



## Application of PIONA GC-FID in Oil Spill Studies: Detail Hydrocarbon Profiling

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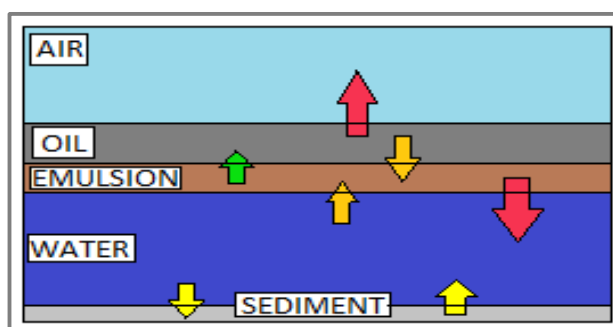
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### ABSTRACT

The fate of oil in the marine environment after an oil spill depends on the physical and chemical characteristics of the spilled oil, the marine environment itself and weather conditions. In this lab based experiment, sea water was generated and exposed to two types of light and heavy Alberta based oil types then were left in the lab for time intervals of 6 h, 30 h and 1 week. The water column beneath the spilled oil was collected and extracted for the presence of BTEX using Hexane: Dichloromethane mixture (3:1). The floating oil was collected and treated with isooctane. Samples were analyzed using a PIONA GC-FID for the presence of BTEX. The results showed that floating oil demonstrates significant loss of BTEX, which was being released to the water column and evaporated to the air. The release of BTEX from the lighter oil to the air and the water column showed a linear decreasing trend while the heavier oil showed an irregularity. BTEX concentrations in the water column increased 2-3 times in the first 24 h in the lighter oil while the heavier oil did not show a meaningful change. This study revealed that 80-97 % of benzene species that existed in the crude oil reached the water column during the experiment in various concentrations. The results of the work in this study demonstrate that BTEX compounds were predominantly released to the air or remained in the emulsion layer. This study verifies the principle that a quick response to clean the contaminated site reduces BTEX release to the atmospheric environment.

### Graphical Abstract



Formation of various layers in a spill condition. Red arrows are considered harmful to the human health and the environment, green shows a positive re-association of emulsified compounds to the oil and yellow show potential of staying in the target layers

**Keywords:** PIONA, Oil Spill, BTEX, Benzene Species, Emulsion Layer.

## INTRODUCTION

Rapid economic development in more than half a century can be mainly justified with the presence of marine transport and the availability of fossil fuels [1]. Crude oil became the main source of development and its transportation is one of the most essential types of marine export/import. Marine transport is associated with incidents and oil spill that cause major environmental problems worldwide however in the past 5 years the number of major spills remained on an average of 6.6 incidents that shows a continuous improvement trend since 1974 [2]. Following an oil spill in a marine environment, it is vital to determine the status of the spill in order to create an adequate response plan. Behavior and fate of oil in the marine environment after a spill are dependent on the physical and chemical characteristics of the spilled oil, the marine environment itself and weather conditions. These factors will impact dispersion, a process by which oil moves horizontally and vertically in water. The spilled oil, through the dispersion process, releases its components, known to be typically over several thousand individual compounds, to the receiving environment. These chemicals, including mono and polycyclic aromatic hydrocarbons and their alkylated analogues are of major health and environmental concerns due to their toxicity as well as increased bioavailability of the alkylated analogues. Once released, these chemicals may end up in the water column, enter the atmosphere through evaporation or sink to the sedimentary environment [3]. The environmental impacts from a spill incident can be both physical and chemical; where beside the release of chemicals, the habitat can be damaged by the presence of heavy oil and emulsion. The rate of emulsion formation is highly dependent on the physico-chemical properties of the oil and the release conditions [4]. While the heavy oil directly destroys the habitat in a physical form, the oil/water emulsion can carry chemicals with tendency of being released to water or remain in the oil phase. This tendency will follow mostly the facts of the log of  $K_{ow}$  (octanol/water coefficient). The environmental concerns of a spilled incident may not be only limited to the completion of spill cleaning activity and may require longer term ecosystem based assessment to ensure the healthiness of the environment [5].

