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# Electronic Noses for Surveillance of Wastewater from Secondary Treatment Plants in Alexandria, Egypt

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## ABSTRACT

Intermittent discharges of industrial chemical pollutants into sewers can have a damaging effect on the processes involved in treating wastewater. They are sometimes able to pass through treatment plants and enter into the receiving water, to exert a harmful effect on the environment and possibly pass down a river to the intakes for water supplies. We evaluated the use of an electronic nose (E-Nose) consisting of ten non-specific metal oxide sensors for monitoring odors of wastewater from influent and effluent samples at three different wastewater secondary treatment plants in Alexandria, Egypt; over a 7-month period; in association with causes of odor by standard laboratory chemical analysis. We analyzed E-Nose sensor responses of all plants using the principal component analysis (PCA), which allowed interpretation and differentiation of samples in terms of origin and quality along the study period. E-Nose sensor responses for influent and final effluent samples from the three secondary treatment plants were significantly associated with Hydrogen Sulphide ($H_2S$, mg/l), Ammonia ($NH_3$, mg/l) and Nitrate ($NO_3$, mg/l). Thus, the E-Nose can be used as a rapid alarm generator towards volatile compounds, e.g. in specific advanced treatment processes to produce reclaimed water from effluent of the domestic wastewater treatment plant under scrutiny.

## 1. INTRODUCTION

Wastewater arriving at sewage treatment plants is highly variable in nature, due to the diurnal nature of human activity and the environmental factors such as rainfall, in addition to shorter term variability effects due to intermittent or accidental discharges of chemical pollutants into sewers (Baawain 2013, Leusch 2013). These latter discharges, usually of industrial origin, can have a damaging effect on the processes involved in treating wastewater and are sometimes able to pass through treatment plants and

enter into the receiving water, to exert a harmful effect on the environment and possibly pass down a river to the intakes for water supplies (Leusch 2013). At present, the major method used to control the effects of such discharges is via trade waste control. However, the ability to readily identify such sources of intermittent discharge using conventional analytical methods can be extremely difficult. As the techniques used to identify complex mixtures of chemicals or specific compounds are relatively sophisticated and expensive, this precludes the cost of regularly collecting a large number of samples. Another difficulty in the detection of chemicals in wastewater is the probability of obtaining a sample of the offending discharge. Therefore, the development of a measurement technique for identifying sudden changes in the wastewater as it arrives at a sewage treatment plant would be very valuable. The Electronic Noses (E-Nose) technology had been used successfully for assessing the quality of water and wastewater with acceptable levels of repeatability and reproducibility (Stuetz 1999, Dewettinck 2001, Persaud 2005, Mohamed 2009).

The E-Nose is an instrument that is designed to detect and discriminate among complex odors using an array of nonspecific chemical sensors. Most odor sensations are produced by mixtures of hundreds of odorants rather than by a single compound. Individual components tend to harmonize or blend together in mixtures leading to perceptual fusion (Brattoli 2011). E-Nose mimics the human olfaction system, which consists of three essential elements: a sensor array which is exposed to the volatiles, conversion of the sensor signals to a readable format, and software analysis of the data to produce characteristic outputs related to the odor encountered (Dewettinck 2001, Clesceri 2005, Persaud 2005). The output from the sensor array may be interpreted via a variety of methods, such as pattern recognition algorithms, principal component analysis, canonical discriminant analysis, cluster analysis and artificial neural networks, to discriminate among samples. Many E-Noses are commercially available today and have a wide range of applications in various markets and industries ranging from food processing, industrial manufacturing, quality control, environmental protection, security, safety and military applications to various pharmaceutical, medical, microbiological and diagnostic applications (Jinks 2001, Mohamed 2002, Mohamed 2013a,b).

The objectives of the present study are to: 1) evaluate the use of E-Nose technology for monitoring odors of wastewater from both influent and effluent samples of three randomly-selected secondary treatment plants in Alexandria, Egypt, 2) investigate seasonal effects of wastewater samples from different types and origin over the period of study, and 3) analyze E-Nose sensor responses in correlation with chemical causes of odors, on bases of standard laboratory analysis techniques of wastewater.

## 2. MATERIAL AND METHODS

### *2.1 Chemical Analysis*

Wastewater samples were taken from three randomly-selected secondary treatment plants in Alexandria, Egypt; namely: El-Hannoville, Km 26, and El-Iskan secondary treatment plants. The geographical locations of the three treatment plants are shown in the Google Earth map of Figure 1. Samples were collected systematically from the influent and effluent of the three treatment plants on a monthly basis for 7 months from March to November 2012.

