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Dissolved/dispersed polycyclic aromatic hydrocarbon spatial and temporal changes in the Western Gulf of Mexico

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Oil extraction and transport activities in the Gulf of Mexico (GoM), along with major marine oil spills and riverine inputs, are exerting environmental pressure on this system by increasing the concentration of oil-related pollutants such as polycyclic aromatic hydrocarbons (PAHs). To fully identify these changes related to oil activities, current PAH levels should be established. Here, we present the PAH concentration and the low molecular weight/high molecular weight (LMW/HMW) ratios obtained in the Perdido Fold Belt area in surface and bottom water at four cruises from May 2016 to September 2017. The Perdido 1 (P1) cruise was conducted in May 2016, the Perdido 2 (P2) cruise in September–October 2016, the Perdido 3 (P3) cruise in June 2017, and the Perdido 4 (P4) cruise in September 2017. Samples were taken during each cruise at up to 3,500-m depth, the deepest ever recorded for the GoM. Results show that the highest concentrations of PAH, LMW PAHs, and HMW PAHs were found in the P4 cruise (1.15, 1.05, and 0.10 µg/L, respectively), well below the 300 µg/L guideline for acute exposure. LMW/HMW ratios show that only the P1 cruise indicates pyrogenic hydrocarbons, while P2, P3, and P4 were petrogenic. The spatial distribution of total PAH, LMW, and HMW showed higher values in the southern and northeastern areas, except for P4, which showed high values related to riverine inputs. The complex hydrodynamic in the region was found to have a significant effect on PAH seasonal changes, river contributions, eddy circulation, and fronts to promote their dispersion.

KEYWORDS

perdido fold belt, dissolved PAH, deep gulf, baseline, environmental assessment

Introduction

The Gulf of Mexico (GoM) is one of the largest marine ecosystems in the world (Yáñez-Arancibia and Day 2004a; Toledo Ocampo, 2005), supporting important ecosystems as well as economic activities such as tourism, fisheries, and the extraction of hydrocarbons. Specifically, in the Western GoM, the Perdido Fold Belt (PFB) is located in the deep waters of the exclusive economic zones of the USA and Mexico, which has the structural capacity to contain oil and gas reservoirs (Patiño Ruiz et al., 2003). Population increase creates further stress on the environment, particularly on the coast of the Gulf of Mexico. The two closest Mexican states to our study zone are Tamaulipas, with a population in 2020 of 3,527,735 inhabitants, and Veracruz, with a 2020 population of 8,062,579 inhabitants (Instituto Nacional de Estadística e Informática [INEGI], 2020).

The presence of oil in the southern GoM is common since there are natural oil seeps (locally known in Mexico as “chapopoterías”), mainly located in the southwestern part of the GoM, where also lies the main Mexican oil extraction industry (Miranda et al., 2004; Murawski et al., 2018). Mitchel et al. (1999); National Research Council Committee on Oil in the Sea (2003), and MacDonald et al. (2015) mentioned that the natural oil input to the GoM ranges between 250,000 and 1.4 million barrels per year. Also, large-scale exploration, transport (Wankhede, 2019), and refining of hydrocarbons are present in its coasts and deep waters (Yáñez-Arancibia and Day 2004a and Yáñez-Arancibia and Day 2004b), since the Burgos basin is the main oil province producing non-associated gas in Mexico (PEMEX, 2013). In addition, extractive activities, transport activities, and also pollutant input by rivers significantly contribute to hydrocarbon concentrations in the GoM (Gracia et al., 2014; Gracia et al., 2016a; Gracia et al., 2016b). Also, two of the main marine oil spills have occurred in the GoM: Ixtoc 1 in 1979–1980 and the Deepwater Horizon in 2010 (Gracia et al., 2016a). Interest in oil pollution in the GoM increased after the Deepwater Horizon accident in 2010, leading to the creation of the Gulf of Mexico Research Initiative (GoMRI), which channeled \$300 million to research the spill. The Mexican federal government also funded three series of oceanographic cruises, covering all the GoM once per year in 2010, 2011, and 2012.

