

# Characterization and assessment of Al Ruwais refinery wastewater

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## Abstract

An extensive bulk parameters and component-based analysis was carried out for Al Ruwais refinery wastewater during the period of June 2002–June 2003. The program included identification of major process and utilities wastewater streams and quantification of these streams relative to the total wastewater generated from all refinery processes. In addition to the high biochemical oxygen demand (BOD) and chemical oxygen demand (COD) reported levels, the analysis showed high concentrations of polyaromatic hydrocarbons (PAHs) and phenolic compounds in the major wastewater streams. Polychlorinated biphenyls (PCBs) were also detected in few streams. The caustic water was found to have the highest levels of the pollutants mentioned above. Dilution of the wastewater with process cooling water serves as the main treatment approach applied to the effluent wastewater before disposal into the sea. Primary as well as secondary treatment units are thought to be essential and strongly recommended to reduce the pollutants to levels below the United Arab Emirates (UAE) standards for marine discharge.

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## 1. Introduction and objectives

Petroleum refining involves the transformation of crude oil into final useful products such as gasoline, gas oil, kerosene and jet fuel, and petrochemical feed stocks. The refined products are produced after a series of separation and treatment processes. After initial crude desalting and fractionation, several treatment and conversion processes are employed to reach the final blending stocks. Examples of conversion processes include thermal and catalytic cracking, steam and catalytic reforming, isomerization, alkylation and lube oil units. Treatment processes on the other hand include naphtha and gas oil desulfurization, sour water strippers and catalyst regeneration units.

Petroleum refining uses relatively large quantities of water, especially for cooling systems, desalting water, stripping steam, and water used for flushing during maintenance and shut down. In addition, surface water runoff and sanitary wastewaters are accounted in the wastewater system. The quantity of wastewater generated and their characteristics depend on the process configuration. As a general rule, approximately 3.5–5 m<sup>3</sup> of wastewater

are generated per tonne of crude oil processed when cooling water is recycled [1].

The composition of the wastewater generated from a refinery is very much dependent on the complexity of the process. Refineries are classified as either hydroskimming, which comprise distillation, reforming and desulfurization, or complex, which include a catalytic cracking unit additional to the hydroskimming refinery. The wastewater may contain a biochemical oxygen demand (BOD) and chemical oxygen demand (COD) levels in the range of 150–350 and 300–800 mg/l, respectively, phenols level of 20–200 mg/l, oil level up to 3000 mg/l, suspended solids of more than 100 mg/l, benzene levels of 1–100 mg/l, benzo(a)pyrene levels of 1–100 mg/l, heavy metals level of 0.1–100 mg/l for chrome and 0.2–10 mg/l for other organic and non-organic pollutants [1].

Most of the studies found in the literature reported the composition of refinery wastewater in terms of bulk parameters such as BOD and COD [2–5]. These parameters are useful in the design and the assessment of the treatment stages. However, they do not give the specific composition of the wastewater that might be essential for specific component treatment and for polishing of the wastewater after secondary treatment. Knowledge of such composition is also useful in marine pollution studies when it is needed to track specific pollutants generated from the refinery in

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the coastal surface water, especially if the coastal water is used as a source for drinking water treatment plant such as desalination plant. Specific component analysis is also necessary for tertiary treatment when a specific component is targeted for removal.

Stepnowski et al. [6] analyzed a refinery wastewater and found out that the predominant fraction was the light petroleum hydrocarbons (up to C12). However, they could not specify the peak groups of aromatic hydrocarbons. Their analysis indicated that most of the heavier fractions were eliminated during the preliminary oil separation stage. Based on their results, the total mean concentration of saturated aliphatic hydrocarbons and aromatic compounds in the studied wastewater was 1534 ppm.

The current work deals with the characterization of a refinery wastewater located in Al Ruwais area at about 350 km west of Abu-Dhabi city, the capital city of United Arab Emirates (UAE). The work serves other objectives to identify potential adverse impacts of refinery wastewater disposed on the marine water on the quality of drinking water produced from a nearby coastal desalination plant. The characterization of the wastewater was performed on two levels; general wastewater parameters determination such as BOD, COD, TPH, sulfate and Kjeldahl nitrogen, and component specific characterization. Due to the special nature of work objectives, aromatic hydrocarbons that show higher persistence in their fate transport properties in surface water, such as PAH, PCB and phenolic compounds were selected in the component specific characterization. The BTEX group, known to exist abundantly in refinery wastewater, was therefore excluded from the current study due to their high

volatility and low persistence in terms of their fate transport environmental properties. An attempt was also made to relate the composition of the wastewater to the various processes within the refinery. Additionally, the contribution of the various wastewater streams to the final wastewater were identified and quantified.

