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Electronic nose for smart identification of roofing and paving grade asphalt

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Abstract

Asphalt is a complex mixture of hydrocarbons, whose properties strongly depend on the source and type of crude oil and refining processes. From a technical standpoint, intensive investigations carried out by the construction sector, above all by road researchers, have attempted to understand relationships between asphalt binder chemical structure, morphology and physical characteristics. Nevertheless, one challenge that the advance research on asphalt products actually face is to transfer this extremely high level of knowledge to applied industrial technologies for finding easy-to-use, quick and cost-effective test methods for quality control and identification of asphalt binders at refinery, terminal and plant. Thus, this paper focused on the development of a protocol for fingerprinting, including identification and discrimination, of asphalt cements using two different electronic noses (e-noses), also known as artificial olfactory systems (AOS). E-nose is a biomimetic non-destructive intelligent sensing instrument, which is designed to mimic the human sense of smell to detect, compare and classify odor sample, producing a qualitative output (fingerprint). Results suggested that a complementary combination of electronic nose technique and well-established analytical methodologies could be successfully used for the identification and discrimination of roofing and paving grade asphalt cements. Specifically, both sensing instruments were able to perform a good discrimination between products characterized by a different chemical nature and to verify the refinery process stability during production and a batch-to-batch crude oil consistency.

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1. Introduction

Asphalt, often referred to as asphalt binder/cement or bitumen, is a dark brown-to-black cementitious material obtained from natural deposits or as a byproduct of petroleum refining process (ASTM D8). More generally, it is defined in the European specifications as a virtually involatile, adhesive and waterproofing material which is completely or nearly completely soluble in toluene, and very viscous or nearly solid at ambient temperature (EN 12597). The vast majority of refined asphalt is used for engineering and construction purposes: primarily as a constituent of products used in paving and roofing applications (Hunter et al., 2015). From a technical standpoint, intensive investigations aimed to better understand asphalt cement composition and behavior provided fundamental insights into the design and evaluation of its post-application performances (Huang and Di Benedetto, 2015; Loizos et al., 2009;). Specifically, asphalt cement is a complex heterogeneous mixture of hydrocarbons, whose properties are strongly influenced by the inherent characteristics of the crude oil from which it was produced and by the refining processes (Planche, 2014). Chemical methods, based on solvent separation, spectrometry and chromatography techniques, have highlighted specific information related to elemental and molecular composition, functional groups and molecule size (Bissada et al., 2016; Masson et al., 2001; Wang et al., 2017). Recently, advanced imaging analysis techniques have represented useful tools for characterizing asphalt surface microstructure (Fischer and Cernescu, 2015; Mikhailenko et al., 2017). Nowadays, the asphalt binder composition and structure are generally assumed to follow the model of a colloidal suspension of asphaltenes micelles in a maltene (saturates, aromatics, resins) matrix (Lesueur, 2009). As far as the physical and mechanical properties, asphalt is considered a viscoelastic liquid characterized by a temperature dependent behavior. Although practitioners have used for decades' empirical test (penetration and ring and ball temperature) to evaluate and grade asphalt binder, researchers tended to move since the '90 towards a performance related approach based on rheological measurements in a wide operating temperature range (Anderson et al., 1994; Redelius and Soenen, 2015; Weigel and Stephan, 2018).

