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A novel method to evaluate chemical concentrations in muddy and sandy coastal regions before and after oil exposures☆

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ABSTRACT

Oil spills can result in changes in chemical concentrations along coastlines. In prior work, these concentration changes were used to evaluate the date sediment was impacted by oil (i.e., oil exposure date). The objective of the current study was to build upon prior work by using the oil exposure date to compute oil spill chemical (OSC) concentrations in shoreline sediments before and after exposure. The new method was applied to OSC concentration measures collected during the Deepwater Horizon oil spill with an emphasis on evaluating before and after concentrations in muddy versus sandy regions. The procedure defined a grid that overlaid coastal areas with chemical concentration measurement locations. These grids were then aggregated into clusters to allow the assignment of chemical concentration measurements to a uniform coastal type. Performance of the method was illustrated for ten chemicals individually by cluster, and collectively for all chemicals and all clusters. Results show statistically significant differences between chemical concentrations before and after the calculated oil exposure dates ($p < 0.04$ for each of the 10 chemicals within the identified clusters). When aggregating all chemical measures collectively across all clusters, chemical concentrations were lower before oil exposure in comparison to after ($p < 0.0001$). Sandy coastlines exhibited lower chemical concentrations relative to muddy coastlines ($p < 0.0001$). Overall, the method developed is a useful first step for establishing baseline chemical concentrations and for assessing the impacts of disasters on sediment quality within different coastline types. Results may be also useful for assessing added ecological and human health risks associated with oil spills.

Author statement

Junfei Xia: Conceptualization, Methodology, Software, Validation, Formal analysis, Writing – original draft, Visualization. Wei Zhang: Conceptualization, Methodology, Writing – review & editing. Alesia C. Ferguson: Conceptualization, Writing – review & editing. Kristina D Mena: Conceptualization, Writing – review & editing. Tamay M Özgökmen: Conceptualization, Methodology, Writing – review & editing. Helena M. Solo-Gabriele: Conceptualization, Methodology, Writing – review & editing, Project administration, Funding acquisition.

1. Introduction

Clarifying impacts of oil on coastal sediment concentrations is important for assessing damage from spills. For example, studies about sensitive habitats (Bae et al., 2018) and public health

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advisories (McCready and Williams 2011; Ferguson and Solo-Gabriele 2016; Ferguson et al., 2017) need detail about chemical concentrations and their distribution in space and time to assess ecological and human health risks as a result of an oil spill (Farrington 2014). In addition to the impacts from oil, impacts of oil on sediment can be influenced by the characteristics of the coastline (Al-Majed et al., 2012) with muddy shorelines showing different impacts than sandy shorelines.

Exposure of shoreline sediments to oil was observed along the Gulf of Mexico (GoM) coast during and after one of the largest oil spills in history, the Deepwater Horizon (DWH) oil spill, that occurred on April 20, 2010 and led to an 87-day continuous and uncontrolled leak of the Macondo oil well, located approximately 65 km southeast from the nearest point on the Mississippi River Delta (Kujawinski et al., 2011; Freudenburg and Gramling 2011; Black et al., 2016). The leak was controlled after a capping stack was installed on the wellhead on July 15, 2010. The total oil released was $4.4 \times 10^6 \pm 20\%$ barrels (about 700,000 m^3) (McNutt et al., 2012). The majority of the coastline impacts were towards the northern direction coupled with longshore currents that transported oil towards the northwest and northeast resulting in visible impacts from eastern Texas through the western Florida coastlines (Liu et al., 2013; Özgökmen et al., 2016). Collection of sediment samples was initiated on April 29, 2010, 9 days after the explosion. The sampling process lasted until June 7, 2012 (BP Gulf Science Data, 2016) providing a large dataset from which to assess the impacts to shoreline sediments.

Although there was a wealth of data collected, comprehensive studies about chemical concentration changes in coastal sediments during the DWH oil spill were limited. Part of the difficulty in conducting comprehensive studies about concentration changes in coastal sediments is due to the large magnitude of the DWH oil spill, as both large and small scale ocean circulation features impacted the movement of the oil such that different Gulf regions were impacted on different dates (Androulidakis et al., 2018) resulting in a patchy distribution of oil landfall (Rouhani et al., 2017). Although Shoreline Cleanup and Assessment Technique (SCAT) reports provide the distributions of visible oil impacts over space and estimate the impact of oil by field observation and geospatial data (Owens and Sergy 2003; Taylor and Reimer 2008), they do not provide the chemical concentration data nor quantified oil impact levels. Additionally most of the SCAT ground observation reports for DWH in muddy coastlines were identified as no oil observed (from ERMA website https://erma.noaa.gov/gulfofmexico/), but from sampling data, the chemical concentrations were detected, and in many cases, at relatively high levels (BP Gulf Science Data, 2016). This discrepancy is likely because low level chemical impacts from oil spills are not visible to the human eye and can go undetected unless measurements are taken (Day et al., 2020; Turner et al., 2019).

Another difficulty in estimating the chemical concentration changes due to oil spills was that the date of impact of the oil spill was different in different regions. So separating a time series of chemical measures to compute before and after impact was not obvious. Previous research about shoreline exposure was mostly based on models and remote sensing images (Price et al., 2006; Özgökmen et al., 2014; Berta et al., 2015; Xu et al., 2016). These methods were very useful when evaluating the offshore distribution of oil. However, in the nearshore, models become more limited due to different processes governing nearshore hydrodynamics resulting in errors when applying offshore models to nearshore areas (Evans et al., 2017). Remote sensing data is available on a daily basis (when cloud conditions are suitable) and is spatially distributed. However, it suffers from its high resolution (30-m scale) which does not provide information on the closeness to the shoreline needed to determine if actual beaching had occurred (Balogun et al., 2020; Mo et al., 2017).

