} = [(m_2 - m_1)/m_1] \times 100 \quad \text{Equation (3)}$$

Where:

m_1 : mass of oil in the sample before spiking (in mg);

m_2 : mass of oil in the sample after spiking (in mg).

Characterization of Oils Extracted from Beach Sand Samples by FTIR

A Shimadzu spectrophotometer, model IR-TRACER, was used with a wavenumber range of $700 - 4000 \text{ cm}^{-1}$ and a resolution of 4 cm^{-1} . The measurements were performed on potassium bromide (KBr) pellets, prepared as follows: dilution of 0.1 mL of the extracted sample into 0.2 g of KBr, pressed under 12 tons of pressure for 15 minutes. Samples were analyzed in transmittance mode (%) using the Happ-Genzel apodization method. Each scan consisted of a total of 45 scans with a resolution of 4 cm^{-1} over the range of $400 - 4000 \text{ cm}^{-1}$. After measurement, a mathematical correction was applied to the spectrum to subtract peaks related to carbon dioxide and water concentration.

Results and Discussion

Optimization of Oil Extraction Conditions from Beach Sand by SPE

The experimental design and the oil contents obtained are presented in **Table 1**. For a detailed data analysis, one parameter can be fixed at a certain level while evaluating the analytical response with variations in the other studied parameter.

Table 1. Factors, levels, coded matrix and responses for 3^2 factorial design with triplicate in the central point for the solid phase extraction

Test	x_1	x_2	Response teor (g/Kg)
1	-1	-1	2.65
2	-1	0	5.47
3	-1	1	6.21
4	0	-1	0.63
5	0	0	1.67
6	0	1	1.98
7	1	-1	1.05
8	1	0	0.58
9	1	1	0.74
10	0	0	1.62
11	0	0	1.63

Fixing parameter x_1 (sample mass) at level -1 (2 g, trials 1, 2, and 3), it is observed that there is an increase in the analytical response when the solvent volume used increases

from 10 mL to 15 mL (corresponding to a 106% increase in the oil content obtained). When varying to a solvent volume of 20 mL, the increase in the analytical response was approximately 13%. Fixing x_1 at level 0 (3.5 g, trials 4, 5, and 6) there is a 165% increase in the analytical response when the solvent volume changes from 10 mL to 15 mL, and an 18% increase when varying from 15 mL to 20 mL. When fixing x_1 at level +1 (5 g, trials 7, 8, and 9), a 44% decrease in the analytical response is observed with the solvent volume increase from 10 mL to 15 mL, and an increase of 27% when varying the solvent volume from 15 mL to 20 mL.

When fixing parameter x_2 (solvent volume used), it is possible to conclude about the influence of the sample mass on the analytical response obtained. In trials 1, 4, and 7, the solvent volume used was 10 mL (level -1), and it is observed that increasing the sample mass from 2 g to 3.5 g caused a 76% decrease in the analytical response, while the variation from 3.5 g to 5 g generated a 66% increase in the response. However, comparing the oil contents obtained considering the sample mass variation from 2 g to 5 g, there is a 60% decrease in the analytical response. A similar behavior can be observed when fixing x_2 at other levels. With the solvent volume of 15 mL (trials 2, 5, and 8), there is a 69% decrease in the response obtained when varying the sample mass from 2 g to 3.5 g, while the variation from 3.5 g to 5 g caused a 65% decrease. In trials 3, 6, and 9, with the solvent volume fixed at 20 mL, a 68% decrease in the analytical response was observed when varying the sample mass from 2 g to 3.5 g, and 62% when the variation was from 3.5 g to 5 g.

From this analysis, it can be concluded that increasing the solvent volume increases the amount of oil extracted; however, a volume above 15 mL does not present significant gains in absolute terms. Regarding the influence of parameter x_1 , a larger sample mass increases the path the solvent must travel, which may hinder its exit from the solid-phase cartridge, impairing the extraction process and causing a decrease in the oil content extracted. An increase in the amount of sand used necessarily implies an increase in the solvent volume for the extraction of oils and greases from the sample.