**2. Refinery description**

The original hydroskimming refinery was designed to process 120,000 barrels of oil per stream day (BPSD), mainly to cater for the export market. Today, the refinery processes 20,000 BPSD above its design capacity. Two condensate processing units were added later with a total capacity of 280,000 BPSD. A simplified schematic diagram for the Ruwais refinery processes is shown in Fig. 1.

In the hydroskimming unit, the crude distillation unit produces kerosene, naphtha, heavy and light gas oil and atmospheric residue. The kerosene is sent to the kerosene hydrodesulfurization unit to reduce the sulfur content and the kerosene leaving the unit is then sent to the kerosene storage. The naphtha is sent to naphtha hydrodesulfurization unit then to the catalytic reformer to produce reformate. The gas oil from the crude unit is sent to the gas oil hydrodesulfurization and the desulfurized gas oil is then sent to the storage. Some naphtha are produced in this process and later sent to the naphtha storage. The atmospheric residue is sent to the vacuum distillation unit, which distills the feed into heavy and light vacuum gas oil and vacuum residue. The vacuum residue is sent to the bunker where it is used as fuel

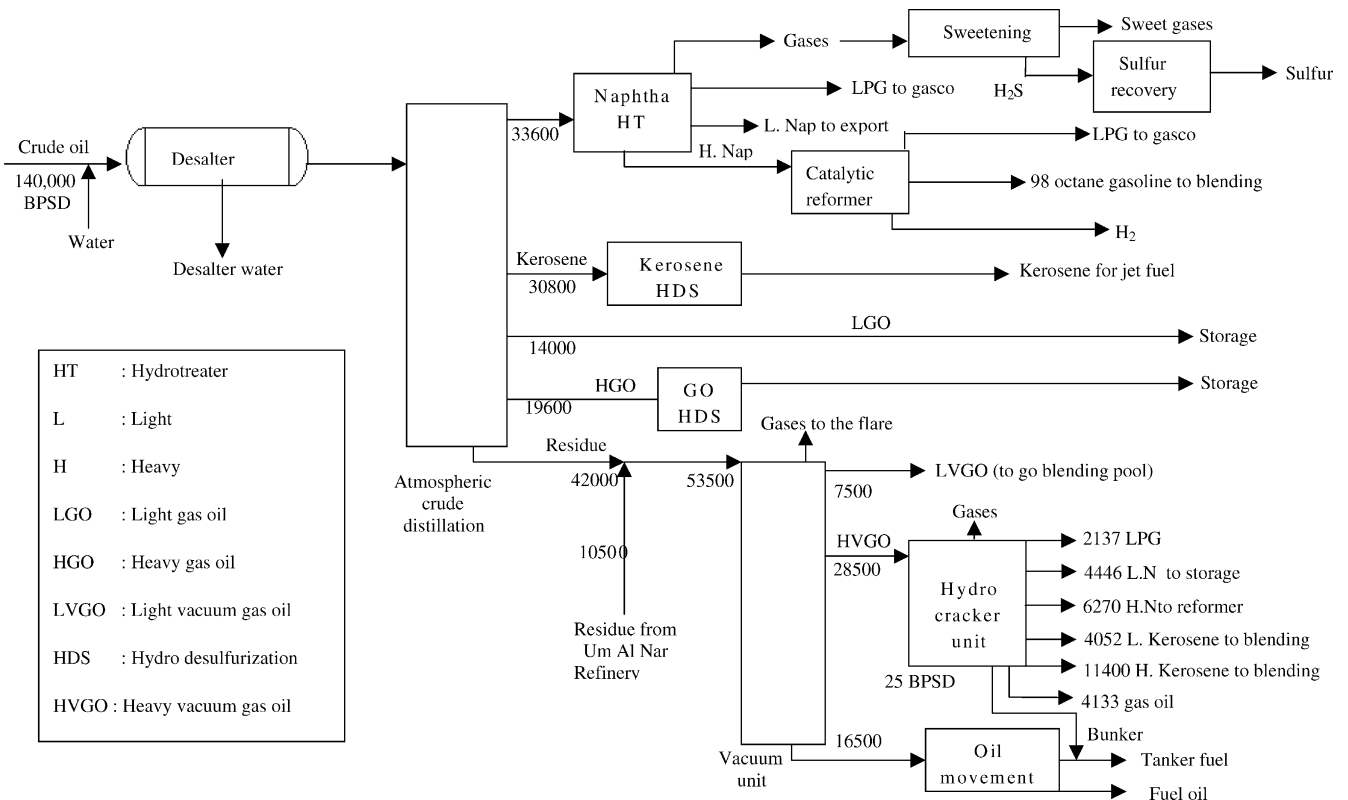


Fig. 1. Schematic diagram of the Ruwais refinery major processes (values in BPSD).

oil. The vacuum gas oil is sent to the hydrocracker where LPG and naphtha are produced.