Extraction activities exert significant pressure on marine and coastal areas in addition to being a source of different pollutants, whose impact on the natural environment (Salcedo et al., 2017; Soto et al., 2017) is evidenced by the decline of coastal and marine water quality (Botello et al., 2015). Specifically, regarding crude oil, the most toxic compounds that constitute it are the polycyclic aromatic hydrocarbons (PAHs), which are present in 3% to 7% of the crude oil, but they are recognized as carcinogenic and toxic to the environment (ATSDR (Agency for Toxic Substances and Disease Registry), 2005). These PAHs, once released to the water column, could be transported (dispersed or accumulated); in the area, there is a tendency to

dispersion related to a constant and rapid forcing of surface water and eddy circulation (Luo et al., 2016; Enríquez et al., 2017; Meza-Padilla et al., 2019). Therefore, transportation to and from the coastal zone is expected, but there is also the presence of eddies and fronts caused by vertical mixing, seasonal changes, and river input. Therefore, the present research aimed to determine the presence and space and time changes of dissolved/dispersed PAHs in the Perdido Fold Belt area in two consecutive years and seasons to evaluate the current pollution status and the processes related to it. In particular, concentrations of dissolved/dispersed PAHs are reported for the first time in the deep areas (over 1,000-m depth) of the GoM.

Materials and methods

The PFB area occupies approximately 27,230 km² in the Western GoM. It is located within the Deep Gulf of Mexico Oil Province (PEMEX, 2013; CNH, 2015) and is home to numerous active systems that produce oil and gas emissions (CNH, 2015).

Water samples were collected in four oceanographic cruises: Perdido 1 (P1), Perdido 2 (P2), Perdido 3 (P3), and Perdido 4 (P4), onboard the B/O *Justo Sierra* along the Western Gulf of Mexico. Water samples at the surface (5 m) and the bottom were collected at 27 sampling stations during cruise P1 in May 2016, P2 September–October 2016, P3 in June 2017, and P4 in September 2017 (Table 1). Cruises in May or June are considered to be in the dry season, and those in September or October are considered to be in the rainy season.

Water samples from the surface (between 1 and 10 m) and bottom (see Figure 1 for depth reference) were collected in 2-L glass bottles previously cleaned with gas chromatography (GC)-grade hexane (Omnisolv, Sigma-Aldrich Corp., St. Louis, MO, USA), extracted on board (see Herzka et al., 2017 for complete methods), and the extracts were taken to the Marine Geochemistry laboratory (Cinvestav, Merida) for further analysis following the method of Zhendi et al. (1994) (for full methods, please refer to Herguera et al., 2017). Briefly, extracts were separated into fractions in an activated silica gel column. The aromatic fraction was eluted with a 50/50 (vol/vol) mixture of GC-grade dichloromethane and hexane (Omnisolv). Analysis was performed in a Perkin Elmer Clarus 500 GC–mass spectrometry (GC-MS) in selected ion monitoring (SIM) mode. Helium was used as a carrier (1.0 ml/min), injector temperature was 290°C with an initial ramp of 25°C to 160°C and a second ramp of 8°C/min to 290°C with a final time of 15 min. A DB% (30 m × 0.25 mm ID, 0.25 μm film) was used. The PAH standards were from Ultra Scientific (North Kingstown, RI, USA). Internal and surrogate standards (terphenyl-*d*₁₄, acenaphthene-*d*₁₀, phenanthrene-*d*₁₀, chrysene-*d*₁₂, perylene-*d*₁₂, and pyrene-*d*₁₀) as well as external standards were obtained from Ultra Scientific. The limits of detection were between 0.003 and 0.0027 μg/L for individual compounds.

TABLE 1 Sampling stations for all four cruises.