The data analysis is supported by the 3D response surface (**Figure 3**) obtained from the mathematical model represented in Equation 4. The mathematical model estimated by linear regression is represented by Equation 4:

$$y = 1.547 - 1.747x_1 + 0.706x_2 + 1.058x_1^2 - 0.103x_2^2 - 0.877x_1x_2 \quad \text{Equation (4)}$$

The standard deviation obtained for the model was 0.026. Analysis of this surface led to the understanding that the best response is achieved with x_1 (sample mass) at level -1 and x_2 (hexane volume) at level 0. Therefore, it was decided to use the variables under these conditions, corresponding to 2 grams of sample packed in the extraction cartridge and the passage of 15 mL of hexane. The ANOVA test showed that the model was well fitted ($F_{\text{calculated}} = 0.340 < F_{\text{critical}} = 2.296$).

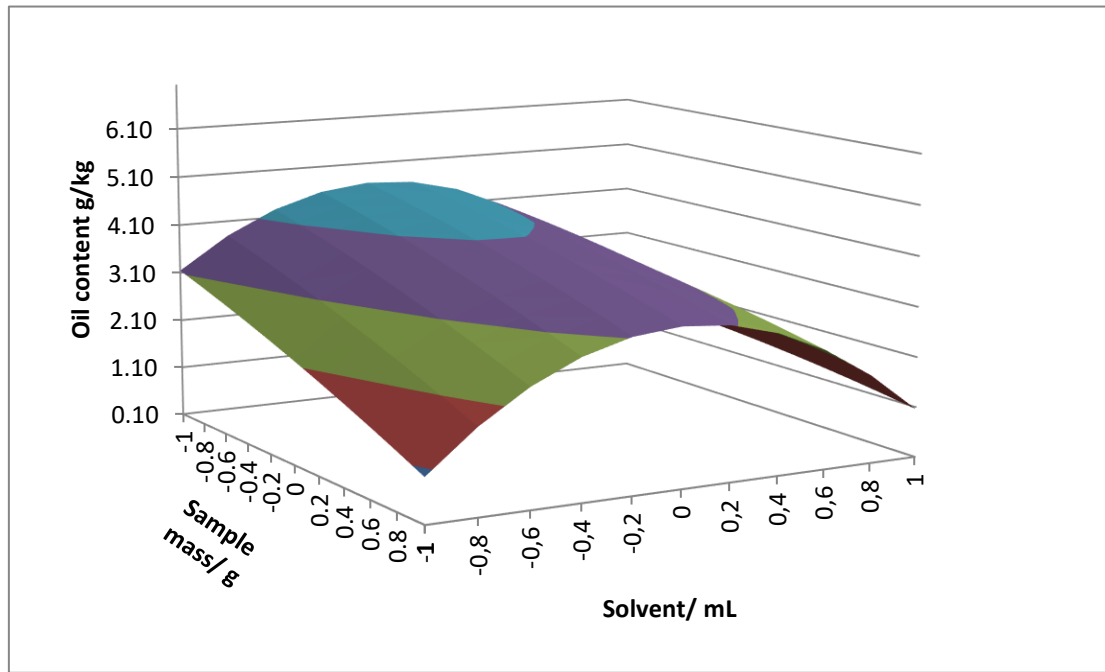


Fig 3 Response surface in a 3D to solid phase extraction

Table 2 shows the recovery results obtained by the proposed method. A sample with a known oils and greases concentration was used in this study, and recovery experiments were conducted at two spiking levels: 0.007 g (lower level) and 0.068 g (upper level) of oil were added to the beach sand. The mass of oil in the studied sample was 0.008 grams.

Table 2. Recovery obtained the proposed method.

Added oil mass/ g	Recovered oil mass/ g	Recovery/ %
0.007	0.007	100
0.068	0.065	96

The proposed method was compared to Soxhlet extraction, adapted from method 5520D described by AWWA (APHA, 2017), a method frequently used for the determination of

oils and greases¹⁷. For this purpose, some samples were subjected to extraction by both methods. The results are shown in **Table 3**. Using the Student's t-test for comparison of means at 95% confidence level, it was found that there is no significant difference between the two methods ($t_{\text{calculated}} = 0.520699 < t_{\text{critical}} = 2.776445$).

Table 3. Comparison between Soxhlet extraction and the proposed method.

Sample	Soxhlet	SPE	relative error
			%
1	2.89	2.93	1.4
2	2.30	2.16	6.0
3	2.60	2.50	3.8
4	1.40	1.49	6.4
5	3.87	3.83	1.0

Quantification of oil content in the sands of Copacabana Beach, Rio de Janeiro, Brazil

Table 4 shows the oil contents found in the sands of Copacabana Beach using the proposed method.

Table 4. Oil content (g/kg) in the sands of Copacabana Beach.