Benzene and its derivatives are the major causes of health and environmental concerns [6]. This group of chemicals may contain single benzene compounds or fused benzene rings of 2 or more. Single benzene ring compounds and their alkylated derivatives are lighter in molecular weight thus are more toxic in the environment. The number of compounds in this group may exceed 50 individual compounds with four of them are categorized as major concerns to human health and the environment. Benzene, toluene, ethylbenzene and xylenes (comprised of ortho-, meta- and para- isomers) are the group of mono aromatic compounds that are known as BTEX in literature (Figure 1). The solubility of BTEX in water follows B>T>E>T order [7]. The physical properties of BTEX are presented in table 1.

**Table 1.** Physical Properties of BTEX (US-EPA, 2019)

BTEX	Formula	Molecular Weight (g mol <sup>-1</sup> )	Vapor Pressure mm Hg at 25°C	Odor Threshold	Log $K_{ow}$ (Octanol/Water)
Benzene	C <sub>6</sub> H <sub>6</sub>	78.11	95.1	1.5 ppm (5 mg m <sup>-3</sup> )	2.13
Toluene	C <sub>7</sub> H <sub>8</sub>	92.15	28.4	2.9	2.69
Ethylbenzene	C <sub>8</sub> H <sub>10</sub>	106.16	9.53	2.3	3.13
Xylene	C <sub>8</sub> H <sub>10</sub>	106.16	6.728	1.1 (as per m-xylene)	3.12

BTEX are naturally occurring in crude oils and consequently in petroleum products from the processes in refineries. Refineries routinely adjust the level of BTEX in order to control the octane number in the final product as appropriate for the market. Moreover, BTEX compounds are used in various industries as part of products or process from nylons to resins and insecticides to printings. As

per the partition constant of octanol to water ( $K_{ow}$ ), BTEX components may dissolve in water or stick to particles and disperse in the environment due to physical forces such as gravity and/or wave [8].

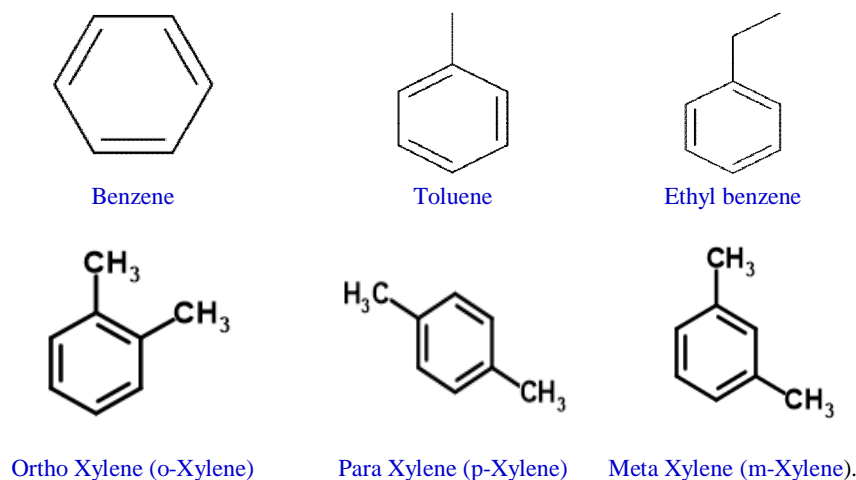


Figure 1. BTEX Molecular Structures.

