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Detection and Identification of Organics and Metals from Industrial Wastewater by ICP-AES, FTIR and GC-MS

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ABSTRACT

It is found that large number of printing and dying industries pollute water by discharging the wastewater containing large proportion of organics and metals. It causes harmful effect on human and plant life. Hence the area of interest is detection of organic compounds and heavy metals. Various analytical methods have been used for characterization of wastewater but out of these methods, some modern techniques like ICP-AES, FTIR, and GC-MS was used in the present study. This study was carried out for the detection and identification of metals and organic compounds from the industrial wastewater. The probable organics present in wastewater were determined by FTIR and GC-MS. For metal detection ICP-AES technique has been used. These results are useful to interpret the extent of purity of water and its harmful effects. Also this analysis can highlight the urgent need for continuous monitoring which will effect on environment.

1. Introduction

Industrial wastewater is the main source which pollutes water. The high concentration of pollutants in the form of organics and metals in the industrial waste water is mainly due to use of various dyes. A dye is natural or synthetic compound which is used for coloring products. Generally dyes are widely used in various industries like textile industry, food industry, printing and pharmaceutical industry, etc. These industries continuously release untreated effluents into rivers and other common sources of water. It is found that the largest consumer of dye stuffs is textile industry [1-3]. Now a day in case of water pollution high concentration of organics is most serious environmental problem [4].

Especially the waste generated by textile dyeing and printing industry contains variety of organic compounds and poisonous heavy metals [3]. The detection of dyes is a difficult process because of the large variety of functional groups in different dyes and their diverse properties [1-3].

The heavy metals and organic compounds affect the quality of water. These heavy metals enter in the human body by different pathways and causes harmful effects [3]. The organic compounds are carcinogenic and mutagenic it affects the quality of river water and indirectly human health [5-8].

2. Experimental Methods

A sample was extracted with dichloromethane (CH_2Cl_2) and organics extracted in organic layer were concentrated and sent for FTIR and GC-MS studies at SICART, Vallabh Vidya Nagar, Gujarat.

Also for the metal detection by ICP-AES technique, the samples were digested in HNO_3-HCl solution [9-11] at SICART, Vallabh Vidya Nagar, Gujarat. The major five peaks were recorded by GC-MS. Five different samples of wastewater from dye and textile industry were collected from Vapi and GIDC-Surat, India. The chemicals used were purchased from LOBA chemicals. The solutions were prepared by using double distilled water.

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3. Results and Discussion

3.1 FTIR and GC-MS Studies

The results obtained during the course of present study are given in Table 1-4. A sample was extracted with dichloromethane (CH_2Cl_2) [9-11] and organics extracted in organic layer were concentrated and sent for FTIR and GC-MS studies at SICART, Vallabh Vidya Nagar, Gujarat. FTIR technique is useful for detection of functional groups. The FTIR spectra of sample-1 and sample-2 are shown in Fig. 1 and Fig. 2 respectively. Below each FTIR spectra functional groups detected are tabulated in Table 1 and 2 respectively.

Table-1 IR frequencies and detected functional groups from FTIR spectra (sample 1)

IR Frequencies	Functional groups and Class obtained	Assignment and remarks
720.33 cm^{-1}	Ar-OH in Phenols	OH out-of-plane deformation
802.55 cm^{-1}	1,2,3,4-tetrasubst benzenes	CH out-of-plane deformation
1021.20 cm^{-1}	Carbon ring in cyclic compounds	Ring breathing mode, strong in Raman
1103.79 cm^{-1}	C-NH_2 in 1^0 aliphatic amines	C-N stretch
1158.10 cm^{-1}	C=S in thiocarbonyl compounds	C=S stretch; strong in Raman
1229.62 cm^{-1}	C-C-N in amines	C-C-N bending
1261.28 cm^{-1}	C-O-C in esters, lactones	C-O-C antisym stretch
1366.53 cm^{-1}	NO_2 in aliphatic nitro compounds	NO_2 sym stretch
1377.62 cm^{-1}	Isopropyl group	CH_3 deformations (two bands)
1463.29 cm^{-1}	CH_3 in aliphatic compounds	CH_3 antisym deformation
1540.22 cm^{-1}	NO_2 in aromatic nitro compounds	NO_2 antisym stretch
1632.98 cm^{-1}	C=O in β-diketones	C=O stretch; enol form
1713.56 cm^{-1}	C=O in ketones	C=O stretch
1742.52 cm^{-1}	C=O in esters	C=O stretch
2851.27 cm^{-1}	CH_3 and –CH_2– in aliphatic compounds	C-H antisym and sym stretching
2921.08 cm^{-1}	$CH_3$and –CH_2– in aliphatic compounds	C-H antisym and sym stretching
3433.02 cm^{-1}	-NH_2 in aromatic amines, primary amines and amides	N-H stretch

