Application of a Conductive Polymer Electronic-nose Device to Identify Aged Woody Samples

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***Abstract*— The identification of aged woody samples is often a difficult task as a result of weathering and physical deterioration over time which removes or obscures distinguishing anatomical features and characteristics required for visual taxonomic determinations. Fortunately, the chemical characteristics of aged woods usually are preserved better than physical characteristics if the wood remains dry in storage. All wood types, determined by the particular plant species from which woody samples are derived, produce and release a unique complex of volatile organic compounds that distinguish individual wood types when headspace volatiles (containing these unique chemical mixtures) are collectively analyzed using an electronic gas-sensing device such as an electronic nose. The advantage of electronic-nose devices over conventional analytical-chemistry instruments, typically used in laboratory chemical analyses, is that the woody source (plant species) from which headspace volatiles are derived may be identified without having to identify individual chemical compounds present in the headspace analyte mixture. Methods were developed for a conductive polymer type electronic nose gas-sensing device, the Aromascan model A32S, to accurately identify aged woody samples derived from wood pieces held in dry storage for long periods of time. An aroma library was developed using diagnostic aroma profile databases (electronic aroma signature patterns) from known woods of numerous tree species. The A32S electronic nose was capable of distinguishing between 44 wood types, providing correct identification determinations at frequencies ranging from 92-99%. The distribution of aroma class components, defined by wood type for each sample analyzed, also could be determined to indicate the relatedness of volatile aroma components that each sample analyte had in common with individual wood aroma classes. This information was useful for determining the taxonomic relatedness of wood types (plant species) based on the headspace volatiles that were produced. Furthermore, principal component analysis provided precise statistical numerical values (quality factors of significance) that indicated the chemical relatedness between wood volatiles based on pairwise comparisons of organic chemical mixtures from individual wood types.**

Keywords- electronic aroma detection; e-nose technologies; volatile organic compounds; wood identification

I. INTRODUCTION

There are numerous situations where the identity of wood types must be known for many commercial or industrial applications, scientific research, or forensic analyses. Conventional methods used for the determination of wood type identities involve examinations of macroscopic and microscopic anatomical characteristics of wood tissues. The identification of wood types becomes more difficult if the wood is exposed to adverse environmental conditions (such as weathering) that result in physical deterioration of the wood, masking or diminishing diagnostic anatomical characteristics required for visual taxonomic determinations. By contrast, most chemical characteristics of wood are not lost during aging or weathering as long as the wood is stored in a dry state, even over extended periods of time. Traditional chemical and microscopic methods used for wood identification hitherto are cumbersome and less reliable because they often require extensive and expensive sample preparation and time-consuming analyses. Thus, there is a need for an analytical device that quickly identifies organic samples such as wood types without the high cost of conventional chemical analyses.

Electronic-nose devices are designed to produce digital electronic signatures of volatile organic compounds (VOCs) released from any source [1-3]. Unlike other analytical instruments, these devices have the capability of identifying organic samples from the VOCs they release without having to identify individual chemical compounds present in volatile mixtures [4-6]. A variety of different sensor types have been developed for various applications including optical sensors [7], metal oxides [8, 9], semiconductive polymers [10-13], and conductive polymers [14-15]. The agricultural and food industries have utilized electronic aroma detection (EAD) technologies to evaluate food quality and product aromas [16-18], food storage life and freshness [19-20], detect industrial wastes [21-22], diagnose plant diseases [23], and for many other applications requiring gas-detection technologies [24-27].

The purposes of this study were to 1) determine if an electronic-nose (e-nose) device, the conductive polymer (CP)-type Aromascan A32S e-nose, has the capability of identifying and distinguishing between dried aged specimens of various temperate North American wood types based on headspace volatiles (given the reduced amount of volatiles released from aged wood), to 2) evaluate the effectiveness (accuracy) of wood-type determinations, and to 3) assess whether e-nose aroma data outputs provide indications of

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taxonomic-relatedness between aged specimens of various hardwood and conifer wood types.