Station	Latitude (degrees north)	Longitude (degrees west)	Depth (m)
B1	25.6242	-96.8412	50.4
B2	25.6255	-96.5115	97.6
B3	25.7406	-96.2548	372.5
B4	25.4595	-96.1125	1,000.0
B5	25.6230	-95.4812	1,448.0
B6	25.6408	-95.4115	1,998.0
C1	25.2664	-97.0334	47.0
C2	25.2519	-96.6985	105.8
C3	25.2492	-96.3490	503.8
C5	25.2508	-95.9295	1,644.5
C6	25.2508	-95.6368	1,971.0
C7	25.2505	-95.3462	2,676.8
D1	24.8688	-97.2857	44.0
D2	24.8710	-96.8917	107.0
D3	24.8701	-96.5999	473.4
D5	24.8723	-96.1100	1,280.0
D6	24.8737	-95.9042	2,215.0
E7	24.4978	-95.6681	2,984.7
F1	23.9944	-97.5335	49.7
F2	24.0000	-97.3060	93.0
F3	24.0065	-97.0845	537.0
F4	24.0020	-96.6835	1,616.0
F5	24.0101	-96.4269	1,760.0
F7	23.9990	-95.6100	3,254.7
F8	24.0025	-95.1752	3,548.0
I	24.9922	-96.2857	1,065.7
III	24.7635	-96.4994	826.0

Recoveries of surrogate compounds were between 60% and 120%.

Distribution maps were performed with “Ocean Data View” version 5.3 (Schlitzer, 2020). Effect sizes were calculated using the package “sjstats” version 0.18.0 (Lüdecke, 2020) in R version 4.0.2 (R Core Team, 2020).

Results and discussion

The highest mean PAH, LMW PAH, and HMW PAH concentrations were found in the Perdido 4 cruise, whereas the lowest mean concentrations for the three fractions were

found in the second cruise (Table 2). The highest concentrations of PAH, LMW PAHs, and HMW PAHs were found in the Perdido 4 cruise (1.15, 1.05, and 0.10 $\mu\text{g/L}$, respectively). The lowest concentrations of PAH, LMW, and HMW were below the detection limit on cruises Perdido 2 to Perdido 4.

To test for the effect sizes of the two categorical variables (“cruise” and “depth”), F-tests produced low values (lower than 1) for the factor “depth” and the interaction term in a two-way ANOVA, suggesting that temporal differences were more important than depth differences, and thus the focus here is on the F-tests for factor “cruise” only. The ω^2 effect size was chosen because ω^2 effect sizes indicate the proportion of variance explained by the independent variables and is a less biased effect