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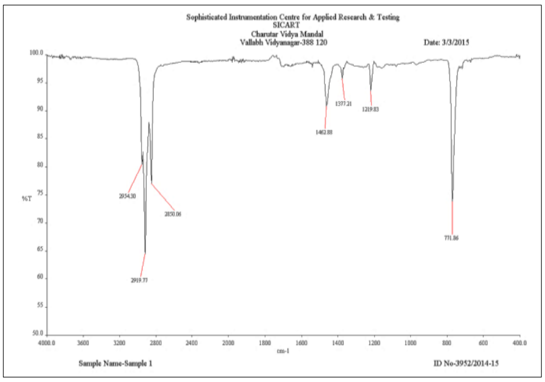


Fig. 1 FTIR spectra of dichloromethane extracted wastewater (sample-1)

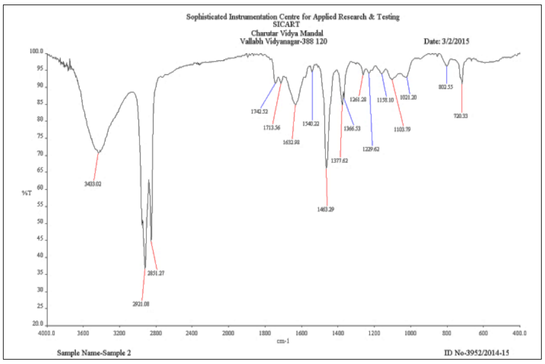


Fig. 2 FTIR spectra of dichloromethane extracted wastewater (sample-2)

Table-2 IR frequencies and detected functional groups from FTIR spectra (sample 2)

IR Frequencies	Functional groups and Class obtained	Assignment and remarks
771.86 cm^{-1}	1,2,3-trisubst benzenes	C-H out-of-plane deformation (two bands)
1219.83cm^{-1}	C-O-C in vinyl ethers	C-O-C antisym stretch
1377.21 cm^{-1}	CH_3 in aliphatic compounds	CH_3 sym deformation
1462.88 cm^{-1}	CH_3 in aliphatic compounds	CH_3 antisym deformation
2830.06 cm^{-1}	$-CH_3$ attached to O or N	CH stretching modes
2919.77 cm^{-1}	CH_3 and $-CH_2-$ in aliphatic compounds	CH antisym and sym stretching
2954.30 cm^{-1}	CH_3 and $-CH_2-$ in aliphatic compounds	CH antisym and sym stretching

The GC-MS technique is advanced and commonly used for determination of organic compounds in water samples [11-12]. Also it is the combined application of Gas chromatography and Mass spectroscopy. The Gas chromatography separates different components in sample on the basis of their retention times resulting in peaks in gas chromatogram. The GC spectra of sample-1 and sample-2 are shown in Fig. 3 and Fig. 4 respectively.

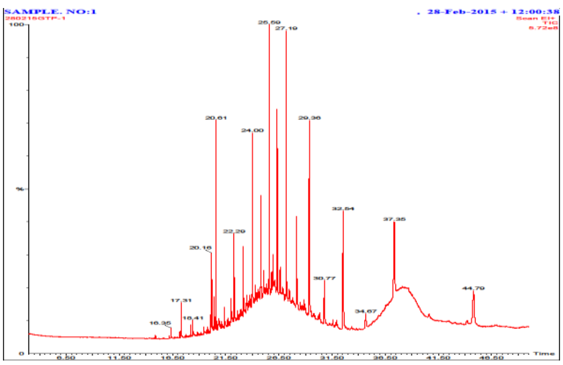


Fig. 3 GC of dichloromethane extracted wastewater (sample-1)