Due to the large spatial and temporal extent and lack of sensitivity of visual methods to detect oil a comprehensive analysis of the impacts of the DWH oil spill to coastal sediments is lacking. Such information is needed to assess how the oil may have increased ecological and human health risks along impacted coastlines. The objective of this study was to develop a method to estimate chemical concentrations in shoreline sediments before and after oil exposure. The readily available and extensive dataset collected during and after the DWH oil spill was utilized to illustrate the performance of the method. Ten chemicals were identified to assess oil spill shoreline impacts, based upon the ability to measure statistical differences in concentrations. The chemical concentrations before and after oil exposure were evaluated for these individual chemicals as well as for the aggregated set of chemicals in both muddy and sandy coastlines. The study approach was designed around two hypotheses. The first hypothesis was that lower levels of contaminants would be found in sandy coastlines relative to muddy ones. The second hypothesis was that these differences would be observed when the clusters were analyzed collectively for all chemicals and when analyzed for individual chemicals.

2. Methods

The methods can be separated into two primary components: 1) method for computing before and after chemical concentrations (BACC), and 2) application of the BACC method for the analysis of the shoreline data collected during the DWH oil spill. The BACC method was based upon estimating the date of oil impact on the shoreline. The method included collocating chemical measurement data from the study site onto a grid which was categorized based upon coastal type. In this research, muddy coastlines were defined as dark brown to black sediments most likely consisting of high organic carbon. Sandy coastlines were defined as white to light tan sediments most likely consisting of low organic carbon. Since there was not enough sampling data in one small grid, grids were aggregated into clusters. Daily averages of chemical concentrations were computed and used to estimate oil exposure dates for each cluster based upon the timing of sudden increases in chemical concentrations. Once the exposure date was determined, the chemical concentrations for each cluster were computed for before and after exposure using two approaches. The optimum results from each approach were then used to develop a final pre-processed dataset (Figure S-1). This pre-processed dataset was then used to analyze before and after concentrations for each cluster (114 total) and for each chemical (252 chemicals total) (Figure S-2). The performance of the top ten chemicals in terms of before and after concentrations are illustrated for four clusters as examples. Analyses were then conducted for clusters as a whole and for clusters identified as muddy versus sandy (Figure S-3).

2.1. The dataset

The original data used in the current study consisted of DWH sediment sampling measurements from the U.S. Environmental Protection Agency (EPA) and from British Petroleum (BP). The focus was on sediments because of their potential to sorb OSCs and relevance to human exposure. These datasets have internal quality control specifications including validation qualifiers and metadata that describes sample preparation and analytical methods. Coastal regions, especially beaches, attract recreational activities which can expose humans to OSCs in the sediments (Ferguson et al. 2020a,

2020b, 2020c). Additionally, oil impacts are observed in sediments over longer time horizons, as opposed to air and water which are diluted more quickly. The longer duration of the chemical signal was necessary for the proposed method to work given the time scales of shoreline exposures which occurred during DWH over a time period of several months. Among the EPA and BP datasets, there were 175 sampling days in 2010, 48 sampling days in 2011 and 4 sampling days in 2012. All the samples were collected after April 20, 2020. Oil impacted the coastline on different dates, and so the background levels correspond to times after April 20 but before the oil made landfall.

Criteria for inclusion of a chemical measure from the EPA and BP datasets was that they represent specific chemicals (as opposed to bulk measures). A total of 252 chemicals met these criteria and were thus used to calculate oil exposure dates for a given cluster and to evaluate the concentration change before and after the exposure date. These 252 chemicals corresponded to a total of 226,574 measurements in 2010 (April to December), 272,547 in 2011 (January, February, March, July, August, October, November) and 36,269 in 2012 (February, June). The total number of measurements for these three time periods was 535,390. At each sampling location, there could have been only one or multiple samples collected over the sampling period. For each sample, multiple chemicals could have been measured. The 535,390 chemical measures evaluated in this study from the EPA and BP datasets correspond to 4114 samples collected at 2251 sampling locations (Xia, 2020).

2.2. Grid setting and coastal type classification

Our research area, corresponding to the northern region of the GoM, extended from longitude −97 to 83 and latitude 28 to 31. We separated this area into a 60 by 280 grid, each grid cell corresponding to 0.05° * 0.05° (about 5.6 km × 4.8 km). This resolution was chosen because it allowed for delineation of the coastline. Major coastal features such as embayments and estuaries are typically at the kilometer scale (Banks et al., 2017), so the 0.05 ° × 0.05 ° resolution allowed for the capture of these major features. The NOAA Medium Resolution Shoreline (https://shoreline.noaa.gov/data/datasheets/medres.html) GoM coastline was used to delineate land from water. At the coast, grid cells were identified as either sandy, muddy, or manmade construction primarily using Google Earth remote sensing imagery. If the grid cell corresponding to the coast was dark green, grey or black on the image, it was defined as muddy. If the grid cell corresponding to the coast was white (including light tan colors) on the image and the width of the white area was larger than 20 m, it was defined as sandy. Human construction consisted of seawalls and rocky shorelines consisting of riprap (Fig. 1). Classification based upon Google Earth imagery was checked against the EPA National List of Beaches (Figure S-4) and SCAT reports, and the classification aligned against these other sources of information.

2.3. Clustering of grid cells

Although the 0.05° × * 0.05° grid was suitable for identifying the shape and features of the coastline, it was too refined in terms of collocating sampling data. To address the coarseness of the sampling data, the grids were clustered.

The first step of the clustering process was to assign the sampling points to the grid cells. Only grid cells containing sampling points were part of a cluster. The extent of the cluster was defined by two features: sediment type (sandy versus muddy versus manmade construction) and by size corresponding to a region consisting of a maximum of 5 × 5 grid cells. All clusters were composed of a homogeneous sediment type so if the sediment type changed within the 5 × 5 region, a new cluster was initiated. If the sediment type was homogeneous a new cluster number was assigned beyond the 5 × 5 regional size. Using this approach, sandy clusters from 1 to 60 and muddy clusters from 1 to 54 were identified in sequential order for a total of 114 clusters. No samples were collected from grids classified as manmade construction, so no clusters were defined for this coastline type. As a result, this study focused solely on coastlines that were muddy or sandy. See more details in the supplemental text.