II. MATERIALS AND METHODS

A. Collection and storage of woody samples

Aged wood blocks (4 cm long × 2 cm wide × 2 cm thick), over 20 years old and derived from an archival reference collection of temperate North American wood types stored in the pathology herbarium collection at the Southern Hardwoods Laboratory (SHL), were utilized in this study. A subset of the collection selected for e-nose analysis included 44 wood types (plant species) from 16 genera, representing both hardwood and conifer tree species. These archival wood blocks were highly desiccated while in storage. Each sample was rehydrated by soaking (complete submersion) in sterile distilled water for 15 min, followed by blotting with Kimwipes tissue paper (Kimberly-Clark Inc., Roswell, GA) to remove any free moisture from the wood surfaces immediately prior to e-nose analysis.

B. Sample preparation and prerun procedures

Wood blocks from each wood type were analyzed separately in a 500 ml Pyrex sampling bottle no. 1395 (Corning Inc., Corning, NY) fitted with reference air, sampling, and exhaust ports on a polypropylene bottle cap. Reference air entered the sampling bottle through a 3 mm polypropylene tube extending to just above the bottom of the sampling bottle. The sampling bottle was maintained at a constant air temperature of 25 C and was purged with filtered, moisture-conditioned reference air for 2 min prior to building headspace volatiles. The sampling bottle was sealed and volatiles from each wood type sample were allowed to build headspace and equilibrate for 30 min prior to each run. Reference air was maintained at 4% RH at 25 C. The sampling bottle was purged with conditioned reference air between runs to remove volatiles from the previous sample.

C. Instrument configuration and run parameters

All e-nose analyses were conducted with an Aromascan A32S (Osmetech Inc.,Wobum, MA) CP e-nose instrument with 32 sensors in the sensor array and 15 V across sensor paths. The response sensitivities of individual sensors, measured as percent changes in electrical resistance response across sensor paths relative to base resistance (%ΔR/Rbase), varied with the type of plastic polymer used in the sensor matrix coating, the type of ring substitutions used to modify its conductive properties, and the type of metal ions used to dope the matrix to improve and modulate sensor response. Detailed analyses containing calibration data for the sensor array were reported previously [23]. The block temperature of the sensor array was maintained at a constant 30 C. Reference air was preconditioned by passing room air sequentially through a carbon filter, silica gel beads, inline filter, and Hepa filter to remove organic compounds, moisture, particulates, and microbes, respectively, prior to humidity control and introduction into the sampling bottle. The flow rate (suction) of sample air at the sampling port was maintained at -702 ml/min using a calibrated ADM 3000 flow meter (Agilent Technologies, Wilmington, DE). Sensor surfaces were purged between runs using a 2% isopropanol wash solution. The instrument was interfaced with a personal computer via an RS232 cable and controlled with Aromascan Version 3.51 software. The instrument plumbing was altered from conventional architecture and specifically configured for static sampling of the headspace by allowing air flow, maintained at 605 ml/min flow rate, coming out of the external vent port of the instrument during analytical runs, and closing the exhaust port on the sampling bottle so that headspace volatiles were taken from a homogeneous static air mass within the sampling bottle.

D. Data acquisition parameters and run schedules

Data from the sensor array were collected at 1 s intervals using a 0.2 detection threshold (y-units), a 15–20 y-max graph scale, and with a pattern average of five data samples taken per run during data acquisition. A uniform run schedule was used consisting of reference air 20 s, sampling time 90 s, and wash 20 s, followed by 90 s of reference air for a total run time of 220 s. A 2 min reference air purge was completed between runs after each sample was removed from the sampling bottle.

E. Construction of reference libraries and validation

An aroma signature reference library was constructed from wood types of known reference woods of angiosperm and gymnosperm species included in this study. All database files were linked to specific (designated) aroma classes defining each sample type or category. The following recognition network options (neural net training parameters) were used for each training session: training threshold = 0.60, recognition threshold = 0.60, number of elements allowed in error = 5, learning rate = 0.10, momentum = 0.60, error goal = 0.010 (P = 0.01), hidden nodes = 5, maximum iterations (epochs) = 10,000, using normalized input data, not actual intensity data. Some parameters were modified for improvement of recognition accuracy. A typical training required 2–35 min, depending on the size of the database applied, using an IBM-compatible personal computer with a minimum of 64 mb of RAM and 350 MHz run speed. Neural net trainings were validated by examining training results that compare individual database files for compatibility or by similarity matches to each specific odor classes by test-assigned odor class distributions among related odor classes included in each library. The specific detailed analytical methods used for identification of unknowns, data processing, and statistical determinations followed the procedures and specifications indicated by Wilson et al. [28].