Nevertheless, one challenge that the advanced research on asphalt products actually faces is to transfer this extremely high level of knowledge to applied industrial technologies for finding easy-to-use, quick and cost-effective test methods for quality control and identification of asphalt binders at refinery, terminal and plant. Monitoring process stability during production (contamination, batch-to-batch consistency, process line change-over), ensuring the fulfillment of quality requirements and discriminating batches before unloading procedures from storage tanks represent only some examples in which operators require real-time and on-site measurements and results on many samples. For this purpose, a technical solution would be represented by the electronic nose (e-nose), also known as artificial olfactory system (AOS). E-nose is a biomimetic non-destructive intelligent sensing instrument, which is designed to mimic the human sense of smell to detect, compare and classify odor sample, producing a qualitative output (Gardner and Bartlett, 1994; Jha et al., 2019 Zhang et al., 2018). An electronic nose is usually based on an array of sensors with different selectivity, that generates complex multi-dimensional data for each measurement, combined with specific pattern-recognition (PARC) algorithms (Burlachenko et al., 2016; Scott et al., 2006, Takamichi, 2016). The interaction between sample vapor molecules and sensors is translated into an electrical signal which identifies a characteristic global odorous signature defined fingerprint or smellprint (Autelitano et al., 2017; Wen et al., 2018). The application and marketing of electronic noses have seen considerable development in recent years as a result of the rapid and great advances in the field of sensor technology, electronics and artificial intelligence (Röck et al., 2008; Wilson and Baietto, 2009). Several benchtop and portable electronic noses, tailored on specific applications and objectives, are currently available on the market based on different types of sensors (Arshak et al., 2004; Pearce et al., 2003). Most applications of AOS concern the agri-food and the industrial production sector, even though homeland security and personal safety, environmental monitoring and biomedical diagnosis are important field of use (Deshmukh et al., 2015; Hsieh and Yao, 2018; Loutfi et al., 2015). But, the potential of electronic noses in the petroleum industry to date is still under-analyzed: only recent studies have used this solution to characterize petroleum-derived products and their odorous emissions in the various stages of production and paving (Autelitano and Giuliani, 2018a and 2018b; Ferreiro-González et al., 2017).

Thus, this paper focused on the development of a protocol for fingerprinting, including identification and discrimination, of asphalt cements using the electronic nose approach. Two e-noses which imply different detection, operation, sampling and response techniques were selected: a benchtop prototype, equipped with metal-oxide

semiconductors sensors, which communicate with an external elaboration unit (ISE Nose 2000) and a fully-integrated portable device fitted with a nanocomposite sensor array (Cyranose 320). The analyzed binders were paving grade asphalt, whose molecular and morphological properties were preliminary evaluated with two well-established analytical techniques, or thin-layer chromatography and flame ionization detection (TLC-FID) and Fourier transform infrared spectroscopy using attenuated total reflection technique (FT-IR/ATR).

2. Materials characterization

Three 70/100 penetration paving grade asphalts obtained from visbreaking process and named respectively A1a, A1b and A2 were used in the experimental program. A1a ($\text{Pen}_{25\text{ }^\circ\text{C}} = 73 \text{ 0.1mm}$; $\text{R\&B} = 49.2\text{ }^\circ\text{C}$) and A1b ($\text{Pen}_{25\text{ }^\circ\text{C}} = 75 \text{ 0.1mm}$; $\text{R\&B} = 49.6\text{ }^\circ\text{C}$) were produced in the same refinery using two different batches of crude oil extracted in the Mediterranean Sea, whereas A2 ($\text{Pen}_{25\text{ }^\circ\text{C}} = 84 \text{ 0.1mm}$; $\text{R\&B} = 46.4\text{ }^\circ\text{C}$) was obtained by refining an Arabian crude oil in another plant.

3. Methods

A global chemical-morphological identification of asphalt cements was carried out following two different complementary methodological approaches: the analytical one and that based on electronic nose. Firstly, in order to separate and identify the compounds that characterize the binder matrix, TLC-FID and FT-IR/ATR were performed. Then, two different e-noses were selected to detect the fingerprint: a benchtop laboratory instrumentation (ISE Nose 2000) and a portable device (Cyranose 320).

