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Detection and Identification of Metals, Nonmetals and Organic Compounds from Industrial Wastewater by Analytical Methods

K.M. Joshi[1,*], V.S. Shrivastava[2]

[1]*Department of Chemistry, Gangamai College of Engg. Nagaon, NMU, Jalgoan – 424 002, Maharastra, India.*
[2]*Department of PG Studies and Research in Chemistry, G.T.P. College, Nandurbar – 425 412, Maharastra, India.*

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The present study was carried out to detection and identification of metals and organic compounds from the effluents of printing and dyeing wastewater of Yeola by using ICP-AES, FTIR, GC-MS, Flame Photometer and physico chemical characteristics. For detection of metals HCl-HNO_3 extract was prepared and impact of organic compounds were detected and identified by using CH_2Cl_2 extracted masses. For removal of the dye from industrial wastewater semiconducting nanomaterials were used.

1. Introduction

In Maharashtra rich and exquisite traditional handloom textiles and handicrafts. Hand-woven textiles like Paithani brocades that have existed for more than 2000 years in original patterns and woven with the same techniques even today. Paithani is a variety of sari, named after the Paithan region in Maharashtra state where they are woven by hand. Made from very fine silk, it is considered as one of the richest saris in Maharashtra.

Water pollution is one of the most serious environmental problems. In the world, industry is one of the biggest sources of pollution, accounting for more than half the volume of all water contamination and for the most deadly pollutants [1]. In the recent years interest has been focused on the use of semiconductor materials asTiO_2 and ZnO. Photocatalyst for removal of organic and inorganic species in the water which is can be a very effective wastewater treatment technology [2]. Porous photocatalytic materials have the combined qualities of high surface area and relatively large particle sizes, as compared with nanoparticulate catalyst powders [3,4]. Most of the industrials wastewater are containing organic inorganic matter and hazardous metals [5].

The heavy metals and organic compounds affects the quality of water. Heavy metals enter in the human body by different pathways and causes harmful effects [6] .The waste generated by the textile dying and printing industry contain a variety of organic compound and toxic heavy metals. They exhibits high Chemical Oxygen Demand (COD) and Biological Oxygen Demand (BOD) [7]. The main objective of the present investigation was to assess the suitability of the wastewater and dyes from Yeola textile industry which is one of the largest handloom manufacturing zone. The coloured effluent of the textile mill industry have got much attention to their dual toxicity these dyes were removed by using nano materials likeTiO_2, ZnO and CdS as a photocatalysts.

2. Experimental Methods

The samples were collected in screw capped polyethylene which was cleaned and washed with deionized water and rinsed with sample two to three times and tightly lid [8]. These samples were solvent extracted [9] with CH_2Cl_2. The obtained mass analysed for functional group by FTIR and GC-MS. Remaining water samples were used for physicochemical analysis as volumetric analysis, BOD, COD and flame photometric analysis where Na^+, Ca^+, K^+, Li^+, was determined by standard methods. For the removal of dye nano sized TiO_2 and ZnO were used as adsorbent.

2.1 Photocatalytic Degradation

The photocatalytic degradation for both dyeing and printing wastewater was determined by mean of UV-Visible spectrophotometer (Systronic -2203). The adsorbent were used as TiO_2 and ZnO. The dye solution was separated from the adsorbent by centrifuging .The removal was observed by spectrophotometrically by monitoring absorbance changes at different wave length. The reaction mixture was irradiated with light source UV lamp (PHILIPS-400Watt) at a distance 30 cm from the reaction vessel. A double distilled water was used throughout the experiment. pH of the dye solution was adjusted upon addition of requisite acid and base.

2.2 Physico-Chemical Analysis

The water samples have been analyzed as per standard procedure-APHA [9]. Chloride is estimated with the argentomeric titration, Mohars method while the other anions, sulphate, nitrate, phosphate and fluoride are determined volumetric methods. The water samples were used for physico chemical meters like pH, Electrical Conductivity (EC), Alkalinity, carbon dioxide(CO_2), dissolved oxygen(DO), Chemical oxygen demand (COD), sulphate $(SO_4)^{2-}$, Chlorides (Cl^-) Hardness,(Ca^{2+}, Mg^{2+}) as per standard procedure. With volumetric analysis BOD, COD, calcium and magnesium are determined with the EDTA titration method. The metals like sodium, calcium, potassium and lithium are measured with the help of flame photometer (Systronic-130) measured with using standard addition method with standard sample of NaCl, KCl and $LiCl_2$ with 10 to 100 ppm solution was standardizes. All other metals estimated by atomic emission spectrophotometer and inductive coupled plasma. All chemicals were AR grade and double distilled water was used for preparation of reagents. The dyes in the water sample was removed by using TiO_2 and ZnO semiconductors with specific concentration.