Sampling Area	Location	Oil content (g/kg)
1	Supratidal zone	3.83 ± 0.271
	Middle beach zone	3.49 ± 0.023
	Intertidal zone	1.49 ± 0.006
2	Supratidal zone	1.00 ± 0.001
	Middle beach zone	0.50 ± 0.001
	Intertidal zone	0.50 ± 0.001
3	Supratidal zone	1.49 ± 0.003
	Middle beach zone	1.00 ± 0.001
	Intertidal zone	1.00 ± 0.002
4	Supratidal zone	2.16 ± 0.289
	Middle beach zone	2.50 ± 0.007
	Intertidal zone	1.00 ± 0.003

Analyzing Table 4, a higher concentration of oils can be observed in the supratidal zone and middle beach zone across the four sampling areas. This fact can be justified, among other factors, by the proximity to commercial establishments along the Copacabana beach boardwalk. Frequent contact with oil used in food processing generates residues that can often reach the sandy matrix. Disposable items such as plastic cutlery and paper

napkins containing greasy food residues are often improperly left on the beach sands, which possibly contributes to the increased oil content in these areas. The regions close to the water in all sampling areas present lower oil contents compared to other regions. Among the possible explanations is the fact that there is less food handling by visitors in these regions. It is noteworthy that these regions are constantly under the action of waves, which facilitates the removal of surface oil, carrying it out to the sea. FTIR analyses indicated a strong spectral similarity between lipid residues extracted from beach sand samples and a standard cooking oil sample, with similarity values ranging from 72.1% to 82.5%. The presence of characteristic absorption bands associated with triglyceride-based compounds, such as the C–H stretching (2850–2920 cm^{-1}) and ester carbonyl vibrations ($\sim 1740 \text{ cm}^{-1}$), suggests that the detected lipid fraction is predominantly composed of cooking oil-like substances. Considering the intense tourism and food-related commercial activities in the study area, cooking oil residues likely contribute significantly to the observed contamination, although other anthropogenic sources of lipids cannot be completely ruled out. The comparison of the FTIR spectrum for a beach sand sample superimposed on the FTIR spectrum of a standard cooking oil sample is shown in Figure 4.

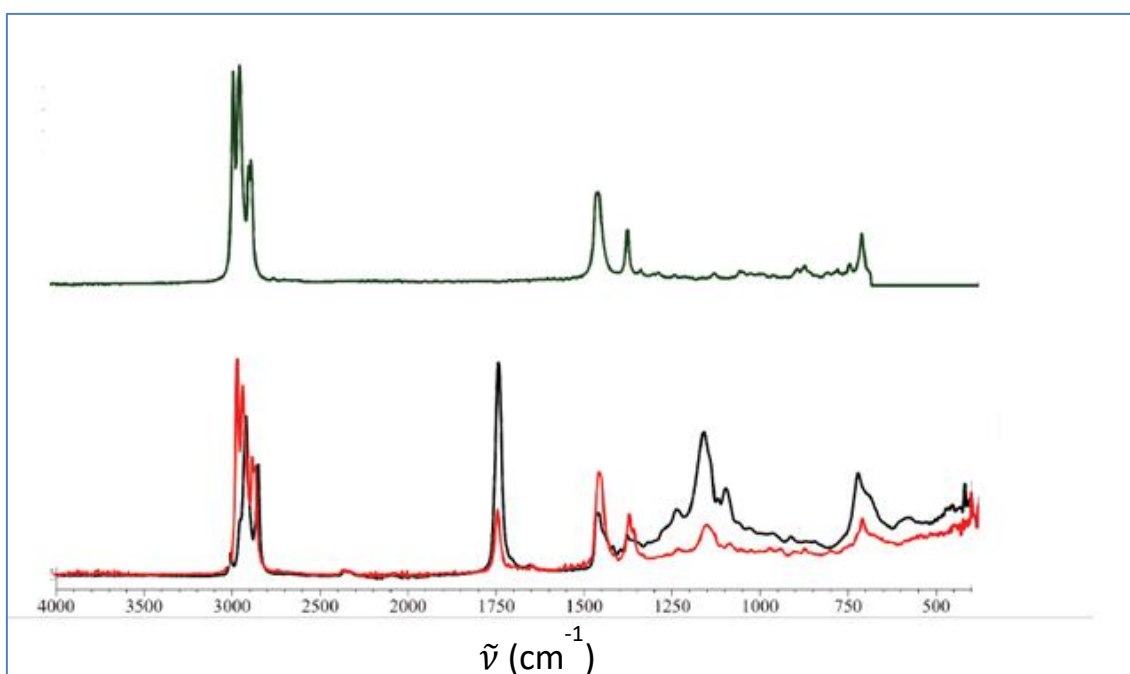


Fig 4 FTIR spectrum of beach sand sample and cooking oil sample
Green: hexane standard.
Black: cooking oil sample.
Red: beach sand sample.