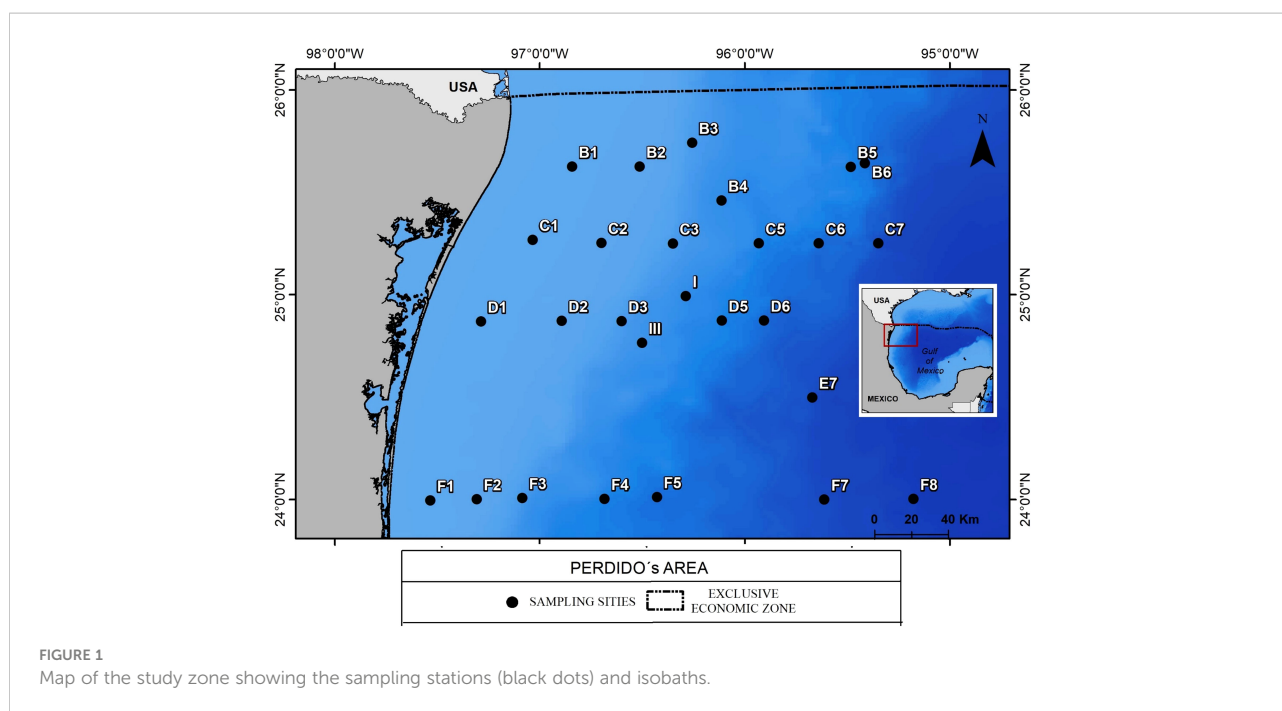


TABLE 2 Mean total PAH, low molecular weight (LMW), and high molecular weight (HMW) PAH concentrations (in $\mu\text{g/L}$) (\pm standard error of the mean) of each cruise.

Cruise	PAH [$\mu\text{g/L}$]	LMW PAH [$\mu\text{g/L}$]	HMW PAH [$\mu\text{g/L}$]
Perdido 1	0.02 (\pm 0.002)	0.005 (\pm 0.001)	0.015 (\pm 0.0005)
Perdido 2	0.0006 (\pm 0.0001)	0.0004 (\pm 0.00008)	0.0002 (\pm 0.00004)
Perdido 3	0.007 (\pm 0.007)	0.004 (\pm 0.0003)	0.003 (\pm 0.0006)
Perdido 4	0.09 (\pm 0.03)	0.08 (\pm 0.03)	0.011 (\pm 0.003)

PAH, polycyclic aromatic hydrocarbon.

size measure than η^2 (Nakagawa and Cuthill, 2007; Lakens, 2013; Calin-Jageman and Cumming, 2019). Percent variance explained by the factor “cruise” for PAH, LMW PAHs, and HMW PAHs is low in all cases, with 22% for HMW being the highest (Table 3). The 95% confidence interval is also higher for the HMW PAH fraction, which indicates a higher dispersion of values. The 95% confidence interval for ω^2 does not include zero for any of the three fractions, which means a “significant” difference in the usual hypothesis testing framework.

Based on differences in their physical characteristics and environmental behavior, some PAH indices could suggest their origin. One such index is the ratio of low-molecular-weight (LMW PAHs with 2 or 3 benzene rings) to high-molecular-weight compounds (HMW, four or more benzene rings). A ratio higher than 1 indicates a petrogenic origin (PAH originates from petroleum), and a ratio lower than 1 indicates a pyrogenic origin (from incomplete combustion of organic matter) (Soclo et al., 2000; Magi et al., 2002; Freitas da Silva et al., 2007). LMW/HMW

TABLE 3 F-test, effect size (ω^2), minimum, maximum, and 95% confidence interval for the “cruise” factor in a two-way ANOVA for the three PAH fractions.

Fraction	$F_{3,54}$	ω^2	Minimum	Maximum	95% CI
PAH	7.3	0.082	0.013	0.156	0.143
LMW	7.6	0.085	0.015	0.16	0.145
HMW	21.3	0.224	0.125	0.313	0.188

PAH, polycyclic aromatic hydrocarbon; LMW, low molecular weight; HMW, high molecular weight.

ratios were below 1 for the P1 cruise (Table 4), and the ratio was higher than 1 for the other three cruises, indicating that these compounds are from petroleum inputs.