Human intake of BTEX is mostly from drinking of water and inhalation. Beside long term effects of benzene that may cause cancer to human, the short term and acute toxicity occurs as skin and sensory irritation, central nervous system depression, and effects on the respiratory system. Prolonged exposure may affect the liver, blood and kidneys in humans. Due to this, a rapid removal of BTEX from the environment seems necessary [9]. According to Oregon Department of Human Services [10], the Maximum Contaminant Levels (MCLs) for BTEX compounds in water for an average individual that drinks 2 L of water per day over a lifetime (of 70 years) are 0.005, 1, 0.7 and 10 mg L<sup>-1</sup> (ppm) for benzene, toluene, ethylbenzene and xylenes. The damaging factor of chemicals released from a spill may require an innovative way of assessment as Peterson et al. indicated in his research [5]. The ecosystem based assessment seems to be necessary even for individual compounds due to bioaccumulation fact of chemical compounds in marine flora and fauna [11].

## MATERIALS AND METHODS

**Chemicals:** Isooctane, hexane and dichloromethane were purchased from Fisher Canada all with purity of 99.9% or higher. The commercial sea salt (Instant Ocean) was supplied from an authentic supplier.

**Glassware:** Glassware were cleaned with water and soap and dried then rinsed with methanol, dichloromethane and hexane, 3 times each then dried in the oven at 65°C for overnight. The glassware then was stored in a dry cabinet shelf prior to use.

**Preparation of Sea Water:** A total mass of 35 g of commercially available sea salt (Instant Ocean) was added to a pre tared 2L beaker on an analytical balance then distilled water was added to reach to a total mass of 1000 g. A glass stirrer was used to completely dissolve the salt. Several similar solutions were prepared and stored for less than 12 h of holding time prior to experiments.

**Oil Spike:** Four glass jars were used for the experiment on each type of oil. Hundred and fifty grams of artificially prepared sea water were added to each glass jar. Three glass jars were spiked with 10 g of oil each and labelled as 6h, 30h and 1W are representing 6 h, 30 h and a week of experimental holding time, respectively. The 4<sup>th</sup> glass jar was remained with no spiked oil and kept as blank. All jars were kept undisturbed on bench top for the designated time intervals of the experiments.

**Extractions:** The experimental jars were taken for extraction. A disposable pipette was used to collect the top floating spiked oil. Another disposable pipette was used to collect the entire seawater beneath the floating oil. A tiny layer between the seawater and the floating oil was eliminated from getting into any of these collected portions. All collected portions were labelled as floating oil or water accordingly with their corresponding interval times. The floating oil was treated with isooctane (1:1; v:v) to eliminate any water interferences in GC analysis. The oil-isooctane fraction was placed into a 2 mL GC vial with no headspace for PIONA analysis by the GC-FID. The collected experimental water was liquid-liquid extracted using hexane:dichloromethane (3:1; v:v) solution with an equal ratio (1:1; v:v) of solvent to water for 30 min. After extraction, a separatory funnel was used to allow the solvent layer to separate. The Hex:DCM solution was rotary evaporated at 40°C and 800 mbar vacuum for 30 min to decrease the solution volume. Then transferred to a 2 mL GC vial for GC analysis.

**GC Analysis:** An Agilent 7890B Gas Chromatograph coupled with Flame Ionization Detector-PIONA (paraffins, iso-paraffins, olefins, naphthenes and aromatics) library-was used for instrumentation and data interpretation. The sample tray was auto-chilled and an auto-sampler was used to provide an accurate sample transfer to the GC. A Restek capillary column was used (Rtx-DHA 100 m x 250  $\mu$ m x 0.5  $\mu$ m) in the GC. A total of 0.2  $\mu$ L of sample was transferred to the liner through injection process with 0.01 min pre-injection and 0.03 min post-injection dwell time. The equilibration time was kept at 0.5 min. A split ratio of 200:1 was employed. The front inlet was kept at 250°C with a pressure of 36.915 psi. The temperature was programmed at 40°C for 8.32 min then ramped at 22°C min<sup>-1</sup> to 48°C then hold for 26.32 min followed by the next ramp of 3.2°C min<sup>-1</sup> to 141°C with no holding time and finally 1°C min<sup>-1</sup> to 300°C with no holding time that configure a total run time of 223.07 min. The FID was set at 300°C with hydrogen flow of 35 mL min<sup>-1</sup>, air flow of 400 mL min<sup>-1</sup> and a combined makeup flow of 25 mL min<sup>-1</sup>. The integration events of individual peaks were manually re-visited for performance accuracy and the results were compared with Dragon software and library.