3.1. Analytical approach

The determination of SARA (saturates, aromatics, resins and asphaltenes) fractions was carried out by using a IATROSCAN MK-5 (Iatron Laboratories Inc.) TLC-FID analytical system, according to IP 469/01 procedure. The separation was made with the TLC method on the S-III chromarods, i.e. quartz rods coated with a thin layer adsorbents, as stationary phase and different solvents as mobile phases, following a three-stage solvent development sequence. Saturates were eluted with heptane, aromatics with a solution of toluene and heptane (80:20 by volume) and resins with a solution of dichloromethane and methanol (95:5 by volume). The detection was performed using a flame ionization detector: the chromarods were scanned (30 sec/scan) through a hydrogen/air flame. For each binder, 20 chromarods were tested and the mean value of the 20 measurements was calculated. Starting from SARA values, three indexes were calculated: the colloidal instability index (CII) as the ratio of the sum of asphaltens and saturates to the sum of resins and aromatic, the $I_{\text{Ar/As}}$ as the ratio of the content of aromatics and asphaltenes and the $I_{\text{As/Re}}$ as the ratio of the content of asphaltenes and resins (Lesueur, 2009; Weigel and Stephan, 2018).

A Nicolet 5PC FT-IR spectrophotometer (ThermoFisher Scientific) was used to determine the asphalt binders' spectrum in the mid-IR region ($4000\text{--}400 \text{ cm}^{-1}$). The analyses were performed by using an ATR cell equipped with a diamond single crystal. The working principle is based on the measurement of changes that occur in a totally internally reflected IR beam, when it comes into contact with the sample. Specifically, an IR beam enters the ATR crystal and it is totally reflected at the crystal to sample interface. This internal reflectance creates an evanescent wave, which protrudes only a few microns beyond the crystal surface and into the sample. In this spectral region, where the sample absorbs energy, the evanescent wave is attenuated or altered. After one or several internal reflections, the IR beam exits the ATR crystal and is directed to the IR-detector, which generates an IR spectrum (Atkins et al., 2017).

3.2. Electronic nose approach

The ISE Nose 2000 (Soatec) is configured as a benchtop prototype characterized by three different units that operate in sequence: headspace sampling, signal acquisition and conditioning, data processing (Fig. 1a). The semiautomatic sampling section was equipped with a 16 positions servo-assisted solenoid valve which was connected to 50 mL septum-sealed glass vials containing the asphalt cement samples ($0.100 \text{ g} \pm 0.001 \text{ g}$) through Teflon tubes, fittings

and disposable 18 G needles. The signal acquisition and conditioning section, which represents the system sensory apparatus, contains the olfactory chamber. The sensor array was arranged with 12 commercial metal oxide semiconductors (MOS) sensors, which were set at 47 °C. They are made of a ceramic sheet, internally heated by an electrical resistance and covered on their surface by a tin dioxide (SnO₂) layer. The elaboration unit (a pc on which the control software operates) allowed the fingerprint identification and the subsequent statistical data post-processing. The ISE Nose working principle is based on the variation of the sensors electrical resistance caused by a surface oxidation of the analyte and by the reduction of the oxygen previously adsorbed and activated on the sensor surface. The fingerprint elaboration provided for a four-stage measurement procedure: baseline, injection, desorbing and purge. The first three phases represent the exposition method, in which the data coming from the sensory module are scanned and stored by the elaboration unit. During the baseline phase (time: 15 s), a controlled flow (50 mL min⁻¹) of chromatographic air (80% N₂, 20% O₂) was continuously maintained on the sensors: the oxygen is adsorbed by the metal oxide surface causing a high sensor resistance (R₀). When injection occurred (time: 5 s), the chromatographic air flow was then diverted into the vial. The carrier gas, enriched with the volatile compounds present in the asphalt binder headspace, was then transported on the sensor surface leading to a reduction in the sensors resistance ($\Delta R = R - R_0$). In the desorbing phase (time: 190 s), the exposition chamber was cleaned by asphalt cement headspace traces with a pure carrier gas flow. Finally, the intra-batch purge step allowed to refresh the sensors' surface and the hydraulic connections and components inside the system (time: 100 s; flow: 500 mL min⁻¹), restoring the baseline electrical resistance values. The whole data set was constituted by 45 independent measurements: 15 specimens for each asphalt.