2.4. Computation of exposure date

Once the clusters were defined, the chemical concentration measures in each cluster (all in units of μg/kg) were averaged on a

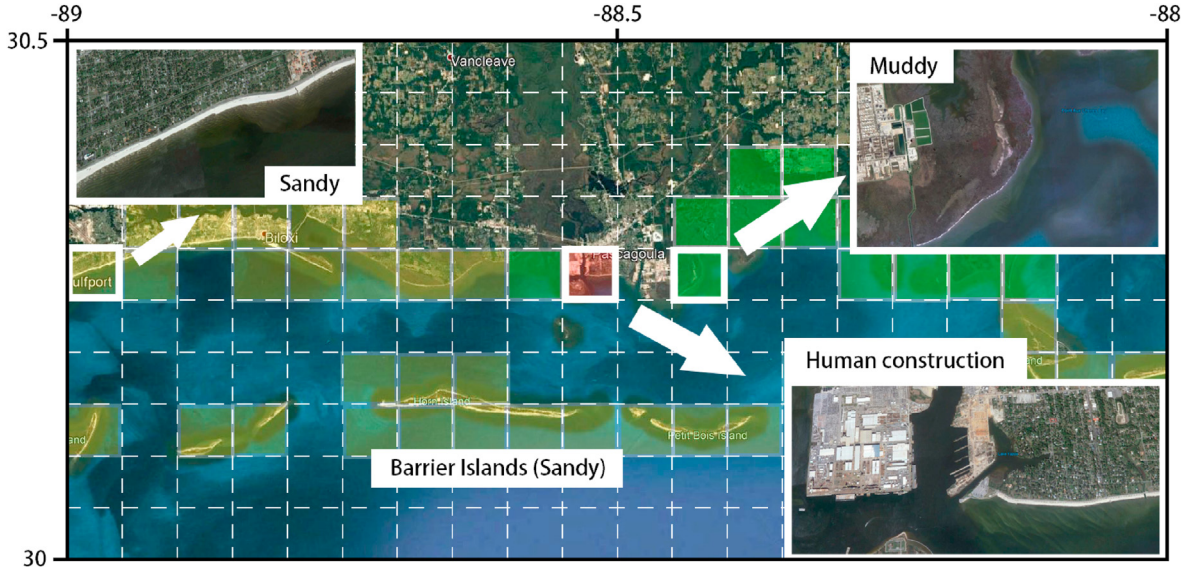

Fig. 1. Examples of grids and coastline feature classification into sandy, muddy, and manmade construction. Grids shaded in green correspond to muddy, in yellow correspond to sandy, and in red correspond to manmade construction. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

day-to-day basis for dates spanning from April 29, 2010 to June 7, 2012. One complicating factor associated with the chemical concentration measures was in terms of defining concentrations that were below detection limits. Detection limits for the chemical concentration measures varied significantly from sample to sample. One advantage of the BACC method is that it can be utilized to evaluate data with different detection limits. To address the inconsistencies with detection limits, the daily mean measure per cluster was pre-processed in two ways. First, for the "all" analysis method, the values below detection limits were set to zero. For the "detected" analysis method, the values below detection limits were not utilized. Daily mean measurements were then computed per cluster using both considerations for below detection limit values.

The computation of oil exposure date was conducted twice, once using the "all" measurement daily means and again using the "detected" measurement daily means. When calculating the exposure date, only the 2010 data was used to minimize the error because the active spill began on April 20, 2010 and the wellhead was capped on July 15, 2010, which means that the exposure from the actual oil spill (as opposed to redistribution of sunken oil) should be in 2010. The calculations for each set of daily means followed the previously described chemical concentration change (CCC) method (Xia et al., 2020). This method estimates the shoreline oil exposure date based upon the timing of rapid increases in OSCs. A large concentration increase between two sampling dates was considered indicative that the chemical impacted the cluster between the times the rapid increase was observed. In brief, the CCC method was based upon the following equation: $r = \frac{C_2 - C_1}{C_1} * 100\%$, where r was the increase threshold. C_1 was the concentration for the first date, $date_1$. C_2 was the concentration for the second date, $date_2$. According to the analysis of different r values by Xia et al. (2020) (See supplemental text for this reference), r was set to 100%. If the increase threshold (r) is larger than 100% (or doubling), the oil exposure date corresponding to the one chemical was set to occur between $date_1$ and $date_2$. The error length was defined as the difference in the dates corresponding to the 95% confidence limits (Xia et al., 2020).

In the new method (i.e., BACC), on a cluster-by-cluster basis, the optimum exposure date was then chosen from the "all" versus "detected" datasets based upon the set that provided the narrower error lengths. In general, the 'all' dataset provided narrower confidence limits if the detection limits were low. This was observed because: a) the zero values substituted for below detection limit values were closer to the low detected values and did not skew the results and, b) by including zeros for non-detects there was more data. The 'detected' dataset had the advantage (narrower confidence limits) when detection limits were high such that the replacement of non-detects with zero would skew the estimation of the average.

2.5. Chemical concentrations before/after oil exposure dates

Once the optimum exposure date was chosen, the chemical concentration measures were then separated based upon "before" and "after" oil exposure in different coastal types. This separation and computation of before and after concentrations is the primary contribution of the BACC method over the exposure date determination of the CCC method. For the BACC method, there were two ways to determine the "before" versus "after" concentrations for a given chemical within a cluster. The first one, called "average," calculated the mean chemical concentration in each cluster before and after the estimated exposure date. The second one called "direct" was based upon the closest measurement in time to the exposure date. The data point immediately before the exposure date was defined as the "before" concentration and the data point immediately after the exposure date was defined as the "after" concentration. The advantages and disadvantages of each method of determining the before/after depends upon the number of data points available within a given cluster for a given chemical. When the error length of the exposure dates was narrow, indicating a sudden increase of the chemical concentration in a short period of time, the "direct" chemical concentrations were considered as more representative. However, when the error length of exposure was wider than the average error length suggesting different chemicals doubling over a longer time, then the 'average' chemical concentrations would be more representative given that the exposure date likely spanned a wider range. (See supplemental text for more details about the error length). For each cluster, a merged dataset of the before and after concentrations were chosen among the "average" versus "direct" approach by conducting a paired *t*-test comparing all of the chemical concentrations in the "before" samples (log transformed and summed across all chemicals) to the concentrations of all of the chemicals in the "after" samples. The dataset (average or direct) that provided the lower p value was then chosen as the before/after concentration dataset for that cluster.

