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# Hazardous odor markers from sewage wastewater: A step towards simultaneous assessment, dearomatization and removal

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ABSTRACT

Odor emissions from Sewage Treatment Plants (STP) may be hazardous and cause nuisance in the surrounding area. Odor is generated by the mixture of volatile organic compounds present in the sewage gas, leachate and leachate treatment systems, sewage sludge and waste materials.

This research aimed to develop a quantitative Odor Assessment and Control Scheme (OACS) by simultaneous use of human sensors and liquid phase adsorption. Assessment of odor intensity was accomplished by the standard Sniffer Panel method of Olfactometry. Psychophysical laws were applied and validated to evaluate odor concentrations in the semi-solid, liquid and gas phases. Parameters of these laws were estimated by a robust nonlinear least square technique. It was observed that Beidler's equation on intensity–concentration relationship for sewage could be suitable for samples carrying intense odor from trickling filter based STP. The temporal reduction in odor concentration correlated well with the reduction in the concentration of the most hazardous volatile organo-sulfur compounds (VOSCs) such as n-Propyl Mercaptan (1-Propanethiol), Ethyl-Methyl Sulphide (Methylthio Ethane) and 3,6 Dimethyl 2,4,5,7-tetrathiooctane (bis 1-[{methylthio}ethyl] disulphide).

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## 1. Introduction

Odor is a matter of serious concern since odor has been recognized as a kind of sensory pollution that gives an unpleasant and disgust feeling and it is often linked directly to the quality of life. The primary cause of ever increasing odor is increase in population, and hence the public nuisance increases with economic growth and rise in living standard. Sewage Treatment Plants (STPs) were the major sources of malodors. STPs generate a vast range of Volatile Organo-Sulfur Compounds (VOSCs) which contribute to malodor. Most of the malodorous compounds (*VOSCs*), produced from various municipal sources associated with the STPs, may cause unpleasant feeling even when present in low concentrations. It has been estimated [1] that each person discharges *1–1.5 g sulfur per day* to the sewerage network and about 70% is of this is sulfate which comes from urine. Domestic sewage typically contains 3–6 mg/l organic sulfur mainly derived from proteinaceous materials [2] and also from sulphonates present in household detergents. Inorganic sulfur, in the form of sulfate may be present in various quantities depending on the hardness of the water, typically in the concentration range of 30–60 mg/l. Sewage contains large quantities of many organic and inorganic components that are usually utilized by a variety of different micro-organisms [3]. The dominant type of microbial activity in sewers is determined by the composition of different components in the sewage, especially electron acceptors (biological oxidants) [4].

A rapid, simple technique to detect odorous compounds was accomplished using an electronic nose. While detecting or quantifying an odor with an electronic nose, the problem of selectivity remains a critical issue since it may sense many compounds at a time due to their cross adsorption on the transducer surface. Applications of electronic nose technology to wastewater monitoring are widely reported [5–7]. There are several reports [8,9] on detection, identification and quantification of gaseous odor collected from the headspace of samples of sewage treatment works by an electronic nose. The electronic nose mimics the human olfactory system which consists of three essential elements: an array of olfactory receptor cells situated in the roof of the nasal cavity, the olfactory bulb which is situated just above the nasal cavity, and the brain. The electronic nose has three equivalent elements: an odor sensor array, a data pre-processor, and a pattern recognition engine [10–15]. An electronic nose,

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based on a 16 sensor array of electro-conductive polymeric chemoresistors, operating in transient flow mode was used [8] to sample the headspace volatiles originating from both bacterial and yeast cultures grown on agar plates. This electronic nose has not been used on-site to sense odor from the Sewage Treatment Plant (STP). The possibility of quantifying the landfill gas (LFG) odor in terms of odor-units per cubic meter (ou/$m^3$) was investigated [9] using a tin oxide sensor array. The main aim was to determine the most appropriate neural machine (MLP networks, RBF networks) model to evaluate the odor concentration and hence the influence of multiple biogas sources. Ideal sewage odor is a mixture of obnoxious Volatile Organo-Sulfur Compounds. Real life field samples from sewage treatment plants contain an even more complex mixture of volatile compounds.

It is well understood that sensing odor using an electronic nose is basically an indirect method. The response is also nonspecific in nature. The system is not applicable for on-site measurements. Unless specific odorous hazard markers are simultaneously assessed and quantified by the human sensors as well as GC–MS (gas chromatography–mass spectrometry), it is virtually impossible to apply specific control techniques.

There are two main methodologies applied to the chemical conditioning of sewage for the control of odors, namely suppression and treatment. Generally, physical processes are applied for waste gas streams where the flow and pollutant concentrations are high. Odor abatement by adsorption is a well-known phenomenon. In adsorption, the surface of a solid always accumulates molecules from a gaseous or liquid environment. Adsorbed odorous compounds may simply remain on the adsorbent surface indefinitely if they are stable and relatively unreactive in air. Some pollutants such as reduced sulfur compounds, including hydrogen sulfide, are more or less rapidly oxidized to other products which are frequently less odorous and sometimes not odorous at all. In many instances, the oxidant products are readily adsorbed and retained.