*Corresponding Author
Email Address: km_joshi53@rediffmail.com (K.M. Joshi)

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2.3 FTIR and GC-MS Studies

The samples were extracted with CH_2Cl_2. FTIR spectra of native and exhausted adsorbents were recorded with the help of Perkin-Elmer spectrum-Gx-I FTIR system. The spectra were recorded between 400 and 4000 cm^{-1}. For GC-MS Perkin-Elmer model Autosystem XL-GC. The obtained mass analysed for functional group by FTIR and GC-MS. The sample were digested in HNO_3-HCl by following the literature method [10]. The metal by ICP-AES, FTIR and GC-MS studies were carried out at SAIF IIT Mumbai, and SICART Vallabh Vidyanagar(Guj.).

3. Results and Discussion

Table 1 Detection of metals by ICP-AES

S. No.	Concentration of Metals (ppm)									
	Cu	Zn	Pb	Cd	Ni	Co	As	Hg	Cr	Fe
1	0.44	2.0	2.96	0.072	0.36	0.82	ND	0.02	0.87	14.26
2	0.23	1.77	1.97	0.014	0.48	0.92	ND	ND	0.82	21.12
3	0.39	0.96	2.83	0.017	0.33	1.0	0.01	0.01	0.21	8.46
4	0.33	2.12	1.92	0.48	0.56	0.82	0.11	ND	0.21	9.59
5	0.38	1.12	0.97	0.56	0.72	0.66	0.13	ND	0.33	16.23
6	0.024	0.16	0.56	ND	ND	ND	ND	ND	1.36	2.97
7	0.33	0.86	0.12	ND	ND	ND	ND	ND	1.91	2.86
8	0.017	0.97	0.012	ND	ND	ND	ND	ND	1.02	3.49
9	0.013	0.14	0.14	ND	ND	ND	ND	ND	0.98	2.68
10	0.024	0.23	0.68	ND	ND	ND	ND	ND	0.87	1.88
11	0.34	3.82	ND	ND	ND	ND	ND	ND	ND	0.92

ND – Not in Detectable limit

Table 2 Detection of exchangeable metals by Flame photometer

S. No.	Exchangeable metals (ppm)			
	Na	K	Ca	Li
1	2.48	0.98	11.62	ND
2	1.26	1.12	44.26	ND
3	0.65	0.78	46.21	ND
4	1.48	1.67	36.56	ND
5	0.78	2.49	38.9	ND
6	0.67	2.04	78.14	ND
7	1.49	0.16	34.2	ND
8	2.89	2.08	26.83	ND
9	0.26	1.56	75.3	ND
10	0.54	1.89	16.8	ND
11	0.47	1.54	67.4	ND

ND – Not in Detectable limit

Table 3 Physico-chemical analysis of Textile Dyeing and printing wastewater, drinking and Ground water samples

ID	COD (ppm)	Hardness (ppm)	Ca (ppm)	Mg (ppm)	Cl^- (ppm)	CO_2 (ppm)	pH	EC (10^{-3}) μS/ cm	CO_3^- (ppm)	HCO_3^- (ppm)
S 1	546	1361	1162	0199	1952	56.4	7.62	8.52	1460	532
S 2	1240	2560	443.6	2116	2861	110.5	8.2	2.89	398	322
S 3	482	4200	462.1	3738	1214	121.5	7.56	2.41	560	428
S 4	514	1389	365.6	1023	3252	102.3	8.35	3.06	763	765
S 5	640	2360	389	1971	1152	89.5	9.3	3.24	520	520
S 6	638	3510	781.4	2729	2451	101.2	9.42	3.08	800	840
S 7	1028	3220	342	2878	1126	113.5	8.26	3.11	1060	715
S 8	705	3560	268.3	3292	1282	101.2	8.52	3.17	270	912
S 9	327	830	753	0077	1206	112.5	8.69	3.78	642	1162
S 10	412	1240	168	1072	834	121.5	9.31	5.64	1180	830
D 1	Nil	132	56.5	0076	96.5	105.4	7.26	8.51	78	108
G 1	3240	170	181.5	0089	782	Nil	7.96	5.78	182	342
G 2	2342	169	169	0180	570	Nil	8.12	2.74	189	489
G 3	1768	195	195	0043	624	Nil	9.03	4.86	438	520
G 4	2415	232	187	0045	792	Nil	7.41	2.38	247	367

The result obtained during the course of present study are given in Table 1-3. The pH value range from 7.41 to 9.42 showing that the water is slightly alkaline (Table 3). These values are out of the maximum permissible limit set by WHO, 9.2 which is safe range for drinking. The concentration of heavy metals in wastewater are being tabulated in Table 1. The concentration of some metals like Hg, As, Co, Ni and Cd was found to be not detected in all wastewater samples. The concentration of Cu was in the range of 0.013 to 0.44 μg/mL.