### 3. Sampling and analysis of oily wastewater

The first step in the sampling plan was to identify and locate the major streams contributing to the total wastewater discharged to the treatment system. The second step was to quantify the wastewater in each of these streams. Fig. 2 shows the major wastewater streams with the percentage contributing to the total wastewater discharged to the wastewater treatment system. Since some streams do not have any flow meters or measuring devices, the percentages shown on each stream are somewhat approximate averages and in some cases were based on simple measurement for open streams such as the flare system. For closed streams, it is based on design data reported in the refinery manual.

As shown in Fig. 2, the oily water received at the treatment system is generated from various processes such as the desalter, treated sour water, process drainage, pump flushes, laboratory and sanitary wastewater and the flare system. The oily water from these processes is routed to a corrugated plate interceptor (CPI) which separates the free oil from the wastewater. The CPI is not designed to remove any dissolved or dispersed oil from the wastewater. On the average, 150 m<sup>3</sup>/h of wastewater is treated and sent to the final mixing pit where the wastewater is diluted with around 10,000 m<sup>3</sup>/h of cooling water used in the refinery processes. A small amount of caustic solution is also discharged into the mixing pit. This solution is generated from the catalyst regeneration in the kerosene sweetening processes. The caustic stream has high COD and relatively high phenol concentration. The CPI effluent is characterized by its high COD and PAH concentration. However, due to the large volume of cooling water, most of these parameters appeared to be very small in the effluent discharged from the mixing pit to the sea.

Samples were taken on a monthly basis from the period of June 2002 to June 2003. These were collected from either sampling ports within the process, from the open mixing pit or the streams discharged to sumps before the CPI. Samples taken from sampling locations were preserved and kept in ice boxes and then transferred to laboratory for analysis. Analyses were repeated for reproducibility of results.

BOD measurements were done using standard BOD test. The COD in this analysis was measured using the manganese III digestion method. For TPH analysis, the sample was acidified to pH < 2 and the petroleum hydrocarbons from wastewater samples were extracted using fluorocarbon-113. Infrared analysis of the solution was performed by direct comparison with standards. PAHs from a known volume of water were extracted using methylene chloride and the solvent was carefully removed by evaporation. Sample extracts are cleaned, if necessary, using solid phase extraction (SPE) using PAH SPE columns. The PAH components in the extract were determined by HPLC using ultraviolet fluorescence and photodiode array detectors simultaneously. Organic nitrogen was converted into ammonium sulfate when digested with concentrated sulfuric acid in the presence of a catalyst. Ammonia liberated after neutralizing the acid by alkali was steam distilled into boric acid solution. The amount of ammonia was estimated by titration using standard sulfuric acid and converted into total Kjeldahl nitrogen. The sulfate content of the wastewater samples was determined using ICP-AES method. The prepared sample solution was aspirated through nebulizer and the resulting aerosol was transported to the plasma torch where excitation occurs. Element specific emission spectra were produced by radio-frequency inductively coupled plasma. The spectra were dispersed by a grating spectrometer, and intensities of the line spectra were monitored at specific wavelengths by a charged coupled detector. A fitted background correction was used to correct the blank signal and matrix effect. For phenol determination, samples were extracted using hexane and prior to analysis, the extracts

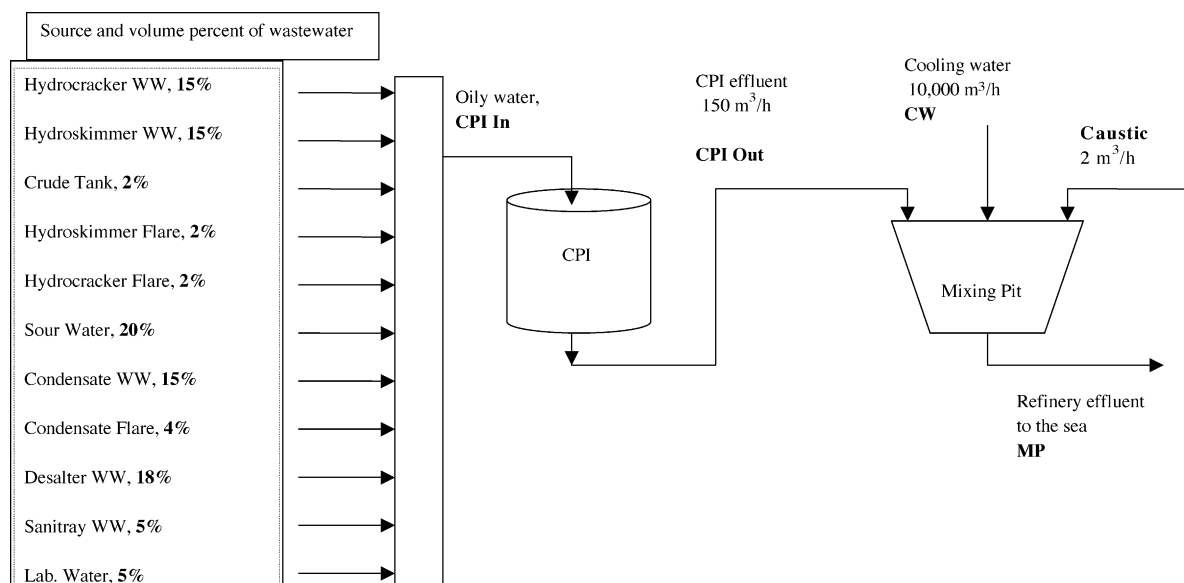


Fig. 2. The oily wastewater treatment system.