Percent variance explained by the factor “cruise” for the LMW/HMW ratio, as indicated by ω^2 , is low at 8% (Table 5). The 95% confidence interval for ω^2 does not include zero for any of the three fractions, which means a “significant” difference in the usual hypothesis testing framework.

The spatial distribution of total PAH, LMW, and HMW (Figure 2) shows a consistent spatial trend for the surface and bottom samples, with higher values in the southern and northeastern parts of the study zone, except for the P4 cruise, which shows high values in front of the Mexican Laguna Madre. There is no obvious oceanographic feature to explain this (Figure 3), although Luo et al. (2016); Enríquez et al. (2017); Meza-Padilla et al. (2019), and Arcega-Cabrera et al. (2021) mentioned that the transport to and from the coastal zone is expected to be related to river input, vertical mixing, and seasonal changes. There is a secondary maximum for the P1 cruise across the Mexican Laguna Madre for total and low-molecular-weight PAH, which coincides with lower salinity values (Figure 3), suggesting a terrestrial input. This agrees with the mean LMW/HMW ratio for this cruise (0.313, Table 3), which indicates a pyrogenic origin for PAH in this cruise. This same pattern was shown by Arcega-Cabrera and Dótor-Almazán (2021), using the fluoranthene/fluoranthene+pyrene index, finding that for surface

TABLE 5 F-test, effect size (ω^2), minimum, maximum, and 95% confidence interval for the “cruise” factor in a one-way ANOVA for the LMW/HMW ratio.

Term	$F_{3,212}$	ω^2	Minimum	Maximum	95% CI
Cruise	5.41	0.082	0.013	0.156	0.143

LMW, low molecular weight; HMW, high molecular weight.

and near the bottom water, PAHs from oil source (petrogenic) and from wood, plants, and mineral carbon (pyrogenic) were corroborated, agreeing with our results.

The dissolved/dispersed PAH concentrations obtained in this study are higher than those reported by Botello et al. (2015) for the coast of Tamaulipas on two cruises made in 2010 and 2011, right after the Deepwater Horizon spill. Botello et al. (2015) reported that all water samples they collected had total PAH concentrations below their detection limit (around 0.03 $\mu\text{g/L}$), while in this study, they were in an approximate range from undetected to 1.15 $\mu\text{g/L}$. For the Northern Gulf of Mexico, Wade et al. (2016) analyzed the results of more than 20,000 water samples collected before and after the Macondo well spill. They reported that 84% of the samples analyzed had results lower than 1 $\mu\text{g/L}$ and consider this to be the “natural” or “background” concentration for the Gulf of Mexico. Wade et al. (2016) also reported that 79% of the samples analyzed had concentrations of less than 0.056 $\mu\text{g/L}$, while in this study, they were in an approximate range from undetected to 1.15 $\mu\text{g/L}$. To assess possible risk to marine organisms, the concentrations found in this study for total, LMW, and HMW PAHs were compared to the guidelines suggested in the Screening Quick Reference Tables (Buchman, 2008). Only the acute exposure level is reported for marine waters, and it is the same for all three fractions, 300 $\mu\text{g/L}$. This value is higher than the maximum concentrations reported here, and thus, the risk to aquatic organisms is very low.

Differences for the previous and actual concentrations of hydrocarbons, and in particular PAH, can be caused by several factors, from differences in analytical methods (although analytical quality controls assure the quality of results diminishing the significance of this factor) or sampling times up to real regional differences for the concentrations of these compounds. Hydrocarbon analyses in any of its fractions in the GoM are complicated by the presence of many natural seeps, known in Mexico as “chapopoterías”. MacDonald et al. (2015) reported the presence of 914 natural seeps in the Gulf of Mexico, which in total discharge to the surface 2.5 to $9.4 \times 10^4 \text{ m}^3 \text{ year}^{-1}$, while they calculated that the Macondo spill in 2010 discharged $22.6 \times 10^3 \text{ m}^3$ to the surface. The high input of hydrocarbons from natural sources to the Gulf of Mexico produces a high background concentration, which makes it more difficult to assess human impacts.