Environmental interpretation of oils and greases contamination in a urban beach sand

In the context of Copacabana Beach, these findings are environmentally plausible due to the intense anthropogenic pressure associated with tourism and coastal urban activities. The beach is characterized by a high daily density of visitors, extensive commercial food services along the shoreline, and continuous human activity, all of which may contribute to the release of lipid-rich residues into the environment. In particular, the improper disposal of used cooking oil by food establishments, street vendors, and temporary beach facilities may represent a significant source of lipid inputs, which can reach the sand through direct deposition or urban runoff.

Although petroleum-derived inputs from maritime activities and urban drainage systems may also contribute to the total oil and grease load, the FTIR spectral features observed in this study appear to be more consistent with oxygenated lipid compounds typically associated with edible oils than with predominantly aliphatic petroleum-derived hydrocarbons. However, due to the inherent limitations of FTIR for source attribution, a definitive distinction between different anthropogenic sources cannot be established.

Overall, the combined gravimetric and spectroscopic evidence suggests that oils and greases contamination in Copacabana Beach sand is primarily driven by anthropogenic inputs related to coastal urban use, with a likely significant contribution from food-related activities. These results highlight the importance of implementing sustainable waste management practices in highly frequented coastal areas.

Conclusion

The results obtained by the gravimetric method coupled with solid-phase extraction (SPE) revealed the presence oils and greases in all beach sand samples analyzed in Copacabana, Rio de Janeiro, Brazil. The concentrations ranged from 0.50 to 3.83 g kg⁻¹, indicating a relevant accumulation of hydrophobic organic compounds in this coastal environment.

From an analytical standpoint, the proposed SPE-gravimetric approach was suitable for the quantification of total extractable oily residues in sandy matrices, showing agreement with the conventional Soxhlet extraction method, as confirmed by Student's t-test ($p > 0.05$). These results support the applicability of the method as a faster and less solvent-intensive alternative for routine monitoring of oils and greases in environmental samples.

The analysis of the studied samples showed that oil contents in the sand were higher in areas closer to the beachfront promenade of Copacabana Beach compared to those in areas under constant wave action, a finding that may be explained by the higher concentration of people consuming food in this area, as well as the transport of oils into the water due to wave action.

FTIR spectra suggest the presence of frying oil residues in the sand, which may be associated with food-related activities in the studied area.

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Conflict of interest

The authors declare no conflicts of interest for this article.

Data availability

No primary research results, software or code have been included and no new data were generated or analysed as part of this review.

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Not applicable

Ethics declaration

Not applicable.

References

American Public Health Association, American Water Works Association, & Water Environment Federation. (2017). *Standard methods for the examination of water and wastewater* (23rd ed.). American Public Health Association.

Amuquandoh, F. E. (2010). Residents' perceptions of the environmental impacts of tourism in the Lake Bosomtwe Basin, Ghana. *Journal of Sustainable Tourism*, 18(2), 223–238. <https://doi.org/10.1080/09669580903298531>.

Baldin, N., & Lopes, R. C. (2009). *Educação ambiental para a reutilização do óleo de cozinha na produção de sabão – Projeto ECOLIMPO*. In *Anais do IX Congresso Nacional de Educação (EDUCERE) e III Encontro Sul Brasileiro de Psicopedagogia* (pp. 1035–1042). Pontifícia Universidade Católica do Paraná.

