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Detection and Identification of organics by FTIR and GC-MS Techniques

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Abstract
Today's industrial and sewage wastewater pose a serious environmental problem and pollute water bodies and soil very badly which affects the flora and fauna of Ecosystem. It is basic need to know the concentration of organic and toxic heavy metals moieties in wastewater before it leaving into the big soil and water bodies. The wastewater generated industrial areas is complex in nature because of the variability in the process system producing organic and inorganic moieties in the wastewater. These industries create water pollution by discharging the wastewater containing large number of organics and other color causing compounds. Hence is needful and area of interest is detection and identification of wastewater components.
This investigation focuses on the detection of organic moieties present in the soil and industrial or sewage wastewater by well-developed technique FTIR and GC-MS which can detect the organic concentration up to 0.01 mg present in the wastewater. The same sample were characterized by FTIR which shows the different IR frequencies of organic. The present extraction technique was employed for the extraction of organic by gas chromatography and mass spectroscopy GC-MS technique.

Keywords: flora and fauna, industrial waste, organic moieties, extraction, GC-MS, FTIR

1. Introduction
The wastewater generated from multi-fold industrial estates is complex in nature because of the variability in the process system producing organic and inorganic load in the wastewater. The scarcity of water resources around the world increases the demands on the use of secondary sources, such as wastewater. In this perspective water recycling and re-use of treated effluent in high water consuming industrial sectors seem be to be a viable alternative to save valuable resources [1, 2].Various kinds of industries are set up in the selected industrial area. Different kind of organic compounds and metallic moieties are present in the wastes of these industries. Some organic compounds remain persistent into the environment and causes disturbance in the ecological cycle. As far as pollution from industries in concern effluent in most of cases are discharged into rivers, streams, pits, open ground or open unallied drains near the factories, thus allowing it to move to low lying depression resulting in severe ground water pollution.
The magnitude of damage caused to our water resources can be estimated from the fact that about 70% of the rivers and streams in India contain polluted water [3]. In the last years, one of the major concerns to water quality is related to the detection of chemical pollutants in both industrial and municipal wastewater. Most of these contaminants, both synthetic organic chemicals and naturally occurring substances, enter the aquatic medium in several different ways and, according to their water solubility, can be transported and distributed in the water cycle [4]. The infrared region (4000-650 cm^{-1}) is of great importance in studying an organic compound. Since infra-red spectra contain a large number of bands, no two compounds will have the same infra-red spectrum (expect optical isomers). In contrast to ultra violet spectra, the infra-red spectra of most organic compounds contain large number of bands. Even very simple molecules can yield an extremely complex spectrum. Infra-red spectroscopy has widely been used for the identification complex and provides numerous maxima and minima that can be used for comparison purpose. In fact, the infra-red absorption spectrum of an organic compound represents one of its truly physical properties. Infra-red spectra are usually plotted as percent transmittance is 0% if all the radiation is absorbed and the transmittance is 100% for no absorption.
For this study, the sample is prepared by extracting industrial wastewater with dichloromethane (CH_2CL_2).In present research the FTIR (Fourier Transform Infrared) spectroscopic analysis on Nicolet Magna-IR Spectrometer-550 at SAIF, IIT, and Mumbai. Absorption bands and characterization are being tabulated in table.1. However from the obtained FTIR results there is prediction of the presence of particular organic compound, but can only get the information about the functional groups, therefore it is need carried out GC/MS analysis for the exactness.

2. Experimental
2.1 Materials and Methods
Sample Collection
The waste water samples were collected in prewashed polyethylene bottles by deionized water was distilled before use. PH and conductivity of the samples were measured while collecting the samples. Each waste water sample was taken four times at four different sampling periods approximately three month apart. All solvents were of AR grade and used without distillation.

2.2 Sample Preparation
2.3 Solvent extraction Techniques
The pH adjusted below 1, 100 µL of 6 mol/L HCl was added to 5 mL of Wastewater containing 1.5 g of sodium chloride was also added. Organic in waste water samples were extracted into 10 mL of Dichoromethane by mechanically shaking for 10 min, in separating funnel and

the organic extracted layer was transferred to a second Becker and solvent was evaporated under reduced pressure on hot plate to concentrate the sample.