## RESULTS AND DISCUSSION

The results of this study were mainly focused on the presence and release of benzene, toluene, ethyl benzene and xylenes (BTEX) contents (xylene refers a total of ortho, meta and para xylene isomers) where remained and/or released into the water column during the experiments. The release of any of above contents into the air was not measured and consequently not reported.

**Density and Viscosity of Oil:** Digital density meter, Anton Paar (DMA, 4500) and Anton Paar Stabinger viscometer (SVM-3000/G2) were used to measure density and viscosity of two crude oils. The results of density and viscosity are presented in [table 2](#) below.

**Table 2.** Viscosity and Density of Original Crude Oil A and B at 15°C

Crude Oil	Viscosity Kinematic mm <sup>2</sup> s <sup>-1</sup>	Density g cm <sup>3</sup>
A	11.540	0.8556
B	991.84	0.95275

Type “A” oil is a lighter oil where the density and viscosity were lower comparing to type B oil. Type “A” oil had observed to have an easier flow in room temperature (23°C) while type “B” oil was resisting against gravity force in flowing in a similar condition. A droplet of each type of oil was dropped on a glass plate and left for a minute under the fume hood then attempted to remove using a laboratory paper towel. Type “B” oil was in need of an addition of a toluene (as removal solvent) for a final wipe from the surface indicating the presence of heavier end (bituminous contents) however type A oil was easy to be wiped clean using a laboratory towel.

**BTEX:** The results of BTEX contents in original crude oil, the experimental water and the floating oil are presented in tables 2 and 3 (below) with crude oils holding 100 percent of BTEX content and the percent of each contents release to water column or remained in the floating oil. Table 3 shows results of type A oil and table 4 demonstrates results of type B oil.

**Table 3.** BTEX Content Percent of Crude Oil A, in Water and in Floating Oil

BTEX	Crude A	Water			Floating Oil		
		6h	30h	1W	6h	30h	1W
Toluene	100	1.88	7.48	3.04	36.49	30.98	27.38
Ethylbenzene	100	5.21	10.70	5.05	40.76	41.25	36.13
Xylene	100	5.48	10.76	5.44	33.14	33.92	29.30
Benzene	100	nd*	nd*	nd*	21.06	9.66	8.76

\*nd; not detected

**Table 4.** BTEX Content Percent of Crude Oil B, in Water and in Floating Oil

BTEX	Crude B	Water			Floating Oil		
		6h	30h	1W	6h	30h	1W
Toluene	100	nd*	4.39	6.86	15.70	6.86	10.31
Ethylbenzene	100	19.54	12.50	17.50	9.61	5.61	20.22
Xylene	100	16.83	10.38	7.44	9.61	5.61	20.22
Benzene	100	nd*	nd*	nd*	9.85	1.94	3.58

\*nd; not detected

**BTEX in Water Column:** BTEX content in water column showed a different trend from type A to B oil. In type A oil, in the first 6 h of the experiment, 1.88% of toluene was released from the oil in the spill simulation to the water column. This number was 5.21 and 5.48 percent for ethyl benzene and xylene, respectively. Benzene was not detected in the water column after the first 6 h of experiments. Toluene and benzene in lighter crude oil (Crude Oil A) showed abundances of 10 fold compared to heavier crude oil (Crude Oil B) while ethyl benzene and xylene showed 14 and 9 times, respectively. The second jar was left for 30 h of retention time. The presence of benzene was not detected after this retention however the other 3 components showed an increasing trend. The release of toluene was measured 7.48% of the original toluene content of the oil followed by 10.70 and 10.67 percent for ethyl benzene and xylene. The last set of jars were left for one week. The results of BTEX content showed a general decrease with the absence of benzene. This can be an indicative of re-association of BTEX content to the floating oil.