Also, in the Perdido Fold Belt area, there are contributions of agricultural and industrial activities, as well as wastewater inputs

TABLE 4 LMW/HMW ratios for all four Perdido cruises.

	Cruise	LMW/HMW
Mean	P1	0.313
	P2	2.21
	P3	1.4
	P4	1.77
Standard error of the mean	P1	0.0646
	P2	0.559
	P3	0.153
	P4	0.384
Minimum	P1	0.01
	P2	0
	P3	0
	P4	0
Maximum	P1	2.61
	P2	17
	P3	4.28
	P4	12.2

LMW, low molecular weight; HMW, high molecular weight.

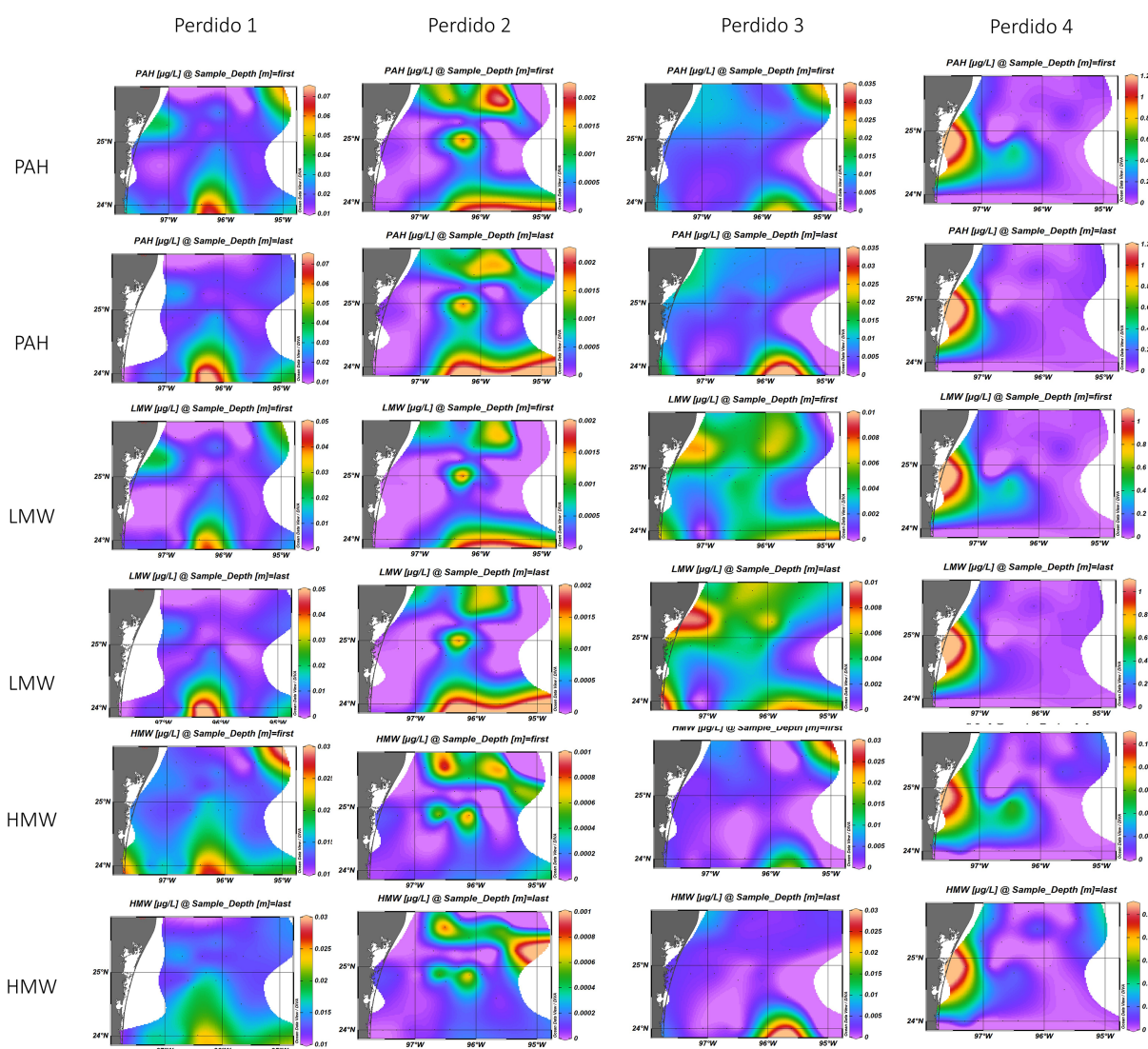


FIGURE 2

Spatial distribution of total polycyclic aromatic hydrocarbons (PAHs), LMW (low molecular weight), and HMW (high molecular weight) for the Perdido 1, 2, 3, and 4 cruises. First = surface sample (ca. 1–10 m); Last = bottom sample (please refer to Figure 1 for approximated depth).

through local river systems (Arcega-Cabrera et al., 2021), and according to Reilly et al. (1991); Le Blanc (1994), and Murawski et al. (2020), there are significant hydrocarbon and metal inputs from the discharge of $140 \times 10^6 \text{ m}^3$ of production wastewater from approximately 3,000 platforms in the GoM. Also, Vidal-Martinez (2021) showed that some of the sampled fish in the area had a medium-to-high metabolite concentration related to exposure and probable damage from PAH that could be stressful for the organisms.

In addition to this, an intriguing possibility is that spatial and temporal differences observed in hydrocarbons and other pollutant concentrations in the Western Gulf of Mexico may be due, at least in part, to east-to-west transport by eddies that promote a rapid and constant forcing of surface water (Luo et al.,

2016; Enríquez et al., 2017 and Meza-Padilla et al., 2019; Guerrero et al., 2020). Therefore, transport to and from the coastal zone is common in this area caused by the vertical mixing and seasonal changes in contributions from river discharges as reported by Arcega-Cabrera et al. (2021) for metals. In addition, a continental water influence, including contributions from the continental shelf and southern part of the GoM, has also been reported by Enríquez et al. (2017) and Meza-Padilla et al. (2019).

Transport in the deep gulf by eddies has been inferred from modeling studies and observational campaigns (Morey et al., 2020). During the time of the Perdido cruises, there were two active eddies in the Western Gulf of Mexico, the Olympus (active from June 2015 to June 2016) and Poseidon (active from April 2016 to April 2017) eddies (Woods Hole Group, 2020).