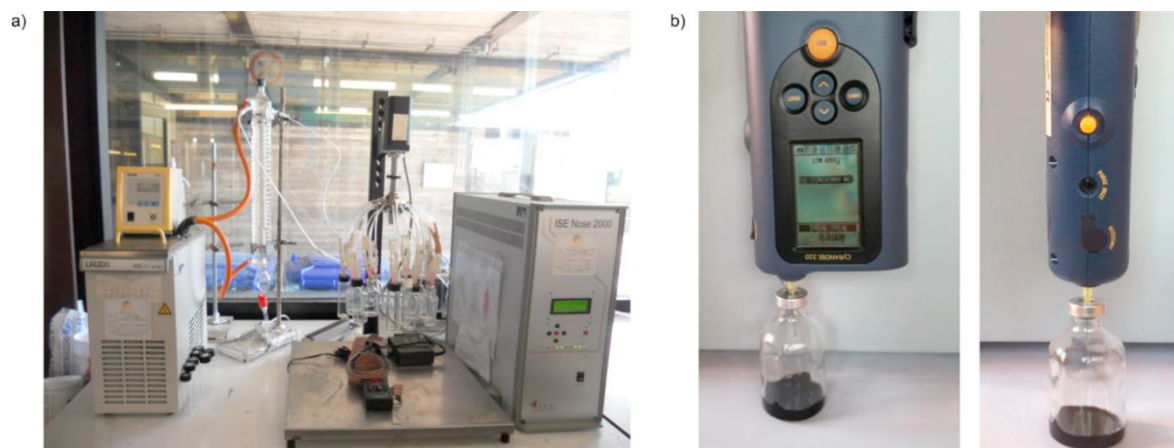


Figure 1. ISE Nose 2000 (a) and Cyranose (b)

The Cyranose 320 (Cyrano Sciences) is a commercially available, handheld and fully-integrated e-nose device: sample delivery system, sensor array, computing system (Fig. 1b). 32 different nanocomposites (polymer + carbon black) chemiresistors with high sensitivity, which are able to convert the vapor concentration into an electrical signal, constitute the NoseChip™ array. Adsorption of volatile compounds on the surface film of the sensor involves a reversible process which causes the polymer matrix expansion and the disruption of the conductive carbon black pathways and reflects into an increasing of the electrical resistance. Asphalt samples (3.000 ± 0.005 g) were placed in septum-sealed 50 mL glass vials for headspace generation. The sampling occurred by means of a disposable 20 G needle and a vent (23 G needle) was inserted into the septum to equilibrate the internal pressure. The temperature of the sensor array substrate was set and maintained at 41 °C. The measurement cycle consisted of a four-stage process: baseline purge, sample draw, purge air intake and purge sample intake. During the definition of the baseline resistance (R₀), ambient air was pulled by a vacuum pump and was led through a tube in the sensor chamber (time: 30 s; flow: 120 mL min⁻¹). Then, in the sampling phase (time: 25 s; flow: 120 mL min⁻¹) the chemical vapors reached the NoseChip™, interacting with the surface of the sensor's active material. Finally, after the sample removal, two cycle of sample gas and air intake purge (total time: 210 s; flow: 180 mL min⁻¹) were applied to refresh the sensors and to purge the pneumatic system of any residual sample vapors, restoring the initial configuration. Using pattern matching

algorithms, data were converted into a unique response pattern necessary to identify the fingerprint. The data set, as well as for the ISE Nose 2000, was constituted by 45 independent measurements: 15 specimens for each binder.

4. Results

The group-type separation into SARA fractions pointed out a difference in the macro-molecular composition between A1a and A1b, i.e. asphalt cements produced in the same plant starting from the same crude oil, compared to A2 especially in terms of fraction molecular weight, testifying a distinct chemical nature (Tab. 1). The most significant difference concerned the key fraction of asphaltenes, or the insoluble in n-heptane but soluble in toluene (ASTM D3279), translating into difference also in the related chemical indexes. Asphaltenes, which are essentially polynuclear aromatic components with high molecular size and weight, strongly influence the asphalt cement behavior and its compatibility with polymers (Lesueur, 2009; Redelius, 2006).

Table 1. Asphalt cements' SARA fraction and chemical indexes: Mean and standard deviation.