Table 1  
Characteristics of treated industrial wastewater at point of discharge into the sea in mg/l suggested by UAE standards

Parameter	Symbol	Suggested limit
Biochemical oxygen demand	BOD <sub>5-20</sub>	50
Chemical oxygen demand	COD	100
Total Kjeldahl nitrogen (N)	TKN	10
Total petroleum hydrocarbons	TPH	15
Phenols		0.1

were cleaned up, as necessary, and the solvent exchanged to hexane.

The target phenols were derivatized with pentafluorobenzyl-bromide (PFBBBr) and analyzed by gas chromatography. PCBs were extracted from a known quantity of sample using an appropriate matrix specific sample extraction technique. Extracts were subjected to sulfuric acid/potassium permanganate cleanup and the cleaned extract was analyzed by gas chromatography on a narrow-bore fused silica capillary column and electron capture detector (GC/ECD).

#### 4. Results and discussion

All the streams shown in Fig. 2 were characterized in terms of the bulk and specific compound parameters. The presence of the pollutants in these streams was tracked back to their origin within the refinery processes. This criteria, however, is not always accurate as some refinery operations are not continuous. Examples would be processes drainage and system flushing. Other process conditions such as small units shut down or maintenance operations may affect the quantity and quality of the wastewater. The stream concentrations were compared to UAE standards as shown in Table 1.

##### 4.1. Biological oxygen demand (BOD)

The BOD of refinery wastewater is usually lower than the BOD of municipal wastewater. This is due to the nature of the refinery wastewater which consists of organic materials that are partially biodegradable. The BOD of the wastewater streams shown in Fig. 3 indicates that the effluent from the CPI still

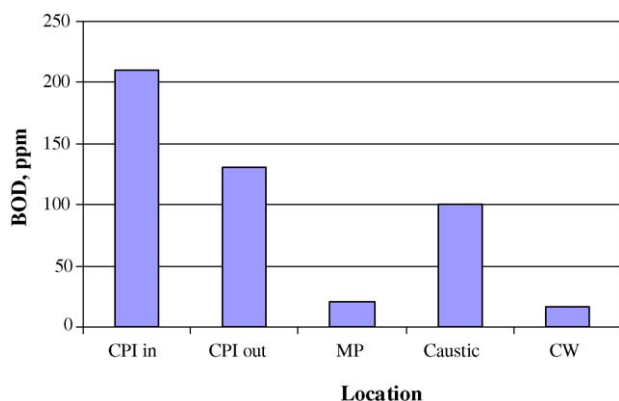


Fig. 3. BOD values of the wastewater streams of Fig. 2.

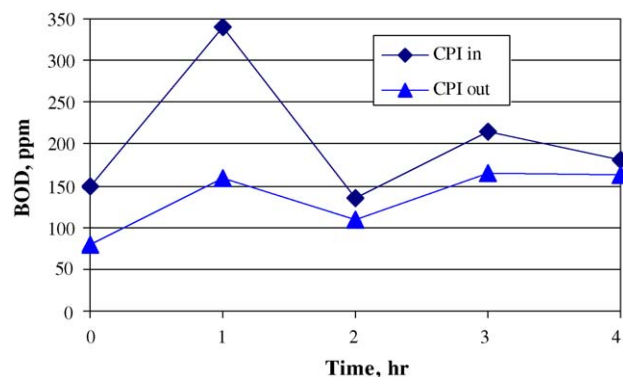


Fig. 4. Variation of BOD with time for the CPI.

carries a BOD level that is higher than the standard (50 ppm) accepted for discharge to the marine environment [7]. The average value in the CPI effluent is about 130 ppm. The BOD value tends to fluctuate due to operational changes in the process in which drainage and flushing are performed on intermittent basis. Fig. 4 shows the BOD fluctuation in the influent and effluent streams of the CPI during a 4-h period. The results show that the CPI has a BOD removal efficiency that ranges from 15 to 54% with an average of 31.9%. The BOD level in the CPI varies due to variation in characteristics of influent wastewater to the CPI. Some of the wastewater streams are intermittent such as the crude tank drainage and the sanitary water. The sanitary wastewater could contribute to the high level of BOD in the CPI influent. The CPI removes the free oil from the wastewater but has limited removal efficiency to the dissolved and the dispersed oil droplets. A possible explanation to the lower BOD removal in some cases could be attributed to this lower removal efficiency of non-free oil droplets. The removed BOD could be the free BOD while the dissolved one remains in the effluent.