3. Results and Discussion

Nowadays, the study of the environmental aspects shows toxic heavy metals and organic moieties badly affects plants and animals. This research study discuss the analysis of sewage and industrial wastewater extracted with CH_2Cl_2 by FTIR and GC/MS. The obtained extracted mass has been analysis for different functional group by Parkin Elmer FTIR and organic compound by Haward Packed Make GC/MS instruments at Sophisticated Analytical Instrument Facility (SAIF), IIT Mumbai. The combination of a best separation technique (GC) with the best identification technique (MS) made GC-MS an ideal technique for qualitative analysis. The GC-MS analysis reveal that the detected pollutant were seriously pose for the ecosystem. The GC-MS spectra of dyeing and printing wastewater shows different hydrocarbons derivatives, Aliphatic and aromatic acids and Phtalate derivatives etc.

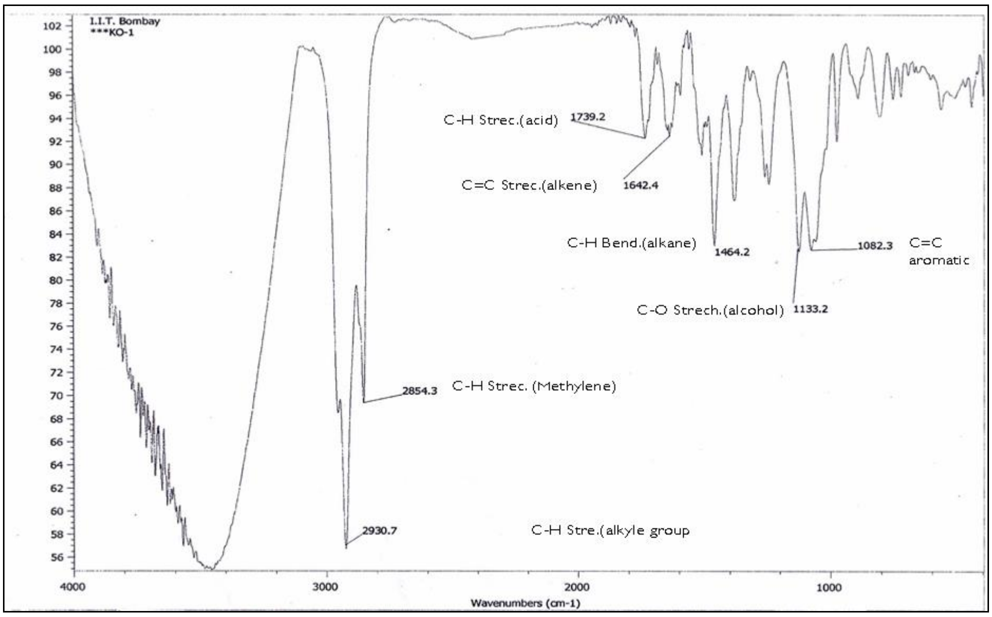


Fig 1: FTIR spectra of text file effluent sample ko-1(dyeing printing house mill pandesara)