Asphalt	Sa [%]	Ar [%]	Re [%]	As [%]	CII [-]	$I_{Ar/As}[-]$	$I_{As/Re}[-]$
A1a	5.7 (0.4)	48.6 (1.7)	20.9 (0.9)	24.8 (0.8)	0.44	1.96	1.19
A1b	5.8 (0.2)	45.5 (1.6)	25.9 (0.6)	22.8 (1.1)	0.40	1.99	0.89
A2	5.2 (0.4)	52.6 (2.7)	26.6 (1.7)	15.6 (1.2)	0.27	3.77	0.59

Sa = Saturates (470-880 g mol⁻¹); Ar = Aromatics (570-980 g mol⁻¹); Re = Resins (800-2000 g mol⁻¹); As = Asphaltenes (800-3500 g mol⁻¹)

The FT-IR/ATR spectra recorded for the three asphalt cements are shown in Figure 2 (4000-400 cm⁻¹ region). Within the IR spectrum, two different types of commonly defined bands or peaks are identified: those associated with specific functional groups (region 4000-1400 cm⁻¹) and those of fingerprint (region 1000-400 cm⁻¹). In the first region, absorption bands, which represent specific functional groups (OH, N-H, C=O, C-H, etc.), appear at fairly constant positions, rather independent of the structure of the molecule in which the group itself is present. In the fingerprint region, vibrational frequencies are greatly affected by the whole molecular structure and spectra are considered specific for a particular molecule but of difficult analytical interpretation. However, some functional group absorption can be identified also in the fingerprint region, especially below 1000 cm⁻¹ (Silverstein et al., 2014; Wang et al., 2018).

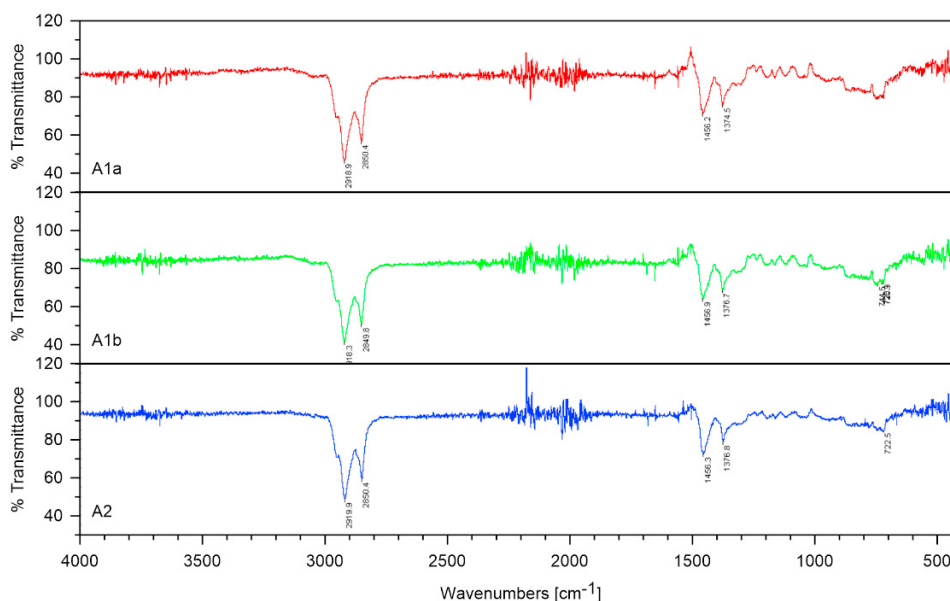


Figure 2. FT-IR/ATR asphalt cements' spectra in 4000-400 cm⁻¹ region

As shown in Figure 2, asphalt binders obviously presented the same functional groups, but very similar spectra also in the fingerprint region. The traces recorded for A1a and A1b were coincident and superimposable, whereas that related to A2, although presenting the bands at the same wavelengths, was slightly vertically shifted.