Caustic water used for the separation of mercaptans from gasoline and kerosene is usually rich in phenols, sulfides and mercaptans. Therefore, the high level of the BOD in the spent caustic can be attributed to the level of degradable components such as phenols. The BOD of the mixing pit discharge is low (17 ppm) and far below the standard for wastewater discharge. This is due to the large volume of cooling water that dilutes both the CPI effluent and caustic streams.

##### 4.2. Chemical oxygen demand (COD)

COD represents most of the organic compounds present and the oxidizable mineral salts such as sulfur compounds. For refinery wastewater, the international standard of COD for marine discharge is 120 ppm for a hydroskimming refinery and 150 ppm for a complex refinery [8]. However, the UAE Federal Environmental Agency has set a 100 ppm for industrial water discharged to the sea [7]. Al Ruwais refinery is however considered as a complex refinery due to the expansion planned for unleaded gasoline and reduced sulfur content in gasoline and diesel. Analysis of the CPI influent stream has shown an average of 420 ppm while the effluent dropped to 242 ppm with

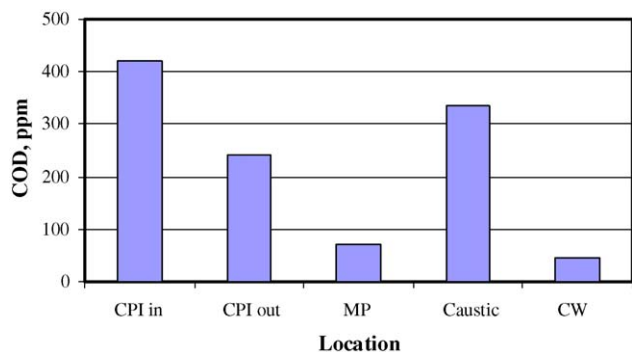


Fig. 5. COD levels in wastewater streams of Fig. 2.

about 42% removal efficiency for COD. This is very close to values published in the literature for CPI [9].

The COD for the streams of Fig. 2 is shown in Fig. 5. The great dilution occurring at the mixing pit reduces the effect of high COD in both caustic and CPI effluent. The high level of COD in the caustic stream is attributed to the phenols and sulfide levels as will be shown later.

#### 4.3. Total petroleum hydrocarbons (TPH)

TPH parameter gives a quantification of all the hydrocarbons in the wastewater. It represents both the soluble and insoluble parts. Hydrocarbons have variable solubility in water. While paraffinic hydrocarbons have very small solubility in water, aromatics are very soluble. For example, *n*-hexane has a solubility of 9.5 ppm while benzene has a solubility of 1780 ppm. Hence, pretreatment process such as the CPI should be more effective in removing the paraffinic part leaving most of the non-volatile aromatics dissolved in the wastewater.

The TPH concentration of the various wastewater streams is shown in Fig. 6. The high TPH in the CPI is attributed mainly to the free hydrocarbons (non-aromatic), which upon treatment is reduced in the CPI effluent by 90%. In the caustic stream, the TPH is due mainly to the phenolic compounds. Due to the heavy dilution, the TPH concentration in the MP discharge is very small (2.4 ppm). No TPH was detected in the cooling water stream. The CPI effluent and the caustic streams contain more than permissible amounts of petroleum hydrocarbons [7].

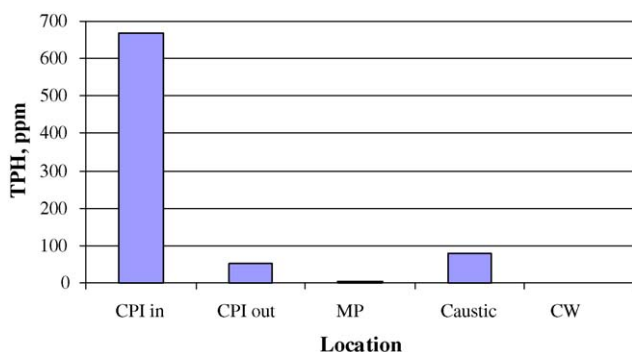


Fig. 6. TPH concentration in the wastewater streams (MP = 2.4 ppm; CW = ND; standard = 15 ppm).

Table 2  
Major PAHs found in the wastewater streams of Fig. 2 (ppm)

PAH	CPI in	CPI out	Caustic
Naphthalene	30.7	0.94	20
Acenaphthylene	3.84	0.02	11.8
Flourene	21.6	ND	0.39

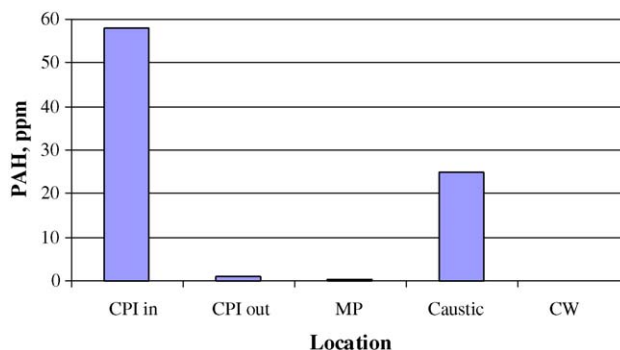


Fig. 7. PAH concentration in the wastewater streams (MP = 0.2 ppb; CW = ND).