Samples were collected in two separate containers; one (2 L) for standard physical (i.e., Biological and Chemical Oxygen Demand, Oil and Grease, Suspended Solids and Setteleable Solids) and chemical (i.e., Hydrogen Sulphide, Ammonia, Nitrate, and Phosphate) analysis at the central laboratory of Alexandria Drainage Co., Alexandria, Egypt; according to the 'Standard Methods for the Examination of Water and Wastewater' (Young 2005). Second tube samples (4 ml) were transported in an ice box immediately to the Medical Biophysics Department, Medical Research Institute, Alexandria University; Egypt where odor profile measurements were carried out by an E-Nose. Once in our laboratory, all samples were transferred to Tanex sealed vials and were left to rest at room temperature (25 ºC) for an incubation period of two hours, to gather as much vapor in the headspace region, before measurement using an E-Nose, as detailed elsewhere (Mohamed 2009).


**Figure 1.** Regional map showing geographical location of the El-Hannoville, Km 26, and El-Iskan wastewater secondary treatment plants in Alexandria, Egypt, as by Google Earth.

*2.2 Electronic Nose Analysis*

For the experiments, we used a portable E-Nose (PEN3, Airsense Analytics GmbH, Schwerin, Germany) with 10 different metal-oxide sensors, that measure independently and register continuously relative changes in electrical conductance due to a vapor or odor in the headspace over a sample. The headspace is the space just above the liquid sample in a tube. The carrier gas, such as dry air, is supplied at the inlet of a long side port needle (20-G, 10 cm long) and the concentration of test odor at the liquid surface carried by the carrier gas is supplied to the E-Nose sensors through a short side port needle (20-G, 7 cm long). Solenoid valves alternately switch the pure carrier gas and

the headspace sample vapor, and the difference in the sensor output is recorded. For monitoring, an internal pump is sucking the sample gas compounds through the sensor array. For dilution, a second pump is transferring filtered reference air into the sensor array. By using this zero gas and comparing it to the signals from the analyzed sample gas the effect of the possible drift of the sensor itself is reduced, which is known as differential measuring technique.

Following a 50 s flushing time and a 10 s zero point trim time period each, sealed Tanex vials containing wastewater samples were connected subsequently to the E-Nose inlet through a Teflon tube (3mm internal diameter) connected to a long side-port needle performing the seal of the vial, for a 60 s measurement period as instructed by producer. A second short needle performing also the seal is used to connect the vial to room dry air. During the flushing period, sensors are rinsed with zero-gas and their signals move back to the baseline (G/G0=1). The driving software interacts with the user by displaying the correct time points to connect and disconnect the sample to the E-Nose inlet. All measurements were repeated twice and results files containing sensors patterns for every experiment were saved for subsequent analysis. Moreover, patterns of each individual sensor were used to calculate average sensor response for influents and effluents of the three treatment plants, which were used for subsequent correlation analysis towards different chemical and physical parameters.

### *2.3 Data Analysis*

All E-Nose measurements were analyzed using the Principal Components Analysis (PCA) technique, as recently reported by our group (Mohamed 2013a,b). PCA algorithm is the orthogonal linear transformation of data from the 10 sensor pattern to a two-dimensional coordinate system. The largest variance by any projection of the data comes to lie on the first coordinate, which is called first main axis; and the second largest variance on the second coordinate, which is called the second main axis. PCA is theoretically the optimum transform for given data in least square terms.

Descriptive statistics were calculated for the mean ± SD of chemical and physical measurements for influent and effluent wastewater samples from the three treatment plants using standard analytical methods. Differences were considered significant at *P*-values less than 0.05. In the correlation study, the average for each sensor response was considered the dependent variable while, the individual physical or chemical parameter was considered the dependent variable. Correlation coefficients *R* and their significance values *P* were calculated for all associations.

## 3. RESULTS AND DISCUSSION

Municipal wastewater is mainly comprised of water (99.9%) together with relatively small concentrations of suspended and dissolved organic and inorganic solids (FAO 2014a). Among the organic substances present in sewage are carbohydrates, lignin, fats, soaps, synthetic detergents, proteins and their decomposition products, as well as various natural and synthetic organic chemicals from the process industries. The main target of wastewater treatment is the elimination of human and industrial effluents without endangering human health or the natural environment. Conventional wastewater treatment consists of a combination of physical, chemical, and biological

processes and operations to remove solids, organic matter and, sometimes, nutrients from wastewater. General terms used to describe different degrees of treatment, in order of increasing treatment level, are preliminary, primary, secondary, and tertiary and/or advanced wastewater treatment. In some countries, disinfection to remove pathogens sometimes follows the last treatment step.