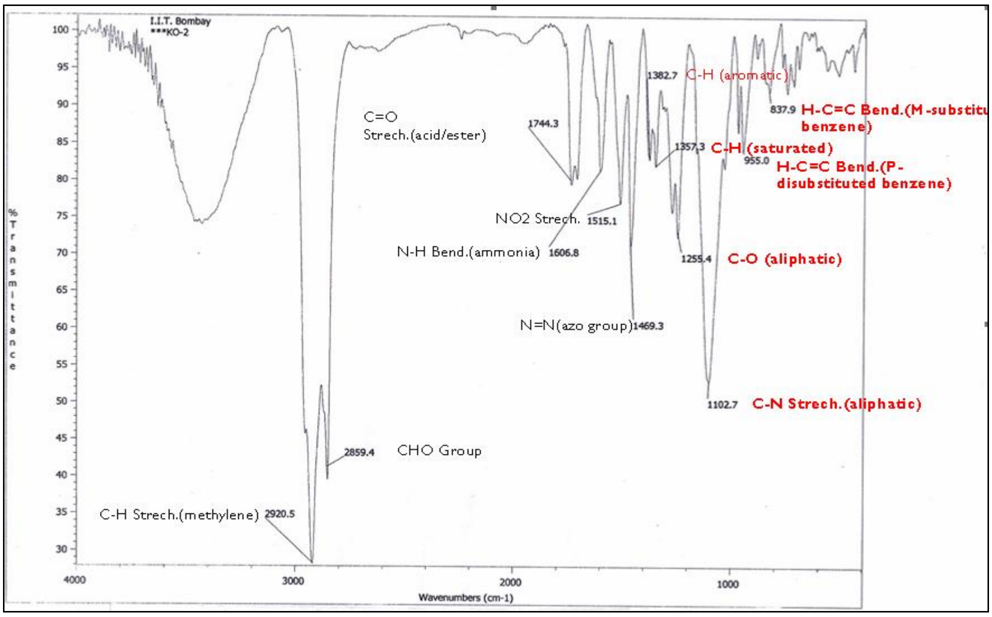


Fig 2: FTIR spectra of textile effluent sample KO-2(kirty dyeing and printing mill, Pandesara)

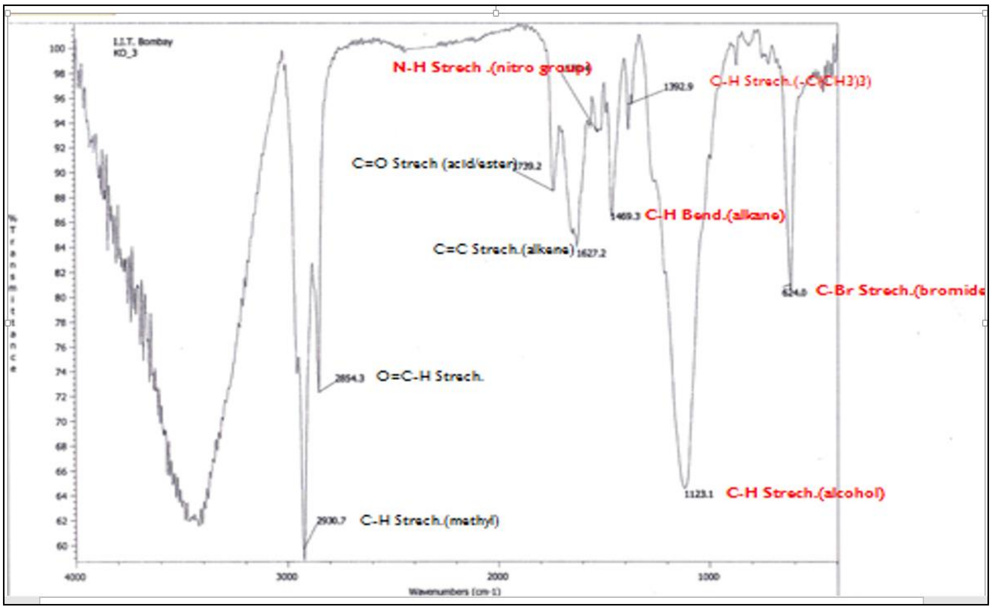


Fig 3: FTIR spectra of textile effluent sample KO-3(Tarana dycing and printing mill, pandesara)

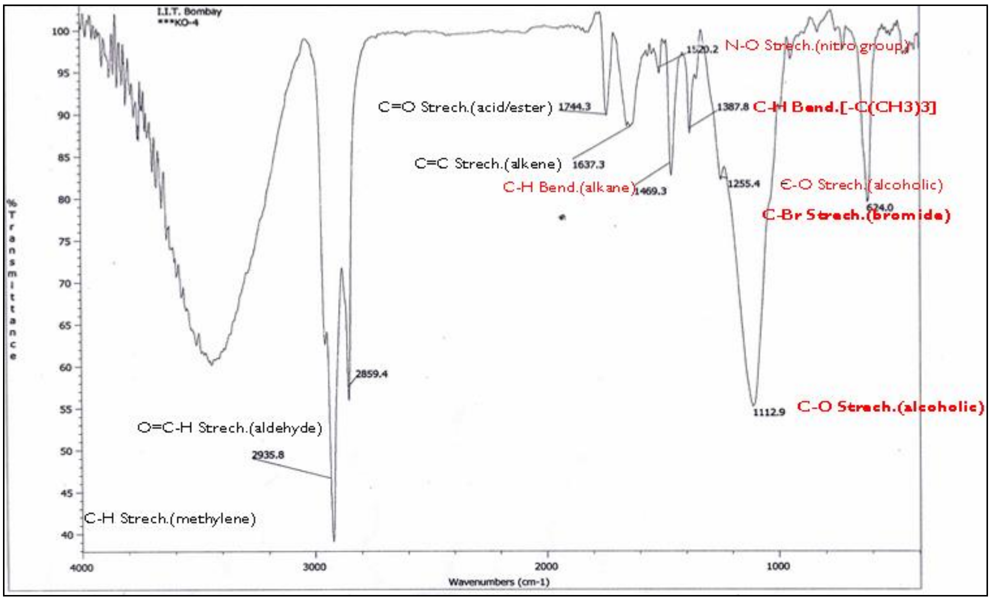


Fig 4: FTIR spectra of textile effluent sample KO-4(Gagan dycing and printinfg mill, Kadodara)