F. Principal component analysis

Detailed comparisons of relatedness of odor classes (wood types) were determined using principal component analysis (PCA) algorithms provided by Aromascan 3.51 software. Three-dimensional PCA was used to distinguish between wood headspace volatiles released from 12 woody conifer plant species. The mapping parameters for three-dimensional PCA were: iterations = 30, units in Eigen values (%), and use of normalized input data.

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III. RESULTS

A. Identification of wood sample types

The A32S conductive polymer e-nose correctly identified the vast majority of the 44 wood types tested based on differences in the aroma profiles of headspace wood volatiles derived from woody samples from the SHL archival wood collection. Correct identifications of unknown wood samples were determined at rates well above 90% (range 92-99%) for all wood types tested except for *Pseudotsuga menziesii*. Wood samples of this tree species were determined to be unknown, indicating that the e-nose could not assign the aroma profile to a specific aroma class, present in the aroma reference library, because the majority of the aroma components within the volatiles from this species did not fall into a single aroma class. The aroma components of *P. menziesii* were distributed more evenly among several aroma classes. However, none of the wood sample identifications were determined to be incorrect or ambiguous, defined as determinations for different wood samples of the same type that were assigned to different aroma classes or wood-identification types in separate runs.

B. Discrimination between wood types

The A32S e-nose also effectively discriminated between the headspace volatiles (aroma profiles) of most wood types tested among twelve conifer species. The aroma profiles of each wood type were further evaluated by neural net training validation during the process of creating a diagnostic aroma library for conifer woods. Following neural-net training, analysis of data for each aroma class (defined by the principal components present in aroma profiles from each wood type) provided a precise breakdown of the aroma class distribution of these principal aroma components present in volatiles among the twelve coniferous wood types as summarized in Table I. The aroma class distribution indicates (on a percentage bases) the proportion of aroma components, present in the headspace volatiles from each wood type, that are in common with principal aroma elements of volatiles from other wood types present in the reference library. Thus, the degree of overlap among principal aroma elements from volatiles of each wood type provides an indication of relatedness between plant species based on the chemical nature of volatiles released from individual wood types. All of the wood types that were identified correctly among the 12 conifer woods had a majority proportion of the aroma profile that was assigned to the principal aroma element characteristic of each individual wood type or plant species. The range of aroma class distributions attributed to an individual principal aroma element characteristic of each wood type ranged from 79.7% in *Abies concolor* to 95.8% in *Pinus ponderosa*. Only aged wood from *Pseudotsuga menziesii* (Douglas fir) had an exceptionally low proportion (15.8%) of aroma components that were attributed to its principal aroma element. Consequently, *P. menziesii* was determined as an unknown aroma profile and could not be identified. The proportion of secondary aroma elements attributed to aroma classes besides the principal aroma element ranged from <1% with several species to as high as 15.3% in *Tsuga heterophylla* with *Abies concolor* aroma elements and 18.1% in *Abies concolor* with *P. menziesii* aroma elements.

Intensity differences, using the difference-mode software option for displaying aroma signature patterns, between sensor outputs of individual sensors in the sensor array provided clues to differences in VOCs that distinguish headspace volatiles of different wood types. For example, a comparison of the differences in sensor outputs in response to volatiles from *Quercus alba* and *Tsuga canadensis* woods may indicate differences in the types of chemical constituents that are present in one wood type, but not the other (Fig. 1). These differences can be deduced from the chemical classes of VOCs that individual sensors in the array are known to be most sensitive to – as determined by direct comparison tests using single-chemical e-nose analyses. The organic chemical classes that individual sensors (within the A32S sensor array) are most sensitive to were determined and reported previously [23]. The strong positive differences between sensor output responses for sensors 1-3 indicate that *Q. alba* wood volatiles may contain short-chain alcohols, carboxylic acids, or aliphatic amines that are absent in *T. canadensis* wood volatiles. Similar deductions are possible to a lesser extend for sensors 4-16 with the exception of sensors 10-12. The strong negative difference between sensor output responses of sensors 20, 23, and 24 indicate that *T. canadensis* wood volatiles may contain long-chain alcohols, short- or long-chain esters, aliphatic ketones, or aromatic hydrocarbons that are absent in *Q. alba* wood volatiles. Further deductions also may be inferred for negative differences observed for sensors 25-32 with the exception of sensors 29 and 31.