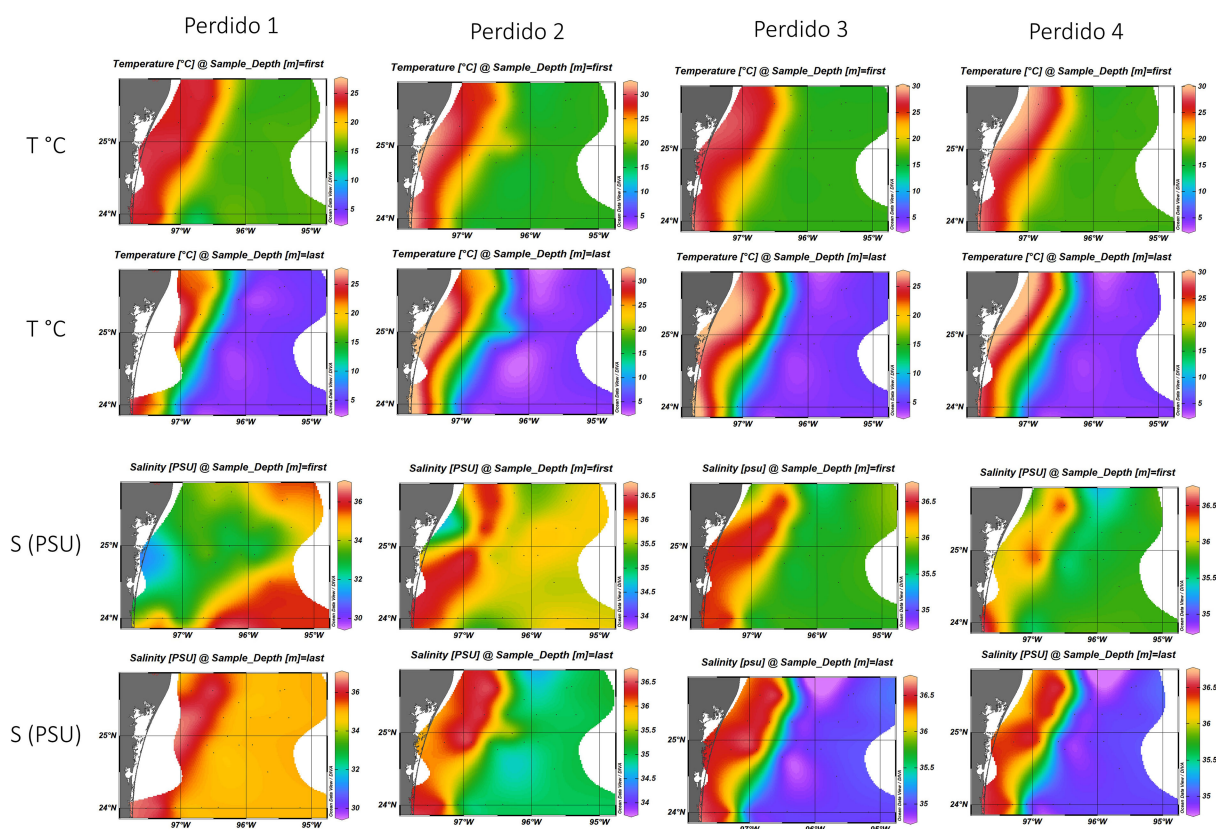


FIGURE 3
Spatial temperature and salinity variations for the Perdido 1, 2, 3, and 4 cruises. First = surface sample (ca. 1–10 m); Last = bottom sample (please refer to Figure 1 for approximated depth).

All of the abovementioned factors could be promoting the PAH observed seasonal changes, causing dispersion of the PAH by being forced from river contributions and also eddy circulation and fronts.

Conclusions

PAH concentration showed significant differences between cruises (seasons), and some of the samples showed a higher concentration than the ones previously reported. These seasonal changes could be related to the presence of natural seeps, riverine input, and oil extraction and transport activities. Therefore, these factors could be promoting changes in the system that could turn out to be stressful for the system at the GoM.

The source of polycyclic aromatic hydrocarbons was mainly petrogenic with exception of the first cruise, suggesting that inputs are mainly from the oil industry in the area, although

riverine input is also present and varies according to hydrological season.

The complex hydrodynamic and diverse sources inputs in the Perdido area are directing the seasonal changes of PAH. Transport from and to the coast, eddies, and fronts are working together to disperse the PAH from riverine input and oil extraction and transport activities.

The results of this research constitute an invaluable reference for PAH behavior at the GoM given the spatial, time, and economic effort made with the extensive sampling; this allows us to achieve a set of robust data, although further research should be performed to better understand the processes affecting the concentration and transport of hydrocarbons in the complex and environmentally relevant system of the GoM.

Data availability statement

The raw data supporting the conclusions of this article will be made available by the authors, without undue reservation.

Author contributions

GG-B designed the sampling scheme; GG-B and FA-C did most of data analysis; all authors contributed to writing the manuscript; VC-M did most of sample analysis. All authors contributed to the article and approved the submitted version.

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Conflict of interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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