#### 4.4. Polyaromatic hydrocarbons (PAHs)

The presence of PAHs in wastewater streams is limited by several regulations because these compounds are highly carcinogenic. There are many PAHs that may exist in refinery wastewater with naphthalene being the predominant component. In the current analysis, the major components that were detected in the CPI and the caustic streams are shown in Table 2. Most of the PAHs are removed in the CPI leaving around 1 ppm in the CPI effluent. Only one PAH was detected in the mixing pit and with a very low level (0.2 ppb of naphthalene). The high removal of PAHs taking place in the CPI can be related to the high volatility of PAHs, in particular the Naphthalene, given the fact that the temperature during the time of sampling was always higher than 45 °C (Fig. 7).

Naphthalene is the predominant component present in most of the wastewater streams, with the highest in the condensate flare (70 ppm). Table 3 shows the concentration of naphthalene in most of the wastewater streams entering to the CPI. The last column in the table shows the stream contribution to the total naphthalene in the CPI influent in percentage. This estimation is based on the relative percentages of the wastewater from these streams to the total wastewater delivered to the CPI.

Table 3  
Naphthalene concentration in major refinery wastewater streams

Stream	Wastewater to CPI (%)	Concentration (ppm)	Contribution to CPI (%)
Crude tank drainage	2	0.31	0.03
Desalter water	18	4	3.4
Hydroskimmer oily water	15	32	21
Hydrocracker oily water	15	35	23
Hydrocracker flare	2	1.7	0.14
Condensate flare	4	70	12



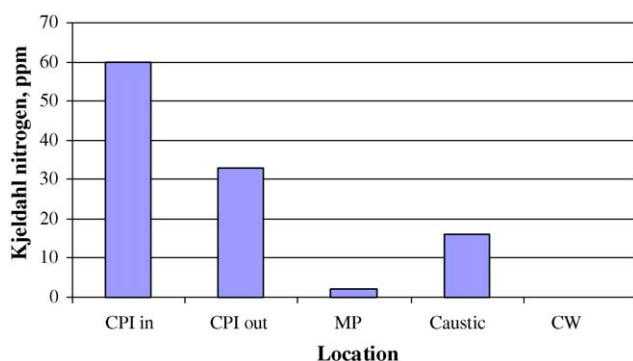


Fig. 8. Kjeldahl N in the wastewater streams (MP=1.8; CW=ND).

The high level of the PAH in the CPI influent is attributed to the high level in some of the feeding streams of wastewater to the CPI. In particular, the hydroskimmer oily water and the condensate flare streams as shown in Table 3. Small quantities were also detected in the desalter water.

#### 4.5. Total Kjeldahl nitrogen (TKN)

The total nitrogen in wastewater consists of both inorganic nitrogen and Kjeldahl nitrogen. Kjeldahl nitrogen represents all the organic nitrogen and  $\text{N}\cdot\text{NH}_4$  in the wastewater. The standard for discharge of municipal wastewater in France is limited to 40 ppm over 24 h. International standards for refinery wastewater vary from 10 to 100 ppm. The standard for marine discharge in UAE is 10 ppm [7].

$\text{NH}_4^+$  is the most common nitrogen compound present in refinery wastewater, which results from two operations; the injection of  $\text{NH}_3$  at the top of the fractionating column to neutralize potential  $\text{H}_2\text{S}$ , which forms  $\text{HSNH}_4$  or the hydrogenation of organic nitrogen when the crude is refined.

The analysis of the CPI streams showed a high concentration of the organic nitrogen as shown in Fig. 8. The high concentration is attributed to the high level of nitrogen in the crude tank drainage water that reached to 249 ppm and to some extent to the hydroskimmer (10.1 ppm) and hydrocracker oily water (6.4 ppm). The contribution of various streams to the total organic nitrogen in the CPI is shown in Table 4. The CPI effluent contains as much as half of the Kjeldahl nitrogen in the influent. The mixing pit has always shown a relatively low concentration of Kjeldahl nitrogen (less than 2 ppm). No Kjeldahl nitrogen was detected in the cooling water stream.