The concentration of Zn, Pb and Cd was found to be detected in the ranges 0.14-3.82, 0.012-2.96, 0.014-0.56 μg/mL respectively. The concentration of Ni and Fe was 0.33-0.72 and 0.92-14.26 μg/mL. The values exit the tolerance limit. The concentration of sodium in the range of 0.78-1.45 micro g/mL. The increase in concentration of Na^+ in the water causes effects on the cardio-vascular diesese and toxima. The concentration of sodium was found to be in the range of 0.26-2.89 μg/mL. The higher concentration of sodium in the water affects the soil permeability and texture and leads to pudding and reduce the rate of water intake [11]. The concentration of potassium in the water sample was 0.16-2.49 μg/mL. High concentration of potassium may causes magnesium deficiency and iron chlorosis (Table 2).

The concentration of calcium was found in the range of 11.62-46.21 μg/mL. The values exit the tolerance limit. A high concentration of calcium makes water hard the soil become soft and allows penetration of water. The concentration of magnesium was found in the range of 7.7-32.91 μg/mL. The high concentration of magnesium have similar effect on soil and water as calcium. According to ISI maximum acceptable and permissible concentration of magnesium was 2.5 mg/L. The magnesium deficiency causes breaking down of chlorophyll molecules with botches on the leaves. The concentration of iron was found in the range 0.92-21.46 μg/mL. These heavy metals from the complex with organic acids, ester and some heterocyclic compounds.

The FTIR spectra of CH_2Cl_2 extracted mass are identified organic compounds. These characteristics frequencies are useful for detection and identification of organic compounds [12].

The FTIR measurements of waste water showed the presence of peaks for large number of functional groups (Fig. 1) viz. 3406.21cm^{-1} –OH stretching, 2925.45 and 2855.48 cm^{-1} C–H stretching vibration, 950.45 cm^{-1} C–H bending vibration, 1262.32 cm^{-1} – C:N amine, 1726.32 cm^{-1}– C=O for carboxylic acid, 1594 cm^{-1} – C=C– aromatic stretching frequency, 1262.32 cm^{-1} C–N stretching, 1116.28 cm^{-1} C–O stretching in primary alcohols. It also contains large number of polar groups on the surface, which is likely to give considerable dye adsorption capacity. The GC-MS spectra of CH_2Cl_2 extracted mass are given in Fig. 2. Identified these characteristics are useful for detection and identification of organic compounds [13].

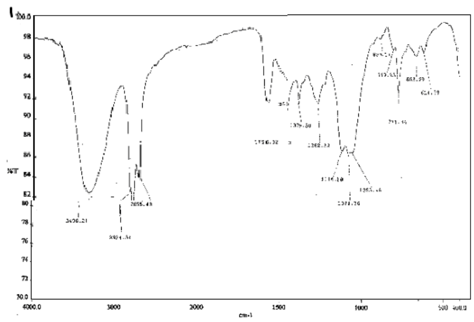

Fig. 2 FTIR Spectrum of sample 1.

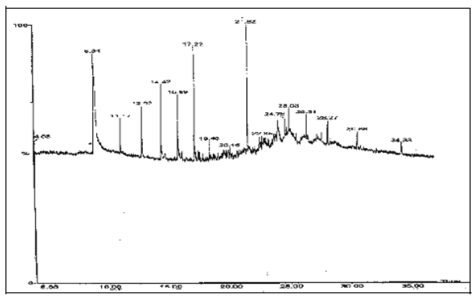

Fig. 2 GC-MS Spectra of CH2Cl2, extracted mass of printing and dyeing wastewater sample 1.

The saturated and unsaturated hydrocarbons, aliphatic acid, aromatic compounds carboxylic acid, ester and some heterocyclic compounds found in both sample. Ester can cause adverse effect on an endocrine system which is comprised the organ and glands, hat secret hambones compounds that are toxic to the endocrine system may causes diseases diabetes mellitus, hypoglycemia, reproductive disorders and cancer [14]. Phenolic compounds impart typical odour and taste to the water and is highly toxic to aquatic life and vegetation even low concentration. When animal and human being use phenol polluted water. They frequently affected by diseases of the alimentary track. It provides toxic effects on brain, lings, kidney liver etc. A limit of 10 mg/L of polyhydric phenol has been suggested to avoid inhibition of biological process. A benzene compounds e.g. 1,2-benzene dicarboxylic acid causes skin irritation, reediness and pain. Excess inhalation may lead to headache and weakness.

4. Conclusion

The result achieved in the present investigation on the quality of the water from the Yeola town shows that all the effluents of the wastewater and printing dye waste physicochemical parameters fall within the permissible limit as per standards. The organic compound affects the quality of soil and ground water of the area. Out of these detected organic compounds some are carcinogenic. The present study with ICP-AES shows that the wastewater consist of different heavy metals. The concentration of heavy metals does not exceed the permissible limit. The organic insoluble salt which makes a water hard and unsuitable for living organisms. Photo catalysis has a large capability for water treatment. It can be utilized for the decomposition of organic and inorganic compounds and removal of trace metals. Therefore the dyeing and printing wastewater should be treated before ponding to open place or river.

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