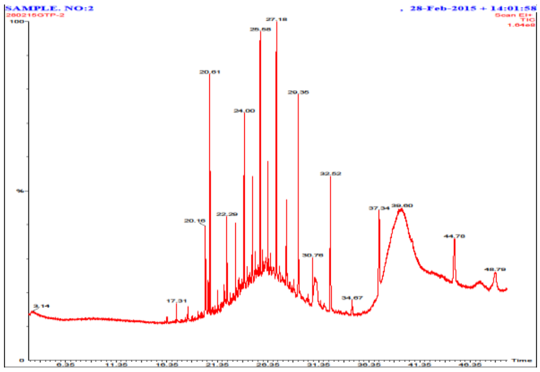
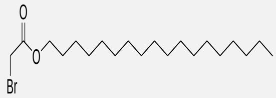
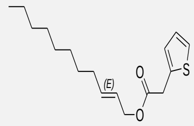
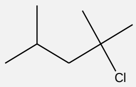
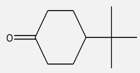
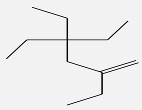
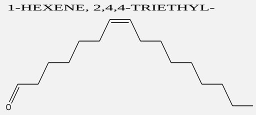
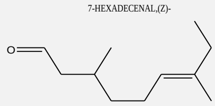
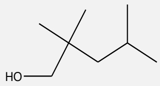
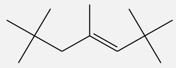


Fig. 4 GC of dichloromethane extracted wastewater (sample-2)

The separated components are then subjected to mass spectroscopy. In our sample five peaks were analyzed with mass spectroscopy. After interpretation of GC-MS spectra we can find the masses of organic compounds present in waste water samples. The MS of five peaks are shown in Fig. 5 and Fig. 6 and the probable compounds present in sample with their structure are shown in Table 3. Due to these organic compounds detected, depletion of dissolved oxygen is a major problem due discharge of oxygen demanding organic pollutant in the surface water. This poses threat to higher forms of aquatic life, if the concentration of oxygen falls below a critical point. Also these organics can affect on human health.

Table 3 Organics detected in wastewater (sample-1& 2)

S. No.	M.W.	Molecular Formula	Name of the compound and Structure
1	390	$C_{20}H_{39}O_2Br$	 BROMOACETIC ACID,OCTADECYL ESTER
2	264	$C_{17}H_{26}O_2S$	 2-THIOPHENEACETIC ACID, UNDEC-2-ENYL ESTER
3	374	$C_{22}H_{43}O_2Cl$	 2-CHLOROBUTYRIC ACID, OCTADECYL ESTER
4	134	$C_7H_{15}Cl$	 2-CHLORO-2,4-DIMETHYLPENTANE
5	154	$C_{10}H_{18}O$	 CYCLOHEXANONE,4-(1,1-DIMETHYLETHYL)-
6	168	$C_{12}H_{24}$	 1-HEXENE, 2,4,4-TRIETHYL-
7	238	$C_{16}H_{30}O$	 7-HEXADECENAL,(Z)-
8	168	$C_{11}H_{20}O$	 6-NONENAL,3,7-DIMETHYL-
9	130	$C_8H_{18}O$	 1-PENTANOL, 2,2,4,-TRIMETHYL-
10	168	$C_{12}H_{24}$	 3-HEPTENE, 2,2,4,6,6-PENTAMETHYL-

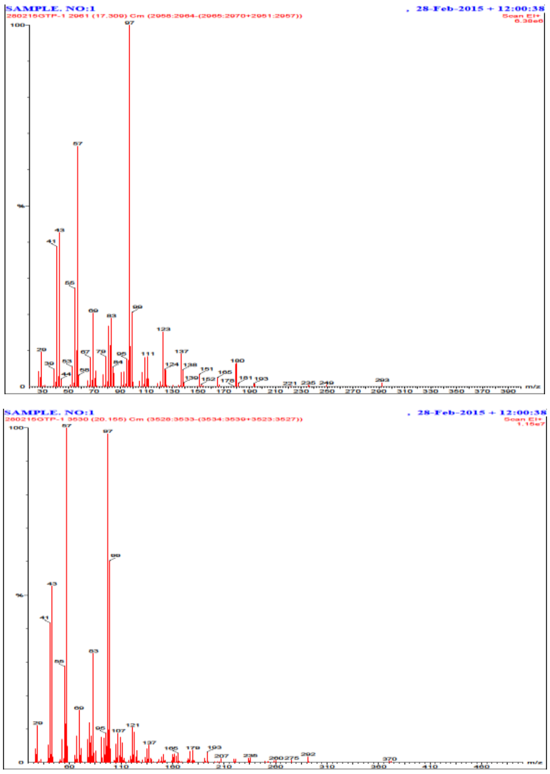


Fig. 5 Mass spectra of sample 1

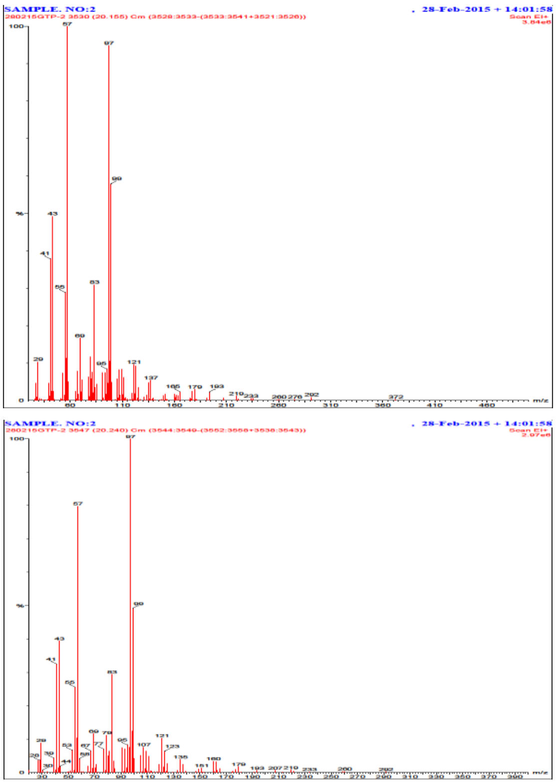


Fig. 6 Mass spectra of sample 2

3.2 Detection of Metals

The concentrations of heavy metals in wastewater are being tabulated in Table 4. The concentrations of some metals like Cd, Pb, As and Hg was found to be not detected in all wastewater samples. The concentration of Cu was in the range of 0.09 to 0.15 ppm. The concentration of Zn was found to be detected in the ranges 0.09-0.92 ppm respectively. In the case of Cr and Fe, the concentrations obtained in the range of 0.46-0.52 ppm and 1.33-6.13 ppm respectively. The values of some metals exit the acceptable limit. These metals if present above permissible limit then it may harm to both plant as well as human life.

Table 4 Detection of metals by ICP-AES

S. No.	Concentration of Metals (ppm)							
	Cu	Zn	Fe	Cr	Cd	Pb	As	Hg
1	0.14	0.77	5.24	0.46	ND	ND	ND	ND
2	0.12	0.89	6	0.52	ND	ND	ND	ND
3	0.15	0.92	6.13	0.47	ND	ND	ND	ND
4	0.09	0.52	1.33	ND	ND	ND	ND	ND
5	0.09	0.09	1.66	ND	ND	ND	ND	ND

ND – Not in Detectable limit

The GC-MS study of sample confirms the presence of organics in it. From this GC-MS data we can interpret possible organic compounds present in the sample. Also the ICP-AES gives the concentrations of heavy metals in wastewater. Finally these results are useful to what extent the water is polluted [13-14]. These organic matter released in water, results depletion of oxygen. [15].

4. Conclusion

The result achieved in the present investigation using GC-MS study of sample confirms the presence of organics and ICP-AES gives the concentrations of heavy metals in wastewater. Also the heavy metals enter in the human body by different pathways and causes harmful effects. Some of the metals and organic compounds detected are toxic to the environment. Day by day as the discharge of wastewater continues from these industries, due to non-biodegradability of heavy metals their concentration in environment continuously increases. It affects the quality of soil and ground water of that area. This analysis can highlight the urgent need for continuous monitoring which will affect on environment.

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