In crude oil type B, after 6 h of retention, the study was unable to detect toluene and benzene however ethyl benzene and xylene showed 19.54 and 16.83 percent release of the original contents from the crude oil into the water column. This observation after 30 h of retention was 4.39, 12.50 and 10.38 percent release of respectively toluene, ethyl benzene and xylene from the original oil to the water column. In general, a week of retention time increased the BTEX content in the water column. Toluene exhibited an increasing content where it reached to 6.86% which showed a similar trend as ethyl benzene with 17.5% of release. Xylene however revealed a reverse trend that presented a decreasing trend of 7.44% that might be the result of re-association to the body of the floating oil. Benzene was not detected in 30 h and a week of retention times.

**BTEX in Floating Oil:** The release of BTEX from the floating oil to the water column were showed a general increasing trend in crude oil type A and an irregular trend in crude oil type B. In crude oil type A, the increase of time from 6 to 30 h and a week of retentions provided more release of BTEX from the floating oil to the water column. Toluene was observed to be remained 36.49% in the first 6 h followed by 30.98 and 27.38 percent in 30<sup>th</sup> h and a week after, respectively. Ethyl benzene was remained the same from 6<sup>th</sup> to 30<sup>th</sup> h at around 41% remaining then dropped 36.13 percent after a week of retention, indicating of around 5% increase in release from the floating oil. Xylene showed a similar trend to ethyl benzene where there was not a significant change from the 6<sup>th</sup> to the 30<sup>th</sup> h of



retention and remained around 33 percent then decreased to 29.30 percent, indicating of around 4% of more release of xylene. Benzene interestingly had a quick release from the floating oil from a 6<sup>th</sup> hour high of 21% to the 30<sup>th</sup> h of around 9.66% followed by a slight drop to 8.76 percent.

In crude oil type B, the increase of time from 6 to 30 h had significant release of toluene, xylene and benzene but ethyl benzene. Ethyl benzene showed a rapid re-association from 6<sup>th</sup> to the 30<sup>th</sup> hour while the re-association happened to toluene, xylene and benzene a week after the beginning of the test. Toluene was observed to be remained by 15.70% in the first 6 h followed by 6.86 and 10.31 percent in 30<sup>th</sup> h and a week after, respectively. Ethyl benzene was initially recorded 13.40% on the 6<sup>th</sup> h followed by 27.38 and 12.38 percent on 30<sup>th</sup> h and a week after. Xylene was in agreement with what toluene had presented. It was recorded 9.61% at the 6<sup>th</sup> h the followed by 5.61 and 20.22 percent for the 30<sup>th</sup> h and a week after. Benzene interestingly showed again the quick drop from the 9.85 to 1.94 followed by 3.58 percent for 6<sup>th</sup> and 30<sup>th</sup> h and a week after respectively.

The behavior of BTEX compounds in simulated spill condition was studied in this research. BTEX contents of spilled oil upon the contact to the surface of sea water released into the water column. This is observed to be a conservative behavior where benzene remained not detected in water from both light and heavy oils for the duration of the study in 3 different time intervals (Figure 2 and 3; left side). Ethyl benzene and xylene appeared to be in the highest abundance of release followed by toluene.

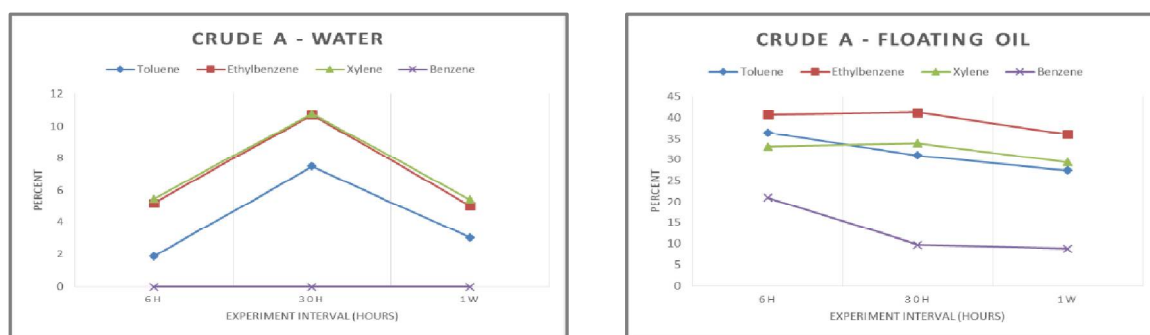


Figure 2. Release of BTEX in Crude A (light); Left: to Water and Right: from Floating Oil.