The asphalt fingerprints recorded by electronic nose displays the changes in sensor resistance as a function of time, where the sensor response is the instantaneous sensor reading minus the baseline sensor reading, divided by the baseline sensor reading $((R-R_0)/R_0)$. Figure 3, by way of example, represents 1 of the 15 fingerprints of asphalt A2, registered by ISE Nose 2000 (Fig. 3a) and Cyranose (Fig. 3b).

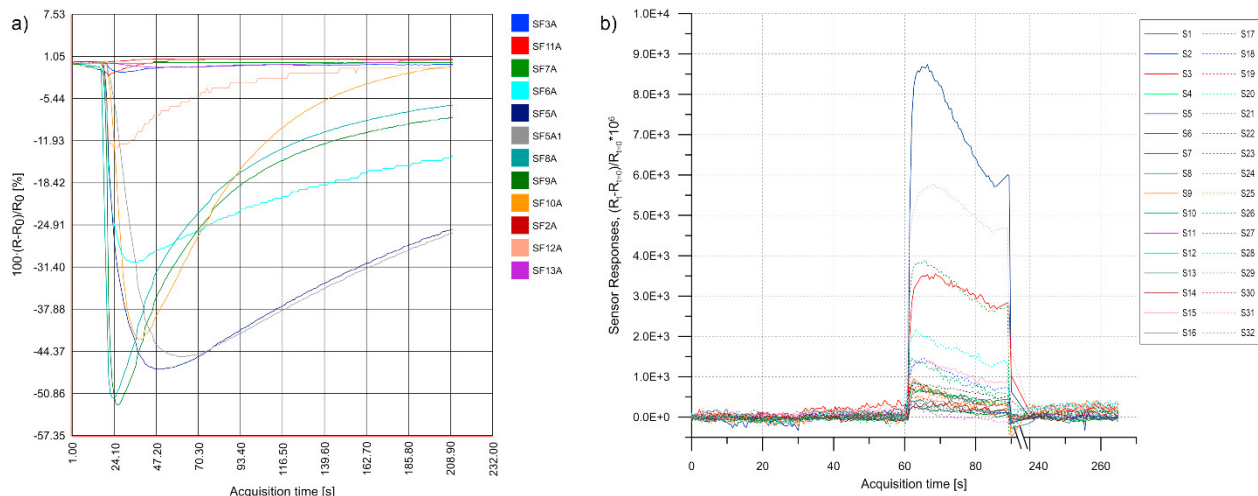


Figure 3. A2 fingerprint registered with ISE Nose 2000 (a) and Cyranose (b).

The data recorded by the e-noses resulted in a set of semi-independent variables (sensor array output) and a set of dependent variables (asphalt cements classes). The data post-processing followed the same PARC approach for both instruments. In order to optimise the PARC system, some sensors were deselected, as a standard practice in the literature. Specifically, 8 of the 12 available sensors were chosen for the ISE Nose 2000 and 24 of 32 for the Cyranose, excluding the sensors which registered almost null maximum relative changes in resistance. The raw digital responses of sensors were converted into the maximum relative resistance change caused by the binder exposure during sampling and baseline stages $(\Delta R_{max}/R_0)$. These values were then normalized, using the variance as standardization parameter, to remove the effects of response size. The normalized data were statistically processed through the principal component analysis (PCA) performed with MATLAB R2016b. PCA is an unsupervised multivariate procedure that reduces data from the sensor responses to a new set of uncorrelated variables, i.e. the principal components (PCs), which are calculated to capture the most variance in the dataset (Jolliffe, 2002). According to three different heuristic cut-off methods, or cumulative explained variance ($> 80\%$), Kaiser criterion and scree-plot, the first two principal components (PC1 and PC2) resulted to be the meaningful and worthy PCs to extract for minimizing information loss. The representation in the two-dimensional space PC1-PC2 (Figs. 4a and 4b) highlights that two different groups or asphalt cements classes were clearly distinguished. Clouds of data representative A1a and A1b were nearly complete overlapping, whereas a significant inter-class separation was visible with respect to asphalt binder A2. These results graphically translate and photograph the “manufacturing history” of the three analyzed asphalts. Specifically, asphalts produced refining different batches of the same crude oil in the same plant showed almost identical fingerprints, testifying a good process stability during production and a batch-to-batch consistency. Moreover, the inter-class separation registered between binders A1 and A2 described a substantial difference in the odorous pattern between products characterized by a different chemical nature.