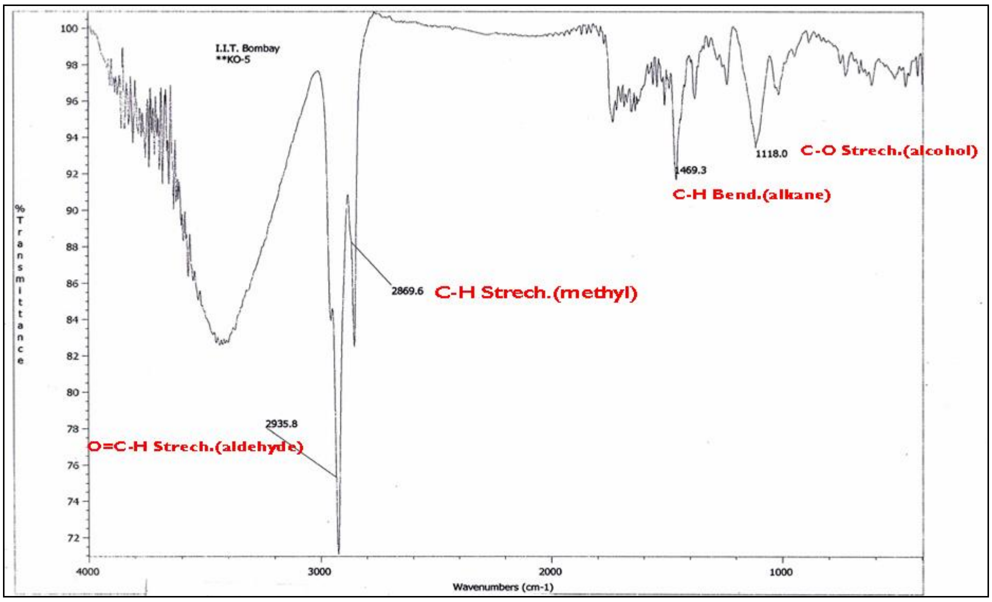


Fig 5: FTIR spectraof textile effulent sample KO-5(Best processor, Kadodara)

Identification of organic compound. Several authors find out the functional groups of various organic compound present in the wastewater and the other samples by FTIR [5-7]. Figure 1 to 5 show the functional group obtained of various organic compounds present in the wastewater and effluent samples.

Table 1: The IR result of the dyeing and printing effluent extracted samples show importance frequency and characteristics band.

Sample No.	frequency (cm^{-1})	Possibility of functional groups
KO-1	2930.7	C-H Streching (alkyl group)
	2854.3	C-H Streching (Methylene)
	1739.2	C-O Streching (acid)
	1642.4	C=C Streching (alkane C=C group)
	1464.2	C-H Bending (alkane)
	1133.2	C-O Streching
	1082.3	C-O Streching
KO-2	2920.5	C-H Streching (-CH_2 gr)
	2859.4	C-H Streching (Methylene gr)
	1744.3	C=O Streching (acid or ester)
	1606.8	N-H bending (-NH_3 group)
	1515.1	=-NO2 Streching (nitro gr)
	1469.3	-N=N- (azo gr)
	1382.7	C-H(aromatic)
	1255.4	C-O Streching(aliphatic C-O)
	955	H-C=C bending (p-disubtituted benzene)
	837.9	C-H bending,meta subtituted aromatic
KO-3	2930.7	C-H Streching (-CH_3 gr)
	2854.3	C-H Streching (=C-H gr)
	1739.2	C=O Streching (acid or ester)
	1627.2	C=C Streching (alkane C=C group)
	1530.4	N-O Streching (-NO_2 gr)
	1123.1	C-O Streching (alcohols)
	2859.4	C-H Streching (= C-H gr)
	1744.3	C=O Streching (acid or ester)
	1637.3	C=C Streching (alkane C=C group)
	1520.2	N-O Streching (-NO_2 gr)
	1387.8	C-H Bending (-$C(CH_3)_3$ gr
	1255.4	C-O Streching (alcohols C-O)
	1112.9	C-O Streching (alcohols C-O)
KO-5	2935.8	C-H Streching (-CH_3 gr)
	2869.6	C-H Streching (-CH_2 gr)
	1469.3	C-H Bending (alkane)
	1118	C-O Streching (alcohols)