Table 4  
Contribution of the wastewater streams to the organic nitrogen concentration in the CPI

Stream	Wastewater to CPI (%)	Concentration (ppm)	Contribution to CPI (%)
Crude tank drainage	2	249	27
Desalter water	18	21	18.9
Hydroskimmer oily water	15	10.1	7.8
Sour water	20	6.3	6.2
Hydrocracker oily water	15	6.4	6.4

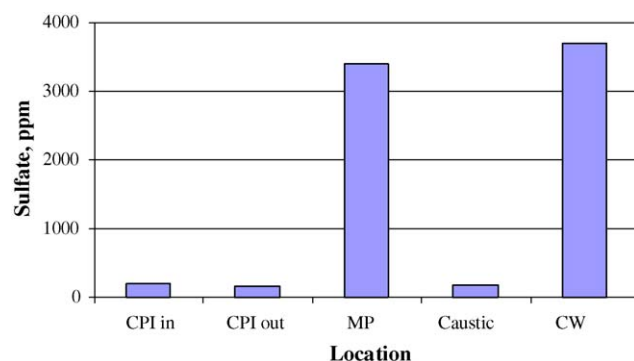


Fig. 9. Sulfate concentration in the refinery wastewater streams.

#### 4.6. Sulfate ( $\text{SO}_4^{2-}$ )

In this work, sulfate was chosen to represent the many sulfur compounds that exist in any refinery wastewater. Sulfur compounds exist in almost all of the wastewater streams. This is due to the presence of hydrogen sulfide, mercaptans and disulfides in the crude oil. Many of the sulfur compounds are removed from the intermediate and finished products to prevent corrosion problems in the process streams and vessels, and to prevent poisoning of the catalysts. Finished products have to contain sulfur in percentage permitted by standards and environmental regulations.

The average concentration of sulfate in the major streams is shown in Fig. 9. The high concentration of sulfate in the CPI influent (around 200 ppm) is due to the high concentration of sulfate in the treated sour water (up to 800 ppm) which is discharged to the CPI after treatment. Since the caustic solution is used for mercaptans removal from the kerosene, a high level of sulfate (180 ppm) and other sulfur compounds was expected in the caustic wastewater stream. The extremely high value of sulfate in the mixing pit represents the normal sulfate concentration in seawater, which is used for dilution in the mixing pit. The share of various streams of sulfate to the total concentration in the CPI influent is shown in Table 5.

#### 4.7. Phenols

Phenols need to be removed from wastewater streams because they are harmful and form other harmful products during chlorination of seawater such as chlorophenols. In refineries, phenols are expected to exist in high concentration

Table 5  
Contribution of the wastewater streams to the sulfate concentration in the CPI

Stream	Wastewater to CPI (%)	Concentration (ppm)	Contribution to CPI (%)
Crude tank drainage	2	559	5.6
Desalter water	18	137	12.3
Hydroskimmer oily water	15	111	8.3
Sour water	20	410	41
Hydrocracker oily water	15	143	10.7
Condensate oily water	15	136	10.2

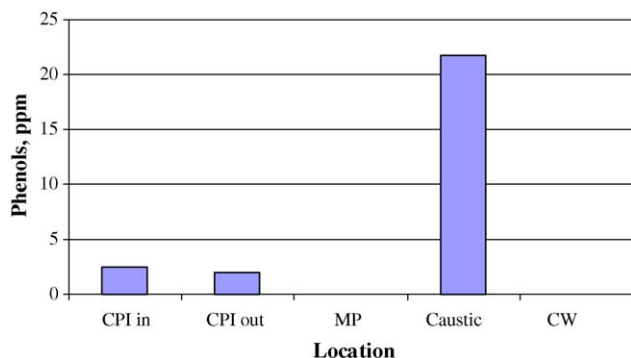


Fig. 10. Phenols concentrations in the wastewater streams (MP = ND; CW = ND).

in the spent caustic streams. The legislations in the UAE limit the total phenols in industrial water discharged to the marine environment to 0.1 ppm.

Fig. 10 shows the concentration of phenols in the CPI and caustic streams. As a pretreatment process, the CPI is designed to remove any free or floating oil. Part of the phenol is expected to be in the free oil and hence it is removed in the CPI. However, the phenol in the dispersed or the emulsified oil is not removed. Around 23% of the phenols are removed from the main wastewater stream in the CPI. The analysis showed that CPI effluent carries a high concentration of phenols (around 2 ppm).

Phenols in the spent caustic characterize the feed that undergoes the sweetening process. In Al Ruwais refinery, this feed is kerosene and the type of phenolic compounds is those present in kerosene. In the current analysis, an average concentration of 21.7 ppm of phenols was detected in the spent caustic stream. Due to the high dilution, no phenols were detected in the mixing pit.

Tracking back these phenols in the process wastewater streams it was found out that the major contributors of phenols in the CPI are the condensate oily water, the desalter wastewater and the sour water as shown in Fig. 11 and Table 6. The condensate wastewater contributes about 51% of the phenols in the CPI influent; the desalter contributes about 11%, while the sour water contributes 9% of the phenols in the CPI influent. Table 7 shows the main phenols that were detected in the wastewater streams.