While primary treatment removes settleable organic and inorganic solids and floating materials, secondary treatment of the effluent removes the residual organics and suspended solids. In most cases, secondary treatment follows primary treatment and involves the removal of biodegradable dissolved and colloidal organic matter using aerobic biological treatment processes (FAO 2014b). Aerobic biological treatment is performed in the presence of oxygen by aerobic microorganisms (i.e., bacteria) that metabolize the organic matter in the wastewater, thereby producing more microorganisms and inorganic end-products (e.g., $CO_2$, $NH_3$, and $H_2O$). Several aerobic biological processes are used for secondary treatment differing mainly in the method of oxygen supply to the microorganisms and in the rate of their metabolism to the organic matter (FAO 2014c). Table 1 shows that the average values for chemical and physical parameters of influent wastewater samples were significantly different ($P < 0.01$) from those of the effluent for El-Hannoville, Km 26, and El-Iskan secondary treatment plants along study period, using standard analytical methods. Moreover, the reported values of each parameter for the three treatment plants are in agreement with earlier reports carried out for Alexandria and Giza, Egypt (FAO 2014b,c).

**Table1.** Average values for chemical and physical parameters of influent and effluent wastewater samples from the El-Hannoville, Km 26, and El-Iskan secondary wastewater treatment plants along study period using standard analytical methods.

| Analysis Parameters | **El-Hannoville** | | **Km 26** | | **El-Iskan** | |
|---|---|---|---|---|---|---|
| | *Influent* | *Effluent* | *Influent* | *Effluent* | *Influent* | *Effluent* |
| Biological Oxygen Demand (mg/l) | 182.57[a]* | 66.43 | 131.86[b] | 59.29 | 132.57[c] | 62.14 |
| Chemical Oxygen Demand (mg/l) | 424.43[a]* | 182.57 | 383.57[b] | 119.43 | 322.86[c] | 107.14 |
| Hydrogen Sulphide ($H_2S$, mg/l) | 3.4[a]* | 0.06 | 1.23[b] | 0.06 | 2.60[c] | 0.06 |
| Ammonia ($NH_3$, mg/l) | 34.00[a]* | 8.79 | 19.93[b] | 8.32 | 22.59[c] | 9.73 |
| Nitrate ($NO_3$, mg/l) | 3.80[a] | 6.97 | 4.35[b] | 5.50 | 4.11[c] | 7.01 |
| Phosphate ($PO_4$, mg/l) | 1.64[a] | 0.80 | 1.54[b] | 0.70 | 2.20[c] | 1.26 |
| Oil and Grease (mg/l) | 37.57[a] | 4.29 | 34.00[b] | 12.29 | 40.14[c] | 16.57 |
| Suspended Solids (mg/l) | 186.71[a]* | 13.57 | 175.00[b] | 13.43 | 133.71[c] | 16.71 |
| Settleable Solids in 30 min. (ml) | 1.10[a] | ND | 0.90[b] | ND | 0.57[c] | ND |

[a, b, c] $P < 0.01$ as compared to respective Effluent treatment plant.
* $P < 0.01$ as compared to Km 26 and El-Iskan secondary treatment plants.

Figure 2 shows a PCA cluster plot for the influent wastewater samples from the three wastewater treatment plants along the study period. All influent wastewater samples from the three treatment plants coincide at all months, except for the month of

June, where the El-Iskan treatment plant was affected by seasonal variations in a large degree, receiving different pollutants than the other months. Figure 3 shows comparable levels of efficiency of all treatment systems for the three secondary treatment plants, which numerical values are shown in Table 1, as evidenced by coinciding PCA clusters for the effluent wastewater samples. Common high-rate processes include the activated sludge processes, trickling filters or biofilters, oxidation ditches, and rotating biological contactors. A combination of two of these processes in series (e.g., biofilter followed by activated sludge) is sometimes used to treat municipal wastewater containing a high concentration of organic material from industrial sources. High-rate biological treatment processes, in combination with primary sedimentation, typically remove 85 % of the biochemical oxygen demand and suspended solids originally present in the raw wastewater and some of the heavy metals (FAO 2014a).

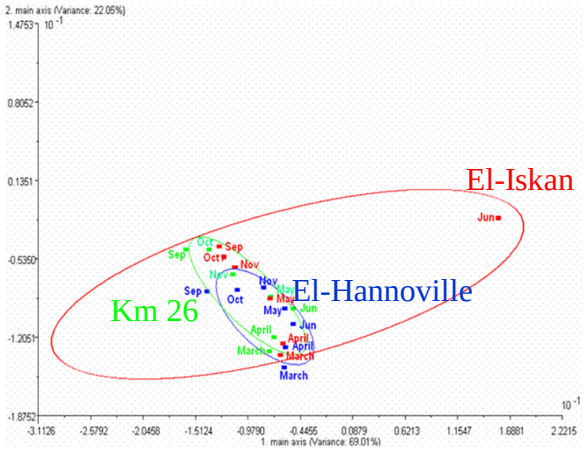


**Figure 2.** Principle component analysis (PCA) for the influent wastewater samples from the El-Hannoville, Km 26, and El-Iskan secondary treatment plants along the study period.