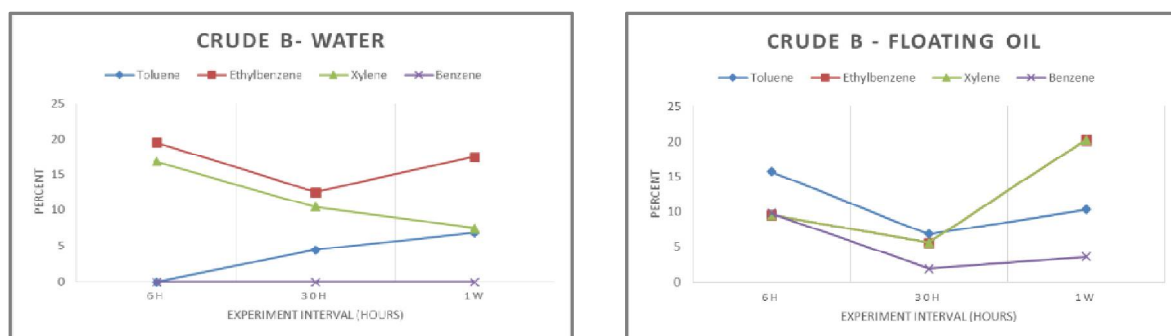
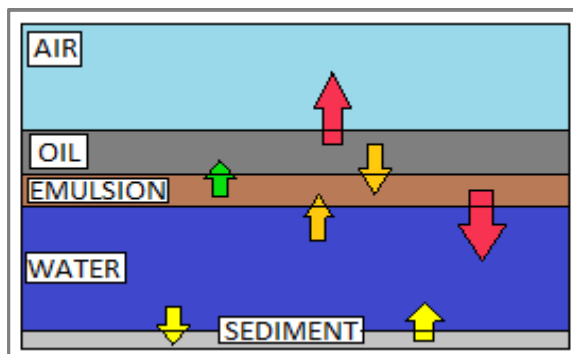


Figure 3. Release of BTEX in Crude B (heavy); Left: to Water and Right: from Floating Oil.

These 3 identified compounds (TEX) showed an increasing release from the 6<sup>th</sup> h to the 30<sup>th</sup> for the lighter oil however for the heavier oil the trend was irregular. Light oil has lower density and viscosity that helps the formation of emulsion layer (Figure 4). From the 6<sup>th</sup> to the 30<sup>th</sup> h, the 24 h of interval provides enough retention time of emulsification.

During this process, more oil is transferred from the oil layer to the emulsion layer and gradually released into the water column however after a week of waiting time this has been decreased and the

TEX compounds may return to the floating oil and re-associated through the emulsion layer. The phase change of TEX from oil to emulsion and water and vice versa is shown in figure 4 and the movement directions are pointed out using colored arrows.



**Figure 4.** Formation of various layers in a spill condition. Red arrows are considered harmful the human health and the environment and green shows a positive re-association of emulsified compounds to the oil. Arrows in yellow show potential of staying in the target layers.

The loss of BTEX from the floating oils (Figure 2 and 3; right side) follows an increasing trend for lighter crude oil where less compounds remain in the floating oil, however for the heavier crude, it shows an irregularity. The lighter oil (A) has the opportunity to release BTEX to both water column through the emulsion layer and to the air. This seems to be a reason where more BTEX compounds are being released to the air while meantime water does not show a significant receiving of them. Crude B however resists evaporation due to its density and viscosity and remains in the floating oil.