Gas Chromatography / Mass Spectrometry (GC/MS) has been found to be very useful for the analysis. In the gas Chromatography, when the sample solution is introduced in to the column, the organic compounds are vaporized and move through the column by carrier gas. They travel through the column at different rates, depending on difference in partition coefficient between the mobiles and stationary phases. Some interference's in GC analyses occur as a result of sample, solvent, or carrier gas contamination, or because large amounts of a compound may be injected into the GC and linger in the detector chloroform and other halocarbon and hydrocarbon solvent are ubiquitous contaminants in environment laboratories [8]. Strenuous efforts should be made to isolate the analytical system from laboratory areas were these or other solvents are in use. An importance sample contaminants is sulfur, which is encountered generally only in base/neutral extracts of water, although anaerobic ground waters and certain wastewaters and sediment/sludge extract may contain reduced sulfur compound, elemental sulfur or polymeric sulfur.

In combined GC and MS, the chromatographic retention parameters provide isomers specificity. While the mass spectral parameter provide class and homologue Specificity. Besides the various technical development in GC and MS a great deal of the progress achieved can be attributed to improvements in sample preparation procedures. Several author find out organic compounds present in the wastewater and the oher samples by GC/MS [9-11]. The GC/MS analysis of textile dyeing and printing wastewater extract was carried out at sophisticated analytical instrument facility (SAIF),IIT Powai, Mumbai on Hewlett Packard GCD 1800Ainstrument having detector – electron ionization,coloumn-HP-5,length -30 m, internal diameter – 0.25mm,carrier gas –Helium and flow rate-1ml/min. The other conditions are given on the GC/MS trace. An entry such as 100-10-260-8M-NEAT-HP5-$CHCl_3$ means that the initial temperature was 100°C for 8 minutes and then heated at the rate of 10°C per minute to 260°C. The GC/MS of the textiles dyeing and printing effluent extracted samples and heir library search compound are given in fig.6. Adding a small amount of mercury or copper filing to precipitate the sulfur as metallic sulfide can eliminate this interference. T he obtained GC/MS results of

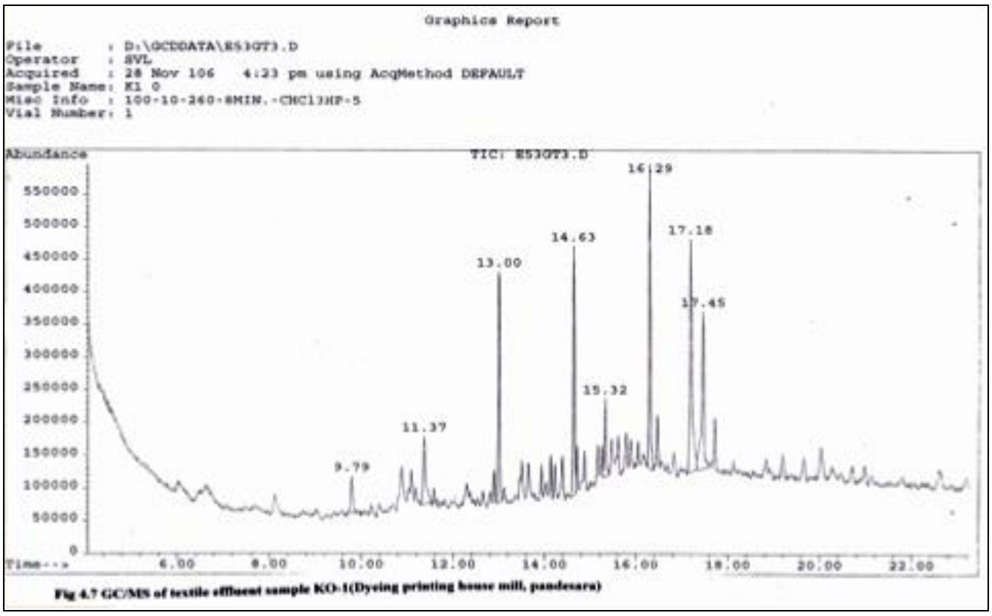
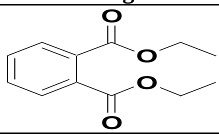
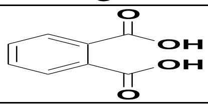
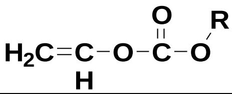
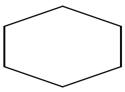
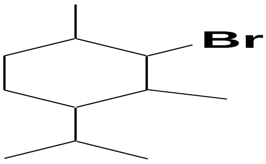
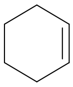
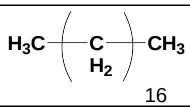
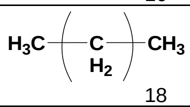
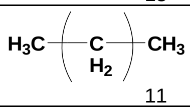
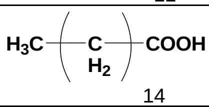
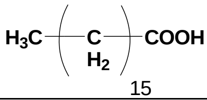
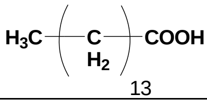
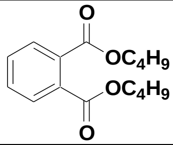
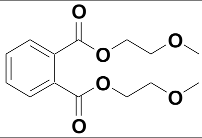