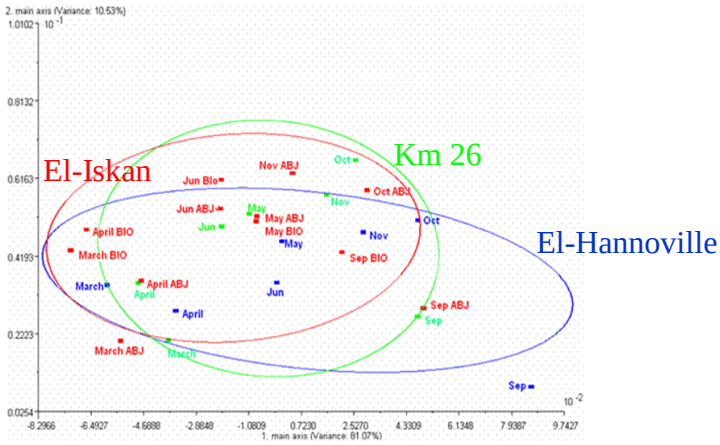


**Figure 3.** Principle component analysis (PCA) for the effluent wastewater samples from the El-Hannoville, Km 26, and El-Iskan secondary treatment plants along the study period.

The statistical analysis (Table 2) showed that the sensors: R1, R2, R3, R5, R6, R8 and R9 correlated significantly ($P < 0.05$) to the chemical causes of odor: $H_2S$, $NH_3$, and $NO_3$ compounds emanated from wastewater secondary treatment plants. Persaud (2005) had proposed earlier utilizing an array of metal oxide sensors sensitive to sulphurous compounds, which are characteristic of organic decay, which have proven to be robust for continuously monitoring odor changes over time at the inlet of a sewage plant.

**Table 2.** Correlation analysis between different E-Nose sensor responses and traditional laboratory analysis for $H_2S$, $NH_3$ and $NO_3$ compounds.

| Sensor Response | | | Hydrogen Sulphide ($H_2S$, mg/l) | | | | Ammonia ($NH_3$, mg/l) | | | | Nitrate ($NO_3$, mg/l) | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Sensor #** | **Mean** | **± SD** | **Mean** | **± SD** | ***R*** | ***P*** | **Mean** | **± SD** | ***R*** | ***P*** | **Mean** | **± SD** | ***R*** | ***P*** |
| R1 | 1.22 | 0.06 | | | *0.41* | *0.03* | | | *0.43* | *0.03* | | | *-0.12* | *0.30* |
| R2 | 1.29 | 0.11 | | | *0.18* | *0.22* | | | *0.21* | *0.18* | | | *0.43* | *0.03* |
| R3 | 1.16 | 0.04 | | | *0.31* | *0.09* | | | *0.47* | *0.02* | | | *0.47* | *0.02* |
| R4 | 1.05 | 0.02 | | | *0.09* | *0.35* | | | *-0.20* | *0.19* | | | *0.23* | *0.16* |
| R5 | 1.08 | 0.02 | 2.41 | 1.83 | *0.41* | *0.03* | 25.51 | 9.38 | *0.45* | *0.02* | 4.09 | 1.68 | *-0.02* | *0.47* |
| R6 | 1.10 | 0.12 | | | *0.40* | *0.04* | | | *-0.04* | *0.44* | | | *0.60* | *0.002* |
| R7 | 1.05 | 0.16 | | | *0.14* | *0.27* | | | *0.23* | *0.16* | | | *0.04* | *0.44* |
| R8 | 1.12 | 0.11 | | | *0.32* | *0.08* | | | *-0.07* | *0.38* | | | *0.55* | *0.005* |
| R9 | 1.10 | 0.18 | | | *0.19* | *0.21* | | | *0.30* | *0.09* | | | *0.05* | *0.42* |
| R10 | 1.13 | 0.06 | | | *0.06* | *0.39* | | | *-0.27* | *0.12* | | | *0.16* | *0.24* |

*R*: correlation coefficient, *P*: significance level.

## 4. CONCLUSIONS

We evaluated the use of an E-Nose consisting of ten different non-specific metal oxide sensors for monitoring odors of wastewater from influent and effluent samples at three randomly-selected wastewater secondary treatment plants in Alexandria, Egypt. We analyzed E-Nose sensor responses of all treatment plants using PCA, which allowed interpretation and differentiation of samples in terms of origin and quality along the study period of 7 months. E-Nose sensor responses for influent and final effluent samples from the three secondary treatment plants were significantly associated with Hydrogen Sulphide ($H_2S$, mg/l), Ammonia ($NH_3$, mg/l) and Nitrate ($NO_3$, mg/l). Thus, the E-Nose can be used as a rapid alarm generator towards volatile compounds, e.g. in specific advanced treatment processes to produce reclaimed water from effluent of the domestic wastewater treatment plant under scrutiny.

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