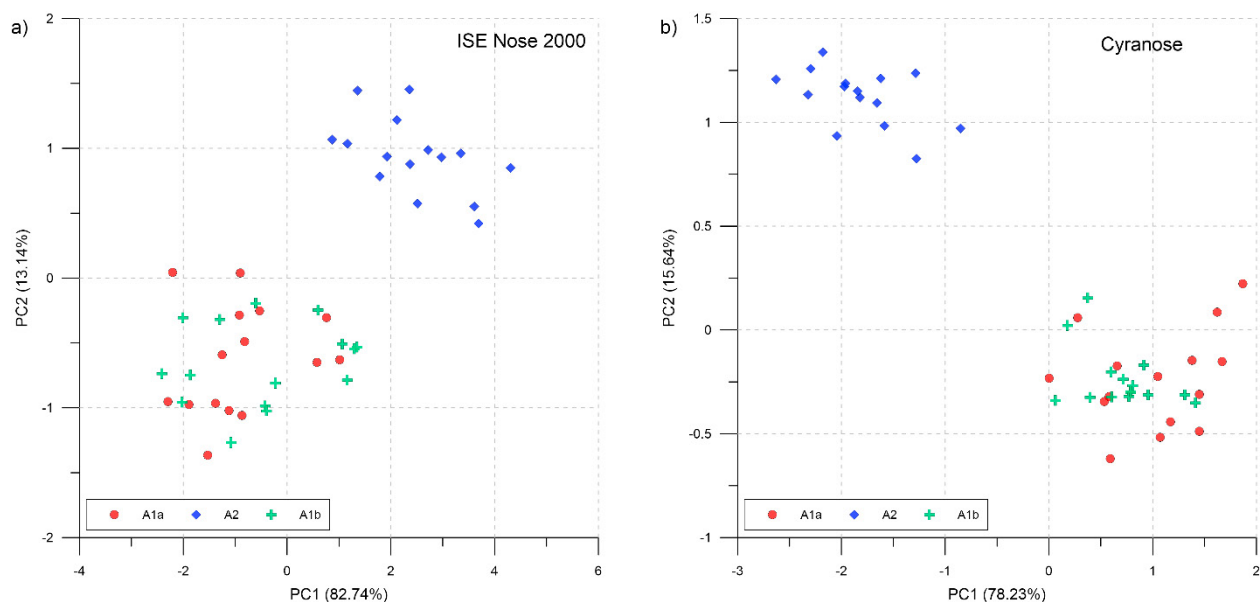


Figure 4. 2D-PCA plot elaborate on measurements performed with ISE Nose 2000 (a) and Cyranose (b).

5. Conclusions

This study of characterization of asphalts based on electronic nose technique and on well-established analytical methodologies showed that a combination of these complementary approaches could be successfully used for fingerprinting asphalt binders. Both sensing instruments were able to perform a good discrimination between products characterized by a different chemical nature and to verify the refinery process stability during production and a batch-to-batch crude oil consistency. Thus, for several asphalt industry sectors which require real-time and on-site characterization and quality control, e-nose represents an excellent cost-effective solution. As described, once calibrated a suitable operational protocol and programmed a classification analysis, these devices perform non-destructive, quick and accurate measurements without any sample pre-treatment and output data are simple to interpret also by non-expert users. Moreover, using pattern matching algorithms, e-noses are able to learn during a one-time training session the odor signature of vapors of interest, creating and developing a rich database of digitized patterns to which all future samples will be compared. Data contained in these libraries could be implemented and associated to chemical quantitative results obtained with analytical instruments, which could moreover allow to optimize and to customize the sensor array to the specific sensing needs.

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