2.6. Analysis of before and after oil exposure shoreline data collected during DWH

The before and after datasets were evaluated to determine whether mean chemical concentrations were statistically different. The data were evaluated as a whole, by comparing before chemical concentrations for all chemicals and for all clusters to the corresponding after chemical concentrations. The data were also separated by muddy versus sandy coastlines to evaluate whether differences in before and after chemical concentrations were statistically different for muddy coastlines and for sandy coastlines. Chemical concentrations in muddy coastlines were also compared to chemical concentrations in sandy coastlines for both before and after oil exposures. Since the concentration data were log normally distributed in both paired and unpaired student t tests, all comparisons were conducted on log transformed chemical concentration data using paired t-tests (unknown variances) with the exception of comparisons between concentrations for muddy versus sandy coastlines where unpaired t-tests (unknown variances) were used.

Additionally, before and after chemical concentrations were evaluated for 10 individual chemicals within a subset of clusters to determine whether differences could be observed on a chemical-by-chemical basis. There were three categories evaluated: muddy, sandy, and all (muddy and sandy together) (Tables S−1). The p values were determined for each chemical within each category. Since muddy had higher p values (less statistical differences) the top 20 chemicals were sorted from low p value to high p value for the muddy clusters. The chemicals chosen within this list were required to have statistically significant p values in all and sandy categories ($p < 0.05$). Only 10 chemicals among the top 20 of the muddy categories met these criteria (See Table 1).

Although the entire northern region of the GoM (longitude −97 to 83 and latitude 28 to 31) was evaluated, three smaller representative regions were chosen to graphically illustrate the performance of the method (Fig. 2). These regions included: west of the Mississippi Delta (Region I), the Mississippi Delta (Region II), and the Mobile Bay (Region III).

Table 1
Top 20 indicator chemicals in muddy coastlines ranked by p value (low to high) for muddy. Top 10 chemicals meeting the p value criteria for sandy and all coastlines are bolded. ND means for the chemical, there was not enough data to calculate the p value for the category.

Chemical Name	Chemical Code	*Muddy p value*	*Sandy p value*	*All p value*
Diploptene	1615-91-4	0.01	ND	0.14
Dibenzofuran	132-64-9	0.02	0.09	0.42
C3-Phenanthrenes/Anthracenes	**85–01-8-C3**	**0.02**	**0.01**	**<0.01**
Chrysene/Triphenylene	**CHR_tPHE**	**0.02**	**<0.01**	**<0.01**
C2-Phenanthrenes/Anthracenes	85-01-8-C2	0.03	0.07	0.01
Pristane	**1921–70–6**	**0.03**	**0.05**	**<0.01**
cis/trans-Decalins	91-17-8-CT	0.04	0.35	0.93
C2-Dibenzothiophenes	**132–65-0-C2**	**0.04**	**0.03**	**<0.01**
n-Tetratriacontane (C34)	**14,167–59–0**	**0.04**	**0.02**	**<0.01**
Retene	**483–65–8**	**0.04**	**<0.01**	**<0.01**
C4-Fluoranthenes/Pyrenes	206-44-0-C4	0.05	0.17	0.02
Normoretane	3258-87-5	0.07	0.87	0.17
C3-Naphthobenzo-thiophenes	224-10-2-C3	0.07	0.17	0.02
C3-Fluorenes	86-73-7-C3	0.08	0.34	0.05
C1-Fluoranthenes/Pyrenes	**206–44-0-C1**	**0.09**	**0.03**	**0.02**
C1-Phenanthrenes/Anthracenes	**85–01-8-C1**	**0.09**	**0.03**	**0.02**
Benzo(e)pyrene	**192–97–2**	**0.09**	**<0.01**	**<0.01**
C3-Dibenzothiophenes	132-65-0-C3	0.10	0.08	0.02
C4-Naphthalenes	91-20-3-C4	0.10	0.05	0.03
n-Nonane (C9)	**111–84–2**	**0.10**	**0.01**	**0.04**