- Barrionuevo, W. R., & Lanças, F. M. (2001). Extração em fase sólida (SPE) e micro extração em fase sólida (SPME) de piretróides em água. *Química Nova*, 24(2), 172–175. <https://doi.org/10.1590/S0100-40422001000200003>
- Torres-Bejarano, F., González-Márquez, L. C., Díaz-Solano, B., Torregroza-Espinosa, A. C., & Cantero-Rodelo, R. (2018). Effects of beach tourists on bathing water and sand quality at Puerto Velero, Colombia. *Environment, Development and Sustainability*, 20(1), 255–269. <https://doi.org/10.1007/s10668-016-9880-x>.
- Belay, M. H.; Robotti, E.; Ghignone, A.; Fabbris, A.; Brandi, J.; Cecconi, D.; Masini, M. A.; Dondero, F.; Marengo, E. (2025). Sensitive and accurate determination of 32 PFAS in human serum using online SPE-UHPLC-HRMS. *Journal of Hazardous Materials*, 485 136780-136793. DOI: <https://doi.org/10.1016/j.jhazmat.2024.136780>
- Benevides, W. D.; Queiroz, J. K. D.; Silva, V. L.; Vale, M. B. (2018). Destino dos resíduos de óleo de cozinha dos restaurantes na areia de praias do município de João Pessoa, PB. In: *Congresso Brasileiro de Gestão Ambiental e Sustentabilidade*, 6, 251-257. <https://eventos.ecogestaobrasil.net/congestas2018/trabalhos/pdf/congestas2018-et-02-008.pdf>.
- Carvalho, R. G.; Kruk, N. S.; Kawachi, E. Y.; Queiroz, P. I. B. (2019). Um método de ensaio para determinação da concentração de óleo em amostras de águas contaminadas com óleos e graxas. *Engenharia Sanitária e Ambiental*, 24 (3), 515–523. DOI: 10.1590/S1413-41522019127950.
- Dadon, J. R. (2002). El impacto del turismo sobre los recursos naturales costeros en la costa pampeana. En J. R. Dadon & S. D. Matteucci (Eds.), *Zona Costera de la Pampa Argentina*, 101-121.
- De Oliveira, E. B.; Barboza, E. G. (2023). Revisão sistemática dos estudos de permeabilidade em praias usando técnicas computacionais. *Pesquisas em Geociências*, 50 (4). DOI: 10.22456/1807-9806.136152.
- Díaz-Solano, B. H.; Esteller, M. V.; Garrido, H. E. S. (2011). Physicist–chemistry and microbiological water quality in aquatic parks. *Hidrobiologica*, 21 (1), 41–54.
- Embratur. Estudo sobre a preferência dos norte-americanos por destinos de sol e praia. (2022). Brasília: Embratur.
Disponível em: <https://sebrae.com.br/sites/PortalSebrae/artigos/turismo-de-sol-e-praia-os-preferidos-no-brasil,465775b5cbf76810VgnVCM1000001b00320aRCRD>. Acesso em: 19 ago. 2025.
- Florindo, D. N. F.; Freitas, S. G. D.; Rodrigues, M. M. S.; Neto, M. M.; Oliveira, K. S. M.; Santos, P. S. B. (2022). Revisão sistemática da literatura: produção de

biodiesel a partir de óleo residual de fritura. *Research, Society and Development*, 11(5), 1–14.

Gireli, E. K. B.; Moreira, L. S.; Provete, C. S.; Cordeiro, S. G.; Brandão, G. P.; Carneiro, M. T. W. D. (2025). Análise comparativa entre coprecipitação e extração em fase sólida para determinação de elementos traço em amostras de água do mar por ICP-MS. *Revista Ifes Ciência*, 11 (1), 1–16. DOI: 10.36524/ric.v11i1.2898.

Honorio, G. G.; Cunha, J. N. da; Assis, K. L. S. C.; Aguiar, P. F.; Andrade, D. F.; Souza, C. G.; D’Avila, L. A.; Archanjo, B. S.; Achete, C. A.; Pradelle, R. N. C.; Turcovics, F.; Neto, R. S.; D’Elia, E. (2019). Free glycerol determination in biodiesel samples using palladium nanoparticles modified glassy carbon electrode associated with solid phase extraction. *Journal of Solid State Electrochemistry*, 23 (11), 3057–3066. DOI: 10.1007/s10008-019-04387-2.

Huettel, M. (2022) Oil pollution of beaches: processes and fate of stranded petroleum hydrocarbons. *Science of the Total Environment*, 36, 100803 – 100812. DOI: <https://doi.org/10.1016/j.coche.2022.100803>

Karsi, M. B. B. (2025). Investigation of oil and grease in surface soils of gas station, automobile repair workshop, urban, recreational area, and rural sites using FT-IR. *Accreditation and Quality Assurance* 30, 153–165. DOI <https://doi.org/10.1007/s00769-024-01624-8>

Khorshid, Z. B.; Doroodmand, M. M.; Abdollahi, S. (2020). UV-Vis spectrophotometric method for oil and grease determination in water, soil and different mediates based on emulsion. *Microchemical Journal*, 160, 105620. DOI: 10.1016/j.microc.2020.105620.