Fig 6: GC-MS of textile of effluent sample KO-1 (Dyeing and Printing house mill, Pandesara)

Representative dyeing and printing effluent samples show the presence of following organic compounds.
We are trying to explain the discussion of identified moieties individually, particularly with respect to name, structural formula, molecular formula and molecular weight. The moieties are gives one by one in table. 2.

Table 2

S. No.	Name of the organic compound	Structure of the organic compound	Molecular formula	Molecular weight
1	Diethyl Pthalate		$C_{12}H_{14}O_4$	222.24
2	Pthalic acid		$C_8H_6O_4$	166.13
3	Allyl ether ester		-	-
4	Cyclohexane		C_6H_{12}	84.16
5	1-bromo, 2-methyl menthane		$C_{11}H_{21}Br$	233.19
6	Cyclohexane		C_6H_{10}	82.14
7	Octadecane		$C_{18}H_{38}$	254.49
8	Eicosane		$C_{20}H_{42}$	282.55
9	Tridecane		$C_{13}H_{28}$	184.36
10	Hexadecanoic acid		$C_{16}H_{32}O_2$	256

11	Heptadecanoic acid		$C_{17}H_{34}0_2$	270
12	Pentadecanoic acid		$C_{15}H_{30}O_2$	242
13	Dibuthyl Pthalate		$C_{16}H_{22}O_4$	278.34
14	Bis(2-methoxyethyl) phthalate		$C_{14}H_{18}O_6$	282.29

4. Conclusion

The result obtained in this present research using FTIR and GC-MS study of sample confirms and shows the presence of organics in wastewater. Many forms of organic compounds in the found in wastewater which are toxic to humans and aquatic environment. In FTIR analysis it is found the presence of alcohol, phenols and amine groups may be responsible for the color of the wastewater due to the complex nature of the wastewater. The contamination of combined multi-complex compounds in wastewater becomes very toxic. To reduce the uncertainty in quantifying contaminant discharges to wastewater or effluent by identifying and surveying specific sources to determine the potential for controlling inputs particularly from small commercial sources and medical establishments. To establish the extent and variability of contaminant entry into Effluent by catchment investigations in relation to precipitation frequency and changes in sludge quality. To critically and independently review the fate, behaviour, degradability, toxicity and environmental consequences of alternative surfactant and plasticing compounds, in collaboration with the related chemical manufacturing industries, to inform decisions of the benefits and disadvantages of product substitution in detergent formulations and plastics manufacture. To determine the extent of volatilisation-deposition cycling of persistent organic pollutants in the environment, identifying the processes controlling the extent and magnitude of diffuse inputs of these substances to wastewater and to provide long-term predictions of changes in release patterns and the consequences for effluent and sludge. To develop a consistent statistical and reporting protocol for national chemical composition data presented in surveys of sewage wastewater sludge quality.

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