C. Principal component analysis

An analysis of seven pine (*Pinus*) species using PCA by pairwise comparisons of headspace wood volatiles in all possible combinations provided greater details of chemical relatedness between species within a single woody plant genus. The results of relatedness of wood volatiles between these pine species were measured using a statistical algorithm called quality factor (QF) analysis that determines the distance between aroma profiles using Euclidean distance units of measurement. The greater the QF value determined from pairwise comparisons of volatiles, the greater the difference (or distance) between the aroma signature profiles of the two aromas being compared. In terms of statistical determinations, a QF value of 2.0 is roughly equivalent to a statistical difference at $P = 0.10$ level of significance. The relatedness among the seven pine taxa varied greatly based on Euclidean distance as indicated in Table II. QF values ranged from 0.1 to >70, indicating a very wide range of chemical relatedness between individual pine species. Among the seven species compared, a QF of 0.1 indicated a very close chemical relationship between *P. strobus* and *P. contorta*, whereas a QF >70 indicated a very wide difference between the volatile VOCs from woods of *P. palustris* compared with *P. lambertiana*. Relatively low levels of relatedness were found between *P. strobus*, *P. resinosa*, and *P. palustris* as a group. Intermediate levels of chemical

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TABLE I. DISTRIBUTION OF ELECTRONIC-NOSE AROMA CLASS COMPONENTS AMONG TWELVE CONIFER WOOD TYPES

Wood Type	Aroma Class Distribution (%)[a]											
	Wood Types (Plant species abbreviations)											
	Acon	**Cdec**	**Claw**	**Jvir**	**Locc**	**Pgla**	**Ppon**	**Pmen**	**Ssem**	**Tdis**	**Tocc**	**Thet**
Abies concolor	**79.7**	–	10.4	4.7	7.1	4.6	–	18.1	–	–	0.1	12.8
Calocedrus decurrens	–	**85.0**	7.1	–	–	1.3	5.3	–	6.9	–	5.5	12.8
Chamaecyparis lawsoniana	10.1	7.6	**88.8**	–	–	2.2	3.6	–	4.3	–	3.2	–
Juniperus virginiana	3.1	–	–	**94.3**	0.9	3.6	1.8	3.4	2.7	2.3	–	3.8
Larix occidentalis	9.0	–	3.5	1.1	**87.0**	–	8.5	14.9	8.1	1.5	5.5	–
Picea glauca	–	1.4	2.4	3.7	1.3	**91.0**	–	6.9	3.9	–	–	–
Pinus ponderosa	–	0.7	3.4	0.9	1.6	–	**95.8**	3.4	–	–	–	–
Pseudotsuga menziesii	11.5	–	–	–	6.8	2.7	0.1	**15.8**	–	6.3	7.1	4.9
Sequoia sempervirens	–	11.7	3.3	5.4	6.8	5.1	–	–	**86.9**	10.5	–	–
Taxodium distichum	–	–	–	–	0.9	3.3	3.0	14.7	8.4	**85.6**	6.5	7.8
Thuja occidentalis	0.6	4.9	–	–	4.4	3.0	0.9	10.9	–	12.8	**88.3**	–
Tsuga heterophylla	12.0	15.3	–	4.6	–	2.4	3.5	10.9	–	8.5	–	**80.9**

a. Mean percent aroma class distributions indicated for each wood type; read from left to right (by row), not top to bottom. Plant species abbreviations correspond to wood types (column 1).

relatedness were found between *P. strobus*, *P. monticola*, *P. lambertiana*, and *P. ponderosa*, but not between *P. monticola* and P. *lambertiana* that are fairly closely related chemically based on wood volatiles.

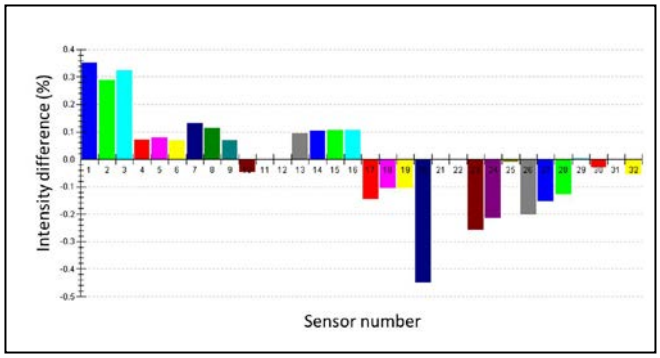

Figure 1. Percentage differences in e-nose sensor output intensity between wood volatiles of *Quercus alba* and *Tsuga canadensis* species.