Khozanah; Yogaswara, D.; Wulandari, I. (2021) Oil and Grease (OG) content in seawater and sediment of the Jakarta Bay and its surrounding. *IOP Conference Series: Earth and Environmental Science*, 789, 012015. DOI: 10.1088/1755-1315/789/1/012015.

Kich, M.; Böckel, W. J. (2017). Análise de óleos e graxas em efluentes por espectrofotometria. *Macapá*, 7 (3), 61–69. DOI: 10.18468/estcien.2017v7n3.p61-69.

- King, S. M.; Leaf, P. A.; Olson, A. C.; Ray, P. Z.; Tarr, M. A. (2014). Photolytic and photocatalytic degradation of surface oil from the Deepwater Horizon spill. *Chemosphere*, 95, 415–422. DOI: 10.1016/j.chemosphere.2013.09.060

Lanças, F. M. (2009). *Cromatografia Líquida Moderna*; Ed. Átomo: Campinas.

Millioli, V. S.; Freire, D. D. C.; Cammarota, M. C. (2004). Tratamento de areia de praia contaminada por óleo cru utilizando reagente de Fenton. CETEM/MCT, 38 p., Rio de Janeiro.

- Moreira, B. M.; Cunha, J. N. da; Paiva, V. M.; Honório, G. G.; Tarley, R. T.; D'Elia, E. (2020). Determination of Free Glycerol in Biodiesel by Molecularly Imprinted Polymer – Based Solid-Phase Extraction (SPE) Using an Enzymatic Spectrophotometric Assay. *Analytical Letters*, 54 (10), 1654–1667. DOI: 10.1080/00032719.2020.1821040.
- Oliveira, E. B.; Nicolodi, J. L. (2017). Oil permeability variations on lagoon sand beaches in the Patos-Guaíba system in Rio Grande do Sul, Brazil. *Marine Pollution Bulletin*, 115 (2), 154–163. DOI: 10.1016/j.marpolbul.2016.12.020.
- Prefeitura da Cidade do Rio de Janeiro. Rio é a quinta cidade com maior crescimento do PIB do turismo no mundo. Publicado em 01 dez. 2022. Atualizado em 02 dez. 2022. Disponível em: <https://prefeitura.rio/setur/rio-e-a-quinta-cidade-com-maior-crescimento-do-pib-do-turismo-no-mundo/>. Acesso em: 09 jun. 2025.
- Rauckyte, T.; Žak, S.; Pawlak, Z.; Oloyede, A. (2010). Determination of oil and grease, total petroleum hydrocarbons and volatile aromatic compounds in soil and sediment samples. *Journal of Environmental Engineering and Landscape Management*, 18 (3), 163–169. DOI: 10.3846/jeelm.2010.19.
- Rodriguez, J.; Ruiz, J. (2010). Conservación y protección de ecosistemas marinos: conceptos, herramientas y ejemplos de actuaciones. *Ecosistemas*, 19 (2), 5–23.
- Rowland, D. K.; Lindley, A. P.; Hall, G. P.; Rossal, M. J.; Wilson, D. R.; Benham, D. G.; Harrison, A. F.; Daniels, R. E. (2000). Effects of beach sand properties, temperature and rainfall on the degradation rates of oil in buried oil/beach sand mixtures. *Environmental Pollution*, 109, 109–118. DOI: 10.1016/S0269-7491(99)00224-9.
- U.S. Environmental Protection Agency. (1998, April). *Method 9071B: n-Hexane extractable material (HEM) for sludge, sediment, and solid samples*. In *Test methods for evaluating solid waste, physical/chemical methods* (EPA Publication SW-846, 3rd ed., Final Update IIIA). U.S. Environmental Protection Agency. <https://www.epa.gov/hw-sw846/sw-846-test-method-9071b-n-hexane-extractable-material-hem-sludge-sediment-and-solid>
- Valentine, D. L.; Mezic, I.; Macesic, S.; Crnjaric-Zic, N.; Ivic, S.; Hogan, P. J.; Fonoberov, V. A.; Loire, S. (2012). Dynamic autoinoculation and the microbial ecology of a deep water hydrocarbon irruption. *Proceedings of the National Academy of Sciences of the United States of America*, 109, 20286–20291.