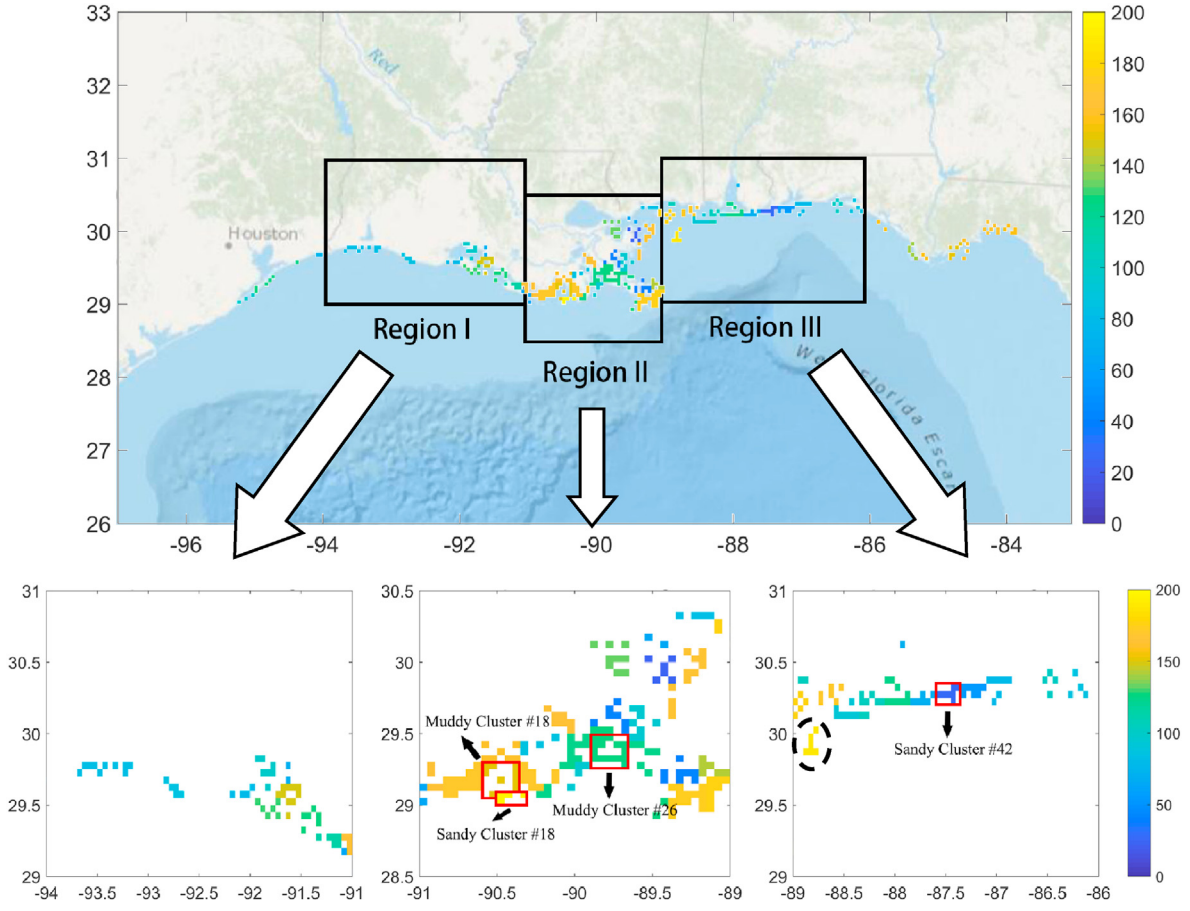

Fig. 2. First exposure dates along the Gulf of Mexico as shown by the color of the grid cells. The values on the color bar correspond to the number of days after April 20, 2010. Three specific regions are highlighted in black squares. Region I corresponds to the west of Mississippi Delta; Region II corresponds to the Mississippi Delta and Region III corresponds to Mobile Bay. Clusters highlighted by red boxes were chosen to illustrate the results for 10 specific chemicals listed in Table 2. Cluster inside a dash circle was an example of late sampled locations. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

3. Results and discussion

3.1. Oil exposure date

Among the 54 muddy clusters, results indicate that 31 were exposed to oil. Among the 60 sandy clusters, 32 were exposed. The exposure dates are shown in Fig. 2. All three regions show oil exposure dates ranging from 20 to 180 days. Most of the late exposure dates were observed in Region II. Within this region, although most of the coastline was exposed to oil, the distribution of exposure dates was patchy with no distinct spatial distribution of areas impacted earlier versus later. This may be due to the spatial heterogeneity of the oil near the shore (Rouhani et al., 2017), variations of nearshore currents and eddies that carried the oil to the shore (Özgökmen et al., 2014), and freshwater inputs from the Mississippi River (Androulidakis et al., 2018) which may have influenced oil beaching. The patchiness may also be influenced by the timing of sample collection. Some areas were not sampled until much later. For example, there were several barrier islands near 30°N 89°W in Region III (dashed oval), which were first sampled on November 13, 2010, 207 days after the oil spill happened.

3.2. Analysis of before and after chemical concentrations for data collected during DWH

When assessing individual measurements, overall, 51,513 measurements corresponded to before oil exposure whereas 471,150 measurements corresponded to after oil exposure. The relative distribution of measurements before and after exposure differed based upon coastal type. More measurements were taken in muddy coastlines after exposure ($n = 442{,}550$) relative to before ($n = 28{,}769$). The number of measurements between before and after for sandy coastlines was more balanced ($n = 22{,}744$ before and $n = 28{,}600$ after). Many of the samples from the muddy coastlines were collected later during the 2011- and 2012-time frames presumably due to studies focused on evaluating accumulation and degradation of oil within this coastal type (Turner et al., 2014a,2014b, 2019). In muddy coastlines, there were 164,061 measurements in 2010, 270,989 measurements in 2011 and 36,269 measurements in 2012. In sandy coastlines, there were 50,014 measurements in 2010, 1330 measurements in 2011, and no measurements in 2012.

When aggregating the chemical measurements by clusters (i.e., location), statistically different concentrations were also observed between before and after oil exposure (Fig. 3). Results show (Fig. 3, panel a) that fewer measurements on a cluster-by-cluster basis were available for the before data ($n = 3293$) compared to the after data ($n = 5304$). The number of chemical and cluster combinations for muddy coastlines was less for the "before" condition ($n = 1826$) relative to the "after" condition ($n = 3560$). For sandy regions the number chemical and cluster combinations between "before" ($n = 1467$) and "after" ($n = 1744$) conditions was again more well balanced.

The difference in the number of chemical and cluster combinations between muddy and sandy regions may have been due to quicker and prioritized coastline clean-up activities in sandy areas (Zukunft, 2010). Of the 900 km of oiled beaches, shoreline treatment was conducted on 660 km (73.3%) of oiled beaches. In contrast, of the 796 km of oiled marshes, shoreline treatment was completed for 71 km (8.9%) of the oiled marshes and associated habitats (Michel et al., 2013). At muddy coastlines, which would include areas dominated by coastal marshes and wetlands, clean up was more difficult. Several studies were conducted to evaluate the degradation of oil within coastal marshes that were not amenable to traditional clean up processes (Turner et al., 2014a, 2014b; Rodrigue et al., 2020), whereas sandy beaches were preferentially cleaned.

Of particular interest is that the distribution of the chemical concentrations before oil exposure (for all chemicals and for all clusters collectively) was statistically less than the chemical concentrations after ($p < 0.0001$) with differences of about 0.567 log units on average. When splitting the data by muddy (panel b) and sandy (panel c) coastlines, again the before chemical concentrations were statistically lower by about 0.326 log units for muddy coastlines and 0.793 log units for sandy coastlines ($p < 0.0001$ for all chemicals for all muddy clusters and for all chemicals for all sandy clusters). When comparing the chemical concentrations between muddy versus sandy coastlines, sandy coastlines consistently had lower chemical concentrations by about 0.563 log units in comparison with muddy coastlines for the before (panel d) condition and about 0.096 log units for the after (panel e) condition. This observation is consistent with the well-known relationships between organic carbon in sediments and retention of organic chemicals (Schwarzenbach et al., 2017; Schneckenburger and Thiele-Bruhn, 2020). This sorption property of organic rich soils allows for the retention of petroleum derived chemicals within muddy coastlines. For example, Garcia et al. (2019) has shown that hydrocarbon concentrations from petroleum contamination correlates with levels of organic carbon within mudflats and mangrove sediments. Similarly, polyaromatic hydrocarbon contamination from oil spills were found to correlate with organic humic acid concentrations within an estuarine river (Andrade et al., 2019). Thus, the higher levels of OSCs in observed organic rich muddy coastlines is consistent with field studies that have found that the organics found in sediments retain petroleum compounds.