The theory of evaporation of BTEX from the floating oil to the air had been discussed elsewhere [3]. The total release of BTEX from the original crude oil to the air and emulsion layer has been mathematically calculated and presented in table 5. Among BTEX compounds Benzene showed the highest level of release to air and emulsion layer for both crudes A and B followed by toluene, xylenes and ethyl benzene. There are limited evidences from this study to prove which phase (either emulsion or air) receives more BTEX contents. Looking at the existing data in various time intervals and comparison to BTEX profile in water shows that most likely air receives more BTEX (exclusively benzene) than emulsion.

**Table 5.** BTEX Content Percent Remaining in Emulsion Layer and Air

BTEX	Air and Emulsion (Crude A)			Air and Emulsion (Crude B)		
	6h	30h	1W	6h	30h	1W
Toluene	61.63	61.54	69.58	84.3	88.75	82.83
Ethylbenzene	54.03	48.05	58.82	70.85	81.89	62.28
Xylene	61.38	55.32	65.26	73.56	84.01	72.34
Benzene	78.94	90.34	91.24	90.15	98.06	96.42

Njobuen wu *et al.* [7] mentioned that the solubility of benzene in water is higher than other BTEX compounds however polarity plays a different rule. BTEX compounds are partially dissolved in water then the remaining of them floats on the top of the water column, In this experiment, the environmental condition remain the same at all times that provided opportunity for all BTEX compounds to float at the surface then dissolve based on their polarity. Benzene is a nonpolar compound that has the tendency to remain in the oil phase than water due to the similarity in polarity. This can be a strong reason that benzene was not detected in the water column. TEX compounds showed some levels of miscibility that can be interpreted as per their partial polarity due to the alkylated attachments such as its methyl and ethyl additions. They were significantly detected in water column.

## APPLICATION

Oil spill is one of the most challenging environmental concerns [3]. A quick evaluation of an oil spill status assists the emergency response team to come up with an appropriate contingency plan. The existing methods require long sample preparation practice and several consequent runs to identify important chemical compounds [8]. This method is fast, reliable, easy to perform and to determine the presence of the most toxic and carcinogenic components of the oil such as BTEX amongst other alkylated aromatic compounds. In addition, environmental chemists and coastal authorities are able to evaluate when an ecosystem is possibly back to service after a spill incident using this set of data from this experiment in addition to other local and federal guidelines [10].

## CONCLUSION

The PIONA analyses of our simulated spill delivered detailed hydrocarbon profiling of BTEX compounds. There were hundreds of compounds that were identified within a single GC run including around 50 benzene species that are usually not being reported to regulatory organizations. The study concludes that the threat of releasing toluene, ethyl benzene and xylenes to the water column is greater than benzene, however the possibility of benzene presence in water cannot be underestimated when dealing with drinking water. The oil-water emulsion layer and air were observed to contain more portion of BTEX than water column and the floating oil. Air seems to be an emerging destination for evaporated BTEX that further cause significant health concerns. The floating layer of oil, in the real marine environment, will be exposed to various physical forces and change such as water turbulence and waves that makes this interpretation more complicated. It seems that an immediate cleanup action remains as the best possible strategy to mitigate release of BTEX to the air. The presence of benzene in the water column must be determined with further analysis using GC-MS to target lower concentrations. The PIONA analysis provides a quick and affordable picture of the spill condition to provide basic information to authorities to make a better decision and helping to adjust quickly an existing contingency plan. Benzene species, as the result of PIONA testing provides a unique view of emerging and potential toxic and carcinogenic compounds to the decision makers and health authorities. This research can be replicated using fresh water sources to provide a picture of possible scenarios if a spill happens in inland waters such as lakes. The collection and analysis of air samples would be a good addition to a similar study in the future to provide further information on toxic and carcinogenic compounds coming to air from a potential spill. This application can be used to further investigate the status of an oil spill in higher magnitude with a simple preparation step and single GC run.

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