The relatedness between aroma profiles of wood volatiles from the seven *Pinus* species, based on 3-dimensional CPA, was graphed in the form of an aroma map that indicates Euclidean distances among the seven pine species (Fig. 2). The percentages of the total variance for this analysis, accounting for the variability explained by each orthogonal principal component (PC), are as follows: PC 1 = 54.5%; PC 2 = 31.8%; and PC 3 = 13.4%, representing the x-, y-, and z-axis of the aroma map, respectively. A high proportion (86.3%) of the variation was explained by the first two principal components (PC 1 and PC 2). Notice that the data points for *P. strobus* are very close to one of the *P. contorta* points and indicate a close chemical relationship based on wood volatiles. *Pinus resinosa* is also fairly closely related to *P. strobus* and *P. contorta*. However, there are very large chemical differences between *P. lambertiana* and *P. palustris*, and between *P. palustris* and *P. contorta*.

IV. DISCUSSION AND CONCLUSIONS

An electronic-nose correct-identification rate above 90% generally is considered quite acceptable for unknown sample sizes greater than n=30 for any one aroma class. The rate of correct identifications can be significantly increased through neural net training to a higher level of specificity and thus lower error rate for e-noses that provide neural net training features in their operating software. The level of discrimination is largely determined by how long the neural net training is allowed to proceed before it is terminated. Specific levels of error rates may be specified and set via training parameters prior to training to reach specific target levels of discrimination for different applications. For example, sample types that have relatively small variations in aroma profiles must be trained to higher levels of discrimination to achieve effective differences between sample types. However, too much training specificity can result in the inability to determine distinctions between sample types. The level of discrimination desired for sample identifications is defined and specified by the specific reference aroma library used for sample identifications which are defined by the level of neural net training used to create each aroma library.

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TABLE II. RELATEDNESS OF SEVEN PINE (*PINUS* SPP.) WOOD TYPES BASED ON 3-DIMENSIONAL PCA OF WOOD VOLATILES

Aroma class	*Aroma class*	*QF value*[a]
P. strobus	*P. monticola*	17.5***
	P. lambertiana	15.3***
	P. resinosa	4.6*
	P. palustris	5.1*
	P. ponderosa	10.3**
	P. contorta	0.1
P. monticola	*P. lambertiana*	2.1
	P. resinosa	6.0*
	P. palustris	20.7***
	P. ponderosa	18.6***
	P. contorta	5.9*
P. lambertiana	*P. resinosa*	9.2**
	P. palustris	> 70****
	P. ponderosa	37.7
	P. contorta	3.8*
P. resinosa	*P. palustris*	4.2*
	P. ponderosa	22.2***
	P. contorta	5.3*
P. palustris	*P. ponderosa*	4.6*
	P. contorta	66.5****
P. ponderosa	*P. contorta*	14.9***

a. Quality factor significant difference levels between aroma classes: * = P < 0.05; ** = P < 0.01; *** = P < 0.001; **** = P < 0.0001. The percentages of the total variance, accounting for the variability explained by each orthogonal principal component (PC), are as follows: PC 1 = 95.26%; PC 2 = 4.27%; and PC 3 = 0.43%.

Because none of the wood sample identifications in this study were determined to be incorrect or ambiguous, the absence of false positives is an advantage for a diagnostic gas-sensing analysis method. Generally, e-noses are set to a level of specificity that preclude false positives and result in unknown determinations for samples that cannot be recognized or that have aroma profiles that are missing from the reference aroma library. The diagnostic specificity can be improved even further by building e-nose methods and libraries that are specific to particular sample types so that false positive determination are exceedingly rare.

The results of e-nose analyses of wood types in the current study were similar to those obtained in other related studies, but with fresh wood samples and similar or different e-nose technologies based on several different gas-sensing principles [28, 29]. Wilson et al. [28] identified and distinguished between twenty-three different angiosperm and gymnosperm wood types using fresh tree cores frozen at -20 C and thawed immediately prior to analysis with a A32S CP e-nose. Baietto et al. [29] utilized and