Results also showed that muddy coastlines had a higher chemical concentration level both before and after oil exposure than sandy coastlines. There were several possible reasons. One possible reason was that the before baseline for muddy coastlines was at a higher level than sandy coastlines requiring heavier impacts from oil in order to see increases above baseline. Additionally, sandy coastlines were better monitored and sampled before the oil exposure date. Many of the muddy coastlines were sampled late, which lead to the lower chemical concentration levels after the oil exposure date (due to weathering) and thus the smaller concentration change.

When calculating the oil exposure date, the chemical concentrations were observed to double more than once in some regions, suggesting multiple exposure dates for these regions (Beyer et al., 2016). Although the methods presented herein could be used to calculate multiple exposure dates, the limited number of measurements given the geographical expanse was not sufficient to evaluate multiple dates of exposure. Also after initial landfall oil degradation patterns may change due to contact with the sediments further complicating the ability to observe doubling of chemical concentrations from onshore oil as opposed to the impacts of degradation in sediments. With additional planning and measurements, a similar approach as presented herein could possibly be used to evaluate multiple dates of oil exposure to shoreline sediments. The method can also be potentially expanded to evaluate the degradation of pollutants after exposure.

3.3. Illustrations of concentration changes by individual chemical

Among the 252 chemicals used in this research, 10 chemicals were found to meet the strongest statistical criteria emphasizing differences between before and after concentrations. These 10 chemicals (Table 2) included 2 straight chain hydrocarbons (n-nonane (C9), n-tetratriacontane (C34)) and 1 branched

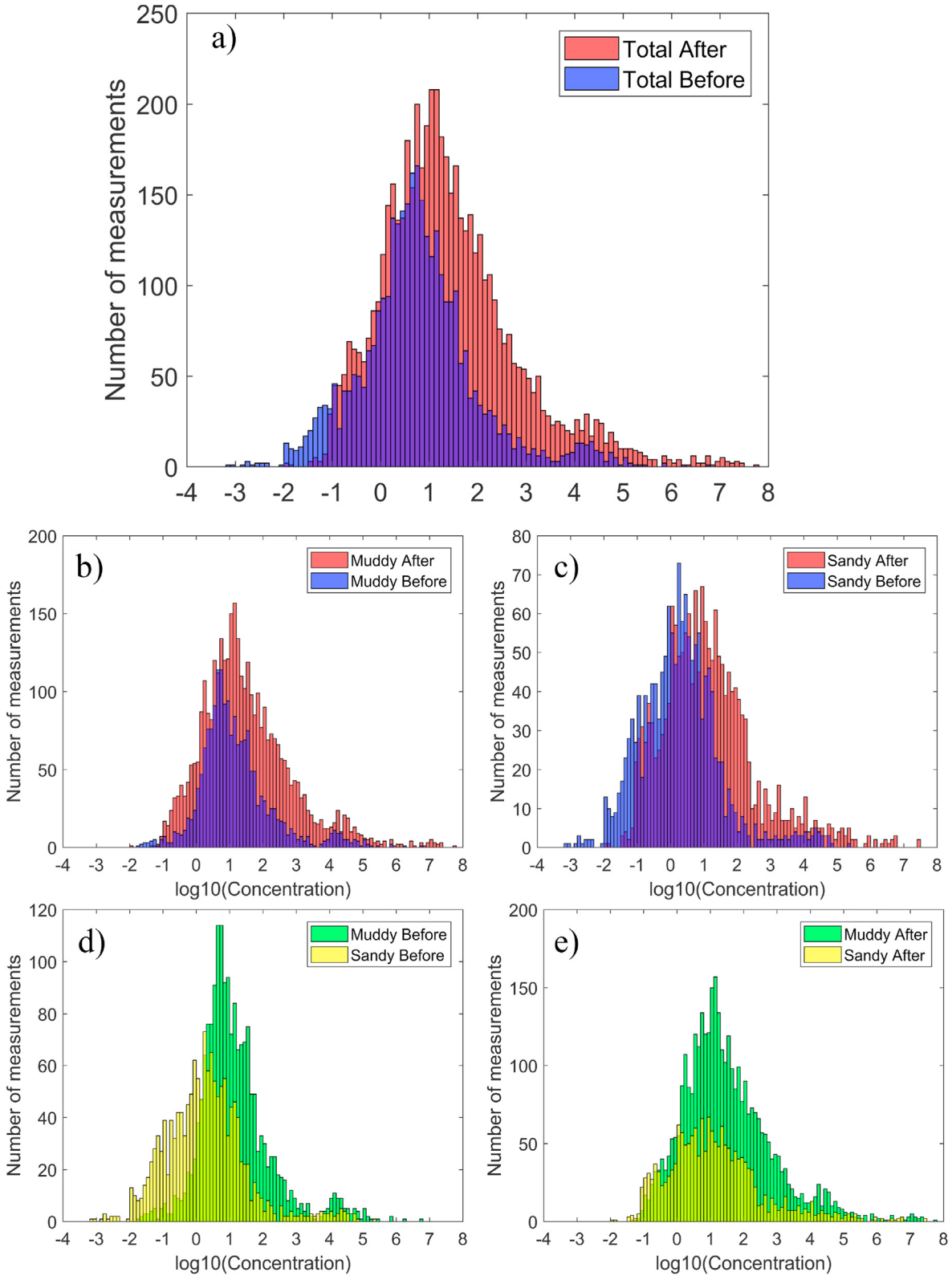

Fig. 3. Chemical concentration distributions of shoreline sediment before versus after oil exposure (for all clusters and for all chemicals). Panel a, b, and c compare before to after chemical concentrations measures for the entire dataset, for muddy coastlines, and for sandy coastlines together. Panels d and e compare the chemical measures of muddy versus sandy for before (panel d) and after (panel e) oil exposure. The width of each bar (or bin width) was 0.1 $\log_{10}$ (μg/kg). The p value for comparison of the collective datasets shown in panels a through e were all <0.0001.