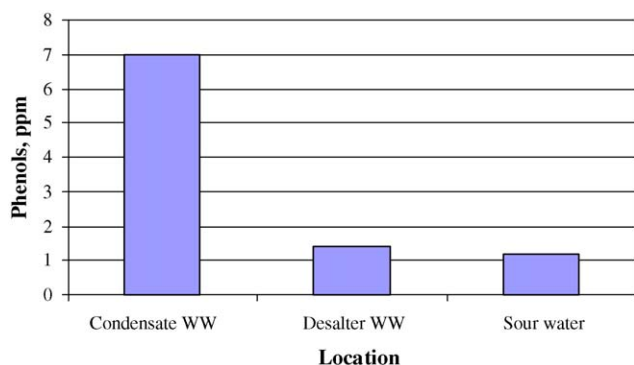


Fig. 11. Phenols in major process wastewater streams.

Table 6

Contribution of the wastewater streams to the phenol concentration in the CPI (total phenols in CPI = 2.76 ppm)

Stream	Wastewater to CPI (%)	Phenol concentration (ppm)	Contribution to CPI (%)
Condensate oily water	15	7	50.82
Desalter water	18	1.5	10.89
Sour water	20	1.21	8.78

Table 7

Major phenolic compounds found in the wastewater streams

Compound	CPI in	CPI out	Caustic
Phenol	0.73	0.62	2.5
<i>m</i> -Cresol	0.88	0.65	6.37
<i>o</i> -Cresol	0.65	0.55	7.97
<i>p</i> -Cresol	0.5	0.31	5.66
2,4-Dimethylphenol	0.64	0.47	6.14
Total + others	3.7	2.6	29.65

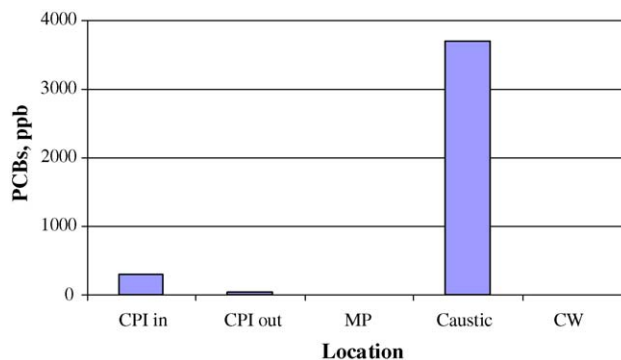


Fig. 12. PCBs concentration in wastewater streams (MP = ND; CW = ND).

#### 4.8. Polychlorinated biphenyls (PCB)

PCBs are oily liquids present within the oil and grease. They are stable components but can be biodegraded. They are known to cause liver and kidney damage and could cause miscarriages, premature deliveries and cancer. There is no standard for discharge in marine environment but EPA has established a 0.5 ppb MCL of PCBs in drinking water.

Fig. 12 shows the maximum concentration of PCBs detected in the wastewater streams. Smaller values were also detected in some analysis. The range of PCBs in the wastewater streams is given in Table 8.

From Fig. 12 and Table 8, it is clear that most of the PCBs are concentrated in the caustic stream. From all the samples and analysis carried no PCBs were detected in the mixing pit

Table 8

Concentration range of PCBs in the wastewater streams (in ppb)

Stream	Minimum value	Maximum value
CPI out	10	67
CPI in	22	375
Caustic	565	3900

Table 9  
Major PCBs found in wastewater streams (in ppb)

Component	CPI in	CPI out	Caustic
PCB-8	86.1	23.4	550
PCB-101	31.4	4.41	2030
PCB-77	16.1	2.41	1208

discharge to the sea. The major PCBs that were detected are given in Table 9.

## 5. Conclusions and recommendations

An extensive sampling and analysis was carried out for Al Ruwais refinery wastewater. The sampling analysis showed some high concentrations of phenols and PAHs in the oily water separation unit effluent and in the caustic water stream. The caustic stream flow rate is less than 2% of the oily water leaving the separator. With respect to the tested PAHs, naphthalene appeared to have the highest concentration. Phenol and cresols were the major components from the phenolic group. Analysis of PCBs has also showed the presence of these compounds in both streams.

The analysis also showed the presence of the PAH and phenolic groups in some streams more than others. Examples of such streams are the treated sour water, the desalter water and the flare system water. These streams need to be sampled and analyzed routinely in order to detect the variation of the pollutants concentration with process operation.

Dilution of the above two streams with cooling water has brought the composition of PAHs and phenols to levels below the standard for discharge to marine environment. To reduce the concentration of the targeted pollutants before dilution, primary treatment unit such as dissolved air flotation could be installed

in order to reduce the concentration of dispersed oil from the wastewater. Experimental work should help to determine the efficiency of such treatment.

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