The main objective of the present research work was to develop a quantitative Odor Assessment and Control Scheme (OACS) to describe the assessment and control of odor contributed by various hazardous VOSCs present within the STPs. Assessment of odors was done by panel olfactometry, with the help of human sensors, whereas specific VOSCs that may be responsible for the odor were measured by GC–MS. Removal of odor (estimated by Olfactometry, using a set of trained sniffers) was correlated to the removal of a specific chemical compound with quite low odor threshold and which contributed largely towards the generation of odor. It may be noted that the odorous VOSCs are also hazard markers. Single exposure to Propanethiol [CAS Number: 107-03-9] [3]$LD_{50}$ (Oral) was 1790 mg/kg (rats) and [4]$LC_{50}$ inhalation (ppm) for 4 h was 7300 mg/kg (rats). For ethyl methyl sulfide [CAS Number: 324-89-5] $LD_{50}$ (Oral) was 4300 – >5000 mg/kg for rats and $LC_{50}$ inhalation (ppm) for 4 h was 21.7/4 h (rats) [16]. Specifically, liquid phase adsorption of 1-propanethiol, ethyl methyl sulfide and disulfide, bis [1-(methylthio) ethiol] from sewage wastewater was carried out with the twofold aims of de-aromatization at source as well as removal of these potentially hazardous odor markers.

## 2. Materials and methods

### 2.1. Sampling and assessment of odor

#### 2.1.1. Design of experiment: sampling

A case study was carried out at Baranagar–Kamarhati sewage treatment plant (Fig. 1) which falls under the purview of the KMDA (Kolkata Municipal Development Authority, Kolkata, India). The plant was situated in Baranagar–Kamarhati Municipal town, Mathkol, near Belgachia Metro Car Shed, Baranagar, West Bengal, India (Lat: 22.643°N; Long: 88.365°E). This plant receives domestic sewage from the population of the Kamarhati and Baranagar municipality and its present capacity is 40 MLD (Million Litre per Day).

Samples of odorous emissions were collected from raw sewage, which was supposed to be a source having the highest levels of malodorous compounds. This was accomplished during the period of May–July, 2012. A big drum was filled with the raw sewage, leaving a headspace of approximately 3900 ml, and then the lid was tightly locked *in situ* using Teflon seals and tape to prevent any loss of volatile compounds during transport to the laboratory and handling in the laboratory before any experiments on odor control assessment by adsorption and olfactometry. The average ambient air temperature during sampling was 39 °C. Samples were collected between 9:30 a.m. and 10:00 a.m. Upon arrival in the laboratory, the drum was weighed. Immediately after weighing, the drum was perforated at the top and VOCs were collected from the headspace of the tank using an air sampling pump (*Make:* Supelco, USA; *Model:* Escort Elf). Air samples were collected through small preconditioned adsorbent tubes (*Make:* Supelco, USA; *Model: ORBO 32*; *Specification:* 6 mm OD, 75 mm in length) according to the ambient air sampling guidelines recommended by the *USEPA* (*Method TO-17*), [17]. Sampling was performed at a flow rate of 2 l $min^{-1}$, for 2 min, providing an air volume of 4.0 l. Simultaneously, olfactometry was also carried out using the panel method, (shown in Fig. 2), as per *VDI Guidelines* [18,19].

#### 2.1.2. Design of experiment: assessment

All the tests were carried out inside an odor-free, clean laboratory with selected and trained panelists for the psychophysical analysis of odor. The trained sniffers were first tested with n-Butanol for representativeness, repetitiveness and consistency as per VDI guidelines for Panel Olfactometry [18,19]. The precursor gas mixture, containing the VOCs, was collected simultaneously for the olfactometry and GC–MS analysis. Once these analyses were over, 230 g of powdered activated carbon (PAC) was added in the sewage sample and properly mixed. This was done for batch equilibrium analysis. Samples were then collected at different time intervals using same method of sampling till an equilibrium condition was attained particularly with respect to the olfactometry outputs. The equilibrium corresponds to that particular odor intensity, which did not change any further. Since the samples for GC–MS could not be analyzed instantaneously after collection, equilibrium was ensured from the odor-intensity scales of the panelists. After sampling, each sorbent tube was placed inside a tightly capped 5.0 ml special tube holder in order to prevent any ambient contamination of the sorbents. These were kept in a freezer until they were processed further for solid phase extraction and solvent desorption of the VOCs.

*2.1.2.1. Solid phase extraction–solvent desorption.* One of the most common sampling techniques is the adsorption of VOCs on solid sorbents. Once the analytes are trapped in sorbents, they must be released for analysis. The most common extraction techniques are solvent extraction (SE) and thermal desorption (TD). Solvent extraction allows higher flow rates, longer sorbent beds and larger total-sample volumes than TD [20–22]. Typically carbon disulfide

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[3] LD stands for "Lethal Dose". $LD_{50}$was the amount of a material, given all at once, which causes the death of 50% (one-half) of a group of test animals. The $LD_{50}$ was one way to measure the short-term poisoning potential (acute toxicity) of a material. The $LD_{50}$can be found for any route of entry or administration but dermal (applied to the skin) and oral (given by mouth) administration methods were the most common.

[4] LC stands for "Lethal Concentration". LC values usually refer to the concentration of a chemical in air but in environmental studies it can also mean the concentration of a chemical in water. For inhalation experiments, the concentration of the chemical in air that kills 50% of the test animals in a given time (usually 4 h) was the $LC_{50}$ value.

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