Table 2
Chemical concentration changes and significance of the differences between before and after concentrations for sandy and muddy regions. The units of the chemical concentrations were μg/kg. "ED" below the cluster number means the exposure date for that cluster where the number represents the number of days after April 20, 2010. The p values were computed using unpaired t-tests. Muddy p values and sandy p values indicated the p value for concentration change of each chemical in all muddy and sandy clusters collectively. The "all clusters" p value corresponds to the concentration change for all clusters regardless of muddy or sandy designation. ND in the table means there were no detected measurements. Of note, since the exposure date was calculated based upon 252 chemicals, some chemicals were missing before concentration averages (For example no measurements were available for the three non-aromatic hydrocarbons for muddy cluster #18).

Chemical Name	Chemical Code	Muddy					Sandy					All Clusters
		Cluster # 18 ED: 153		Cluster # 26 ED: 126		All Muddy Clusters	Cluster # 18 ED: 200		Cluster # 42 ED: 104Para Run-on–>		All Sandy Clusters	
		Before	After	Before	After	p value	Before	After	Before	After	p value	p value
n-Nonane (C9)	111-84-2	ND	3.15	37.07	68.74	0.10	0.90	31.82	0.70	2.78	<0.01	0.04
n-Tetratriacontane (C34)	14,167-59-0	ND	857.4	85.77	45,369	0.04	21.94	471.1	6.11	86.90	0.02	<0.01
Pristane	1921-70-6	ND	30.68	26.17	52,102	0.03	22.18	84.22	53.00	13.98	0.05	<0.01
C2-Dibenzothiophenes	132-65-0-C2	0.60	5.37	3.31	3015	0.04	6.53	40.99	0.92	4.35	0.03	<0.01
C1-Phenanthrenes/Anthracenes	85-01-8-C1	1.33	7.00	10.14	4710	0.09	6.89	42.65	1.25	5.00	0.03	0.02
C3-Phenanthrenes/Anthracenes	85-01-8-C3	4.53	10.59	4.98	4796	0.02	16.08	112.8	3.92	6.40	0.01	<0.01
Retene	483-65-8	2.17	8.94	2.28	90.45	0.04	1.46	31.28	1.18	9.19	<0.01	<0.01
C1-Fluoranthenes/Pyrenes	206-44-0-C1	3.47	8.74	6.86	1039	0.09	5.41	58.84	0.54	17.95	0.03	0.02
Chrysene/Triphenylene	CHR_tPHE	2.32	13.86	7.89	980.1	0.02	5.64	61.08	0.08	11.17	<0.01	<0.01
Benzo(e)pyrene	192-97-2	0.83	4.79	4.74	462.4	0.09	2.69	41.27	0.07	9.85	<0.01	<0.01

hydrocarbon (pristane). The remaining 7 were polyaromatic hydrocarbons (PAH) or their alkylated forms. These included a sulfur containing PAH with two aromatic rings (C2-dibenzothiophenes), three phenanthrene homologues each with three aromatic rings (C1-phenanthrene, C3-phenanthrene, retene), one with four rings - three of which are aromatic (C1-fluoranthene), one with four aromatic rings (chrysene), and one with five aromatic rings (benzo(e) pyrene). Of note is that most of the chemicals that showed statistical differences before and after oil exposure were PAHs which tend to be the more persistent components of OSCs (Shinde et al., 2020). Alkanes, three of which were listed in the top 10, are more readily degradable (Bociu et al., 2019) and would likely be an indicator of less weathered oil impacts.

Although statistics were computed for 114 clusters (54 muddy and 60 sandy), 4 clusters were chosen for illustrative purposes. Two muddy clusters in Region II with different locations were chosen (muddy cluster #18 and #26), given that this region had sufficient measurements. For sandy clusters, one was chosen in Region II (sandy cluster #18) and another in Region III (sandy cluster #42).

Results for individual chemicals (Table 2) show that for all clusters (no separation between muddy and sandy), concentrations were statistically different between before and after oil exposure. The p values in sandy regions were lower than in muddy regions, which indicates that the means of the before and after were different at greater level of confidence and thus there was greater certainty in the estimation of the oil exposure date in sandy regions. However, when evaluating the results on a cluster-by-cluster basis some reversals were observed. One reversal can be explained by the proximity of the muddy versus sandy coastline to the wellhead. For example, muddy cluster #18 was close to sandy cluster #18. Sandy cluster #18 was slightly to the southeast of muddy cluster #18, closer to the wellhead. The geography implies that oil reached the sandy cluster first and more heavily than the corresponding muddy cluster. This influence of geography was reflected in the chemical concentration levels. For this specific case the sandy cluster, which was closer to the wellhead, was characterized by higher chemical concentrations relative to the muddy cluster. As an exception to the general observation of muddy clusters with higher chemical concentrations, almost all the chemical concentrations after the oil exposure date in sandy cluster #18 were higher than those in muddy cluster #18.

Because of the reversal in chemical concentration trends, the baseline calculated with our method might not be a 'true baseline'. Specifically, the before concentration for sandy cluster #18 may not represent the true baseline. It deviates from the sandy versus muddy trend and is characterized by elevated OSC levels suggesting that the before levels may include impacts from oil. Similarly, muddy cluster #26 may also reflect prior impacts from oil given the higher before levels compared to muddy cluster #18 which is in the near vicinity.

3.4. Potential standardization of indicator chemicals

One approach to standardizing oil spill monitoring would be to narrow down the list of chemicals to be measured in an oil spill. Two hundred and fifty-two different chemicals were measured during and immediately after the DWH explosion. The set of 10 chemicals evaluated in greater detail in the current study could represent a potential subset of indicator chemicals to document when shoreline oil exposures occurred and the concentration changes before and after oiling. The concept of an indicator chemical is drawn from the microbiology literature where fecal indicator bacteria are used to trace the presence of sewage impacts to beaches (Barreras et al., 2019; Kelly et al., 2018; Donahue et al., 2017). More recently chemical tracers (chemicals that are found in sewage such as pharmaceuticals, caffeine, and sucralose) have also been used to document potential sewage impacts to beach environments (Biel-Maeso et al., 2018; Buerge et al., 2003). In a similar fashion, indicator chemicals can serve to trace the presence of oil impacts to beaches. Although the oil spill community has extensive fingerprinting capabilities to identify the source of oil by evaluating ratios of chemicals found in the field (Lourenço et al., 2020; Passow and Stout 2020; Han and Clement, 2018; Baszanowska and Otremba, 2014), chemical tracers of oil exposure dates would serve a different purpose, to determine when the coastline was impacted through measurements during the spill. Chemical tracers could also help to evaluate the degree to which chemicals changed before and after an oiling event. One topic of future work is to evaluate whether the top 10 chemicals identified in the current study would be useful for estimating shoreline exposure dates for other spills.

