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Detection and Identification of solvent extracted organics by FTIR and GC-MS

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ABSTRACT

The present paper deals with the detection of organics in wastewater from dye industry in Sachin and Pandesara, GIDC, Surat, India. The eight different samples were collected from dye industry in Sachin and pandesara, GIDC, Surat, India. Sample was extracted with dichloromethane (CH_2Cl_2) and organic layer was sent for FTIR and GC-MS. The probable organics present in wastewater were determined by FTIR and GC-MS. Along with detection of organics some physicochemical parameters like pH, TDS, conductivity, sulphates, chlorides, COD etc were also studied, respectively.

Keywords: Dye wastewater, FTIR, GC-MS, COD, solvent extraction.

INTRODUCTION

Dyeing industry is a major source of pollution in India today. The organics are present in high concentration in the effluents of textile industry which are mainly responsible for color of dyes. Dyes (mainly azo dyes) are extensively used in different industries like different in, textile industry, food industry, printing and pharmaceuticals industry etc. These industries continuously release untreated effluents into rivers and other common sources of water. Azo dyes and their pigments are most commonly used synthetic colourants. Wastes having dyes can be of many different structural varieties like acidic, basic, disperse, azo, anthraquinone based and metal complex dyes among others. The textile industry is the largest consumer of dye stuffs [1]. A dye is natural or synthetic compound which is used for coloring products.

Water pollution is one of the most serious environmental problems [2]. Water pollution has many cause and characteristics; one cause of water pollution is discharge of variety of pollutants in by industries including water toxins, oils, heavy metals etc. 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]. The wastewater from the industries is main source which pollutes other important sources of water. The quality of water can be assessed by studying some parameters like TDS (Total Dissolved Salts), COD (Chemical Oxygen Demand), pH, temperature, electrical conductivity etc [5]. Synthetic dye industry is largely responsible for pollution [6].

MATERIALS AND METHODS

A sample was extracted with dichloromethane and organics extracted in organic layer were concentrated and sent for FTIR and GC-MS studies to SICART, Vallabh Vidya Nagar, Gujrat. The major five peaks were recorded by GC-MS.

The chemicals used were purchased from LOBA chemicals. The solutions were prepared by using double distilled water. Eight different samples of wastewater from dye industry were collected from Sachin and Pandesara, GIDC Surat, India. The pH of sample was measured by using Equiptronics digital pH meter (Model-E610) fitted with glass electrode. The electrical conductivity and TDS were measured by using a conductivity-TDS meter (Systronics conductivity-TDS meter 308). The physicochemical parameters were determined as per standard methods APHA [7] and Trivedy and Goel [8]. COD was determined by using potassium dichromate reflux method. Sulphates were analyzed by Gravimetric method and chlorides by $AgNO_3$ titration method [9].

RESULTS AND DISCUSSION

Detection of organics by FTIR and GC-MS: A sample was extracted with dichloromethane [10]. The organic layer was separated and was sent for FTIR and GC-MS to SICART Vallabh Vidyanagar, Anand. FTIR technique is useful for detection of functional groups. The spectra of FTIR are shown in fig-1 and functional groups detected by FTIR are tabulated in table1. The technique GC-MS is combined application of Gas chromatography and Mass spectroscopy. The Gas chromatography seperates different components in sample on the basis of their retention times resulting in peaks in gas chromatogram. The GC of dichloromethane extracted wastewater sample is shown in fig 2.

Table1. IR frequencies and detected functional groups from FTIR spectra

IR Frequencies	Bond and Functional groups obtained
772.04 cm^{-1}	Aromatic C-H
1218.94 cm^{-1}	C-O-C
1634.06 cm^{-1}	Aryl conjugated C=C
3436.39 cm^{-1}	-OH group
591.37 cm^{-1}	C-Cl (Alkyl) or C-Br (Alkyl)

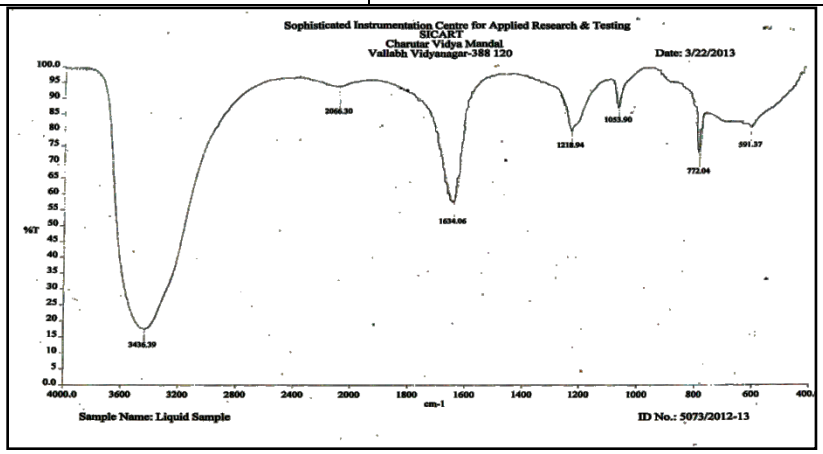


Fig-1 FTIR spectra of dichloromethane extracted wastewater sample

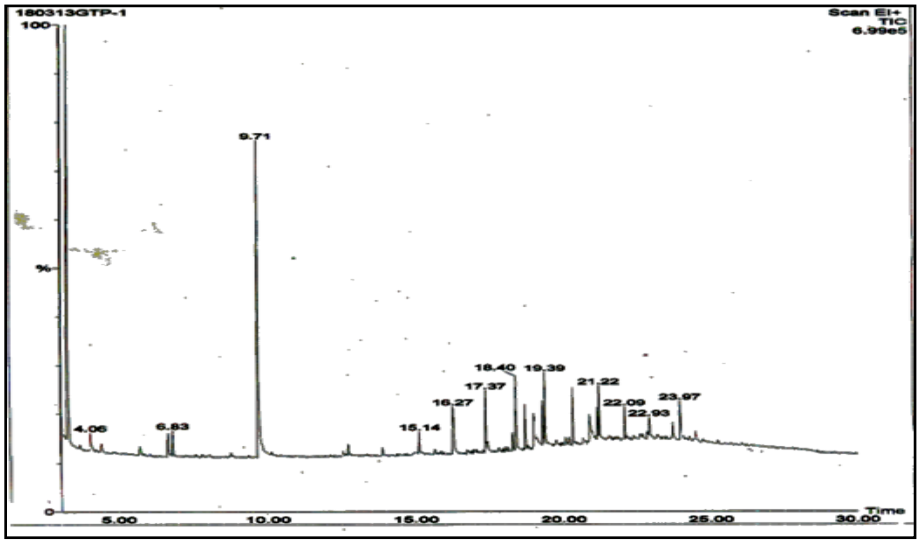
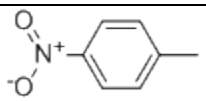
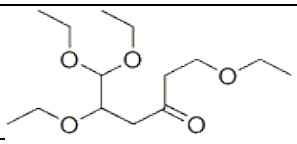
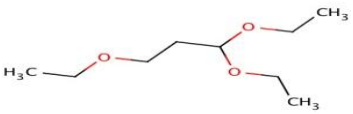
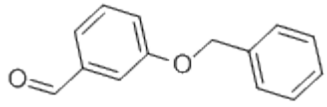
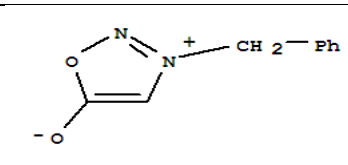


Fig-2 GC of dichlomeathane extracted wastewater sample

The seperated components are then subjected to Mass spectroscopy. In our sample five peaks were analyzed with mass spectroscopy. In this way with the help of combined application of GC and MS we can find the masses of organic compounds present in that sample after interpretation of specta. The MS of five peaks are shown in fig- 3-7 and the the **probable** compounds present in sample with their structure are shown in table-3.

Table-3 Detected organic compounds

Sr. No.	Molecular Weight	Molecular Formula	Name of the compound	Structure
1	**137**	**$C_7H_7O_2N$**	**1-Methyl-4-Nitrobenzene**	
2	**276**	**$C_{14}H_{28}O_2$**	**1,5,6,6-Tetraethoxy-3-hexanone**	
3	**176**	**$C_9H_{20}O_3$**	**3-Ethoxypropionaldehyde diethyl acetal**	
4	**212**	**$C_{14}H_{12}O_2$**	**3-Benzyloxybenzaldehyde**	
5	**176**	**$C_9H_{10}O_2N_2$**	**1,2,3-Oxadiazolium,5-hydroxy-3-(phenylmethyl)**	

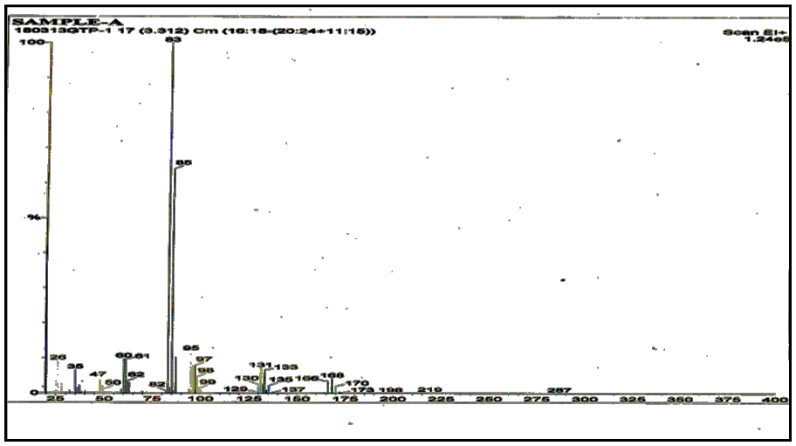


Fig-3 Mass spectra of peak 1

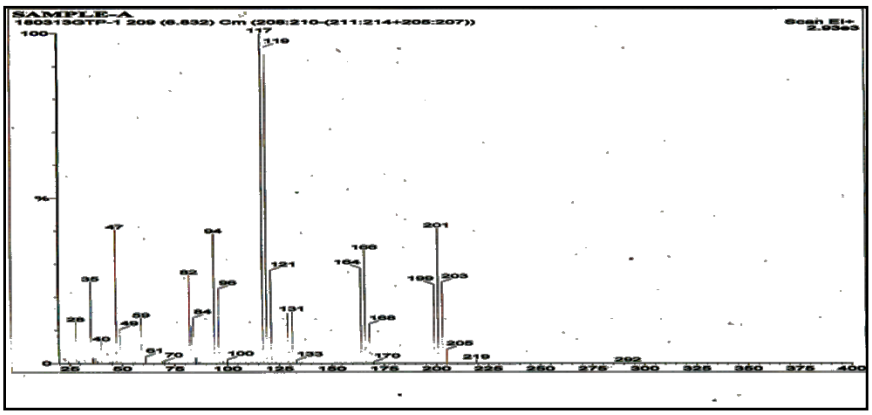


Fig-4 Mass spectra of peak 2

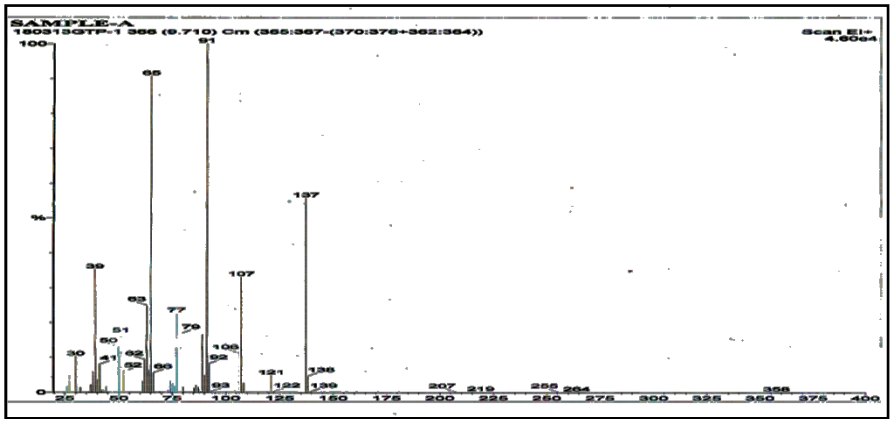


Fig-5 Mass spectra of peak 3

Physicochemical analysis of water sample: pH for the collected samples ranges in between 5.40 to 9.21. pH is measure of acidity and alkalinity, either acidic or alkaline water is harmful so pH of good quality water should be in the vicinity of 7.3 the result of pH measured shows that wastewater sources wastewater sources from which the samples have been collected can result in adverse change in pH of water bodies making it undesirable for human consumption. The high values of electrical conductivities suggest presence of metals in sample [8].

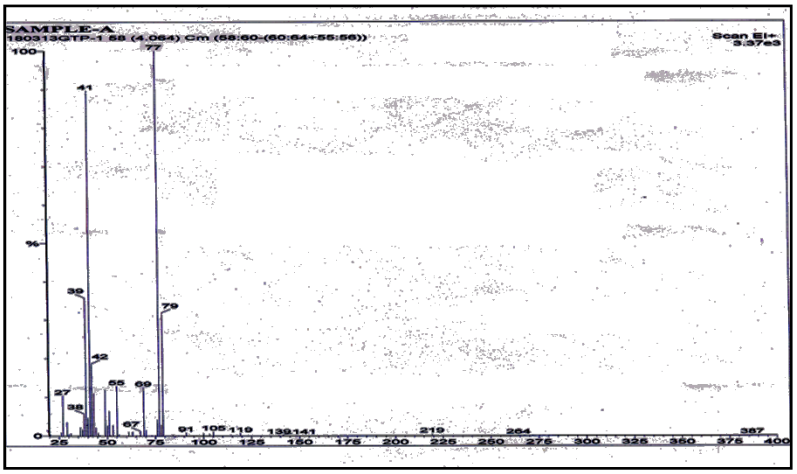


Fig-6 Mass spectra of peak 4

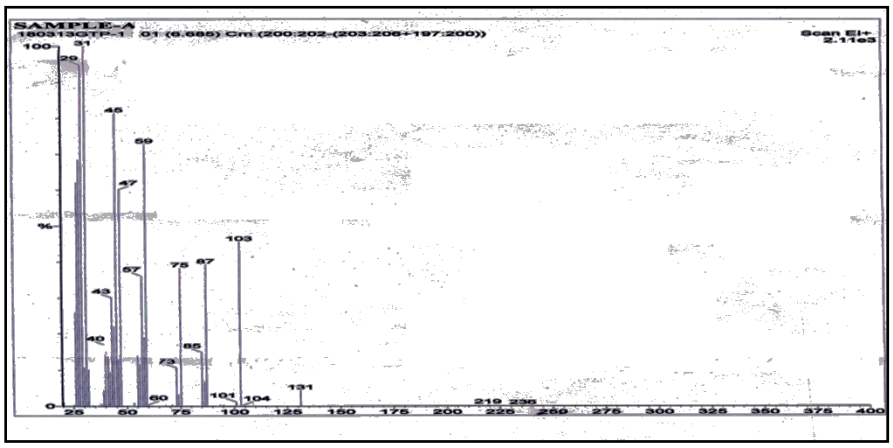


Fig-7 Mass spectra of peak

The sulphate content also exceeds permitted level. The values of chloride content are high which is harmful for agriculture crops, high chloride content imparts a salty test to water [9]. Total dissolved salts for collected samples ranges between 1189 to1897 which is very much higher than permissible limits.The results obtained from physicochemical study of water sample are given in **table-3.**

Table-3 Physico chemical characteristics of dye waste water
All the values in above Table are in mg L^{-1} except ph and electrical conductivity (µmho)

No. of Sample	pH	Electrical conductivity EC	Cl^-	SO_4^-	COD	TDS
Sample 1	9.21	.860	321	98	1780	1236
Sample 2	6.80	.539	129	86	1465	1189
Sample 3	5.75	1.230	231	136	1398	1800
Sample 4	6.25	2.050	426	201	1722	1600

Sample 5	5.40	.678	720	125	1644	1849
Sample 6	7.21	.940	563	139	1760	1358
Sample 7	6.93	1.980	298	90	1860	1769
Sample 8	8.50	1.132	468	67	1620	1897

APPLICATIONS

The study of a sample by GC-MS confirms the presence of organics in it, and also we can interpret possible organic compounds present in sample. The results of study of physicochemical parameters are useful to what extent the water is polluted.

CONCLUSIONS

The study of a sample by GC-MS confirms the presence of organics in it, and also we can interpret possible organic compounds present in sample. The results of study of physicochemical parameters shows that to what extent the water is polluted and how it can be harmful for other sources of water when it comes in contact with them. The values of physicochemical parameters are exceeding than those permeated by WHO, it suggest the extent of pollution.

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