Differences in individual chemical performance were observed in terms of muddy versus sandy coastlines. Some of the identified individual chemicals have relatively low p values in muddy regions but relatively high values in sandy regions, like pristane. Other chemicals have a relatively high p value in muddy regions but

relatively low value in sandy regions, like n-nonane. Some chemicals have low p values in both muddy and sandy regions, like C3-phenanthrenes/anthracenes and chrysene/triphenylene. This information could be potentially used to help decide which chemicals could be used to indicate the oil exposure level in different regions (Tables S−1).

When deciding which chemicals to use, oil chemistry and local background levels appear to play a role in why some chemicals have clearer demarcations between before and after oil impacts. For example MC-252 oil associated with DWH is rich in C3-phenanthrenes/anthracenes, chrysene and benzo[e]pyrene (Montas et al., 2020) and this may be the reason why these chemicals were in the top 10 for providing a better statistical difference (lower p values) between before and after oil exposure.

3.5. Consistency of sample collection program

When reviewing the available datasets for chemical measures (e.g., the BP and EPA datasets), few measurements occurred at fixed locations. Most measurements were opportunistic, or convenience measurements given access to the site. The dataset was augmented with carefully designed field studies which were typically geographically limited and limited in duration. Taken as a whole, the data lacked consistency. No sample was measured for all of the chemicals, and sampling locations varied. The lack of uniformity made the interpretation of the data more difficult requiring aggregation of the available information in time and space as presented in the current study. Such datasets are not uncommon especially in response to disaster events (e.g. Hurricane Katrina response (Anonymous 2005)). During such conditions, coordination is difficult among agencies and so data is gathered on an as available basis to assess potential impacts of disasters.

Given the large range of sampling locations and frequencies, the procedure developed through this study required analyses of the chemical data using different averaging methods. Many of the measures were below detection limits (>63.2%) requiring decisions about how to handle concentration measures below the sensitivity of laboratory equipment. The decision to omit or substitute values below detection limits relied on the number and distribution of chemical measures within a cluster. The method developed through this study allowed for choosing an averaging method that optimized the identification of the exposure date, the critical first step of separating measurements into before versus after oil exposure. Such an approach would be useful for evaluating surges of contaminants for which impact dates are not well known. Examples of applications of the method beyond oil spills can include, for example, the impacts from large scale regional contamination events such as harmful algal blooms, and even Saharan dust on water quality. Similarly the procedure developed through this study also required decisions concerning how to set the concentration before versus after (using either the average or direct method). Again, here the optimum decision was based upon the number and distribution of chemical measurements within a cluster. The merged dataset was based upon identifying the method that provided the strongest p value within a given cluster in terms of detecting differences in chemical concentrations over time. Such an approach is well suited for evaluating large regional datasets where samples were collected in a nonuniform fashion.

4. Conclusions

The objective of this study was to develop a method to estimate chemical concentrations in shoreline sediments before and after oil exposure. We have developed the BACC method to compute chemical concentrations before and after oil exposure. The BACC method builds upon the earlier Chemical Concentration Change (CCC) method which was used to estimate shoreline oil exposure date. One advantage of the BACC method is its ability to preliminarily assess baseline chemical concentrations prior to oil spills. Such results would be useful for risk assessments that evaluate oil spill impacts to ecosystem and human health. Future work should integrate additional statistical approaches, such as Monte Carlo methods. The current approach did provide a range of exposure dates which is an important first step to assess this large data set. A Monte Carlo approach would be useful in providing a second layer of uncertainty bounds by providing confidence limits for the chemical concentrations before and after oil impact.

Our first hypothesis of lower levels of contaminants in sandy coastlines relative to muddy ones was supported by the results of this study. When comparing chemical concentrations between muddy versus sandy regions, sandy coastlines consistently had lower chemical concentrations ($p < 0.0001$) by about 0.563 log units in comparison with muddy coastlines for the before condition and about 0.096 log units for the after condition. Overall, sandy regions had lower average chemical concentration levels both before and after oil exposure and a smaller change in chemical concentration level, compared with the muddy regions. Results suggest that muddy regions may be more susceptible to ecosystem impacts due to higher chemical concentrations, which is likely due to stronger sorption of chemicals.

Our second hypothesis (significant differences in before versus after and muddy versus sandy when the clusters are analyzed collectively for all chemicals and when analyzed for individual chemicals) was also supported by the results of this study. Using the BACC method, a new processed dataset was created that separated chemical concentrations by location (clusters), coastal types, and before and after oil exposure dates during the DWH oil spill (See supplement file). Taken together the differences in chemical concentrations between before and after exposure were statistically significantly different ($p < 0.001$). The differences were 0.567 log units of concentration, with a difference of 0.326 log units for muddy coastlines and 0.793 log units in sandy coastlines. This analysis thus provided a quantified level of oil impact.

When clusters were evaluated on a chemical-by-chemical basis the more measurements the more statistically significant the differences were between the before and after measurements. The chemicals shown in Tables 1 and 2 were considered optimum for distinguishing concentrations between before and after impacts. These chemicals included predominantly PAHs which is the component of oil that is most toxic and the most difficult to degrade. Future studies should focus on evaluating the concept of listed indicator chemicals to standardize oil spill measurements. Additionally, spatially-representative, stationary, and regularly-monitored stations would be useful for assessing the impacts of future spills and other disasters. With increased coordination in sample collection and analysis, less data would be potentially needed and the BACC method could be used to initially assess concentration baselines, estimate exposure dates, and quantify impact levels in different regions.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplement Data

All the results have been placed in the supplement, including the estimated exposure dates using the "all" dataset and the "detected" dataset. The before and after concentrations were determined using the "average" and "direct" methods. The supplement contains results on a per chemical and per cluster basis for both of these approaches using the "all" and "detected" methods for estimating exposure date.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.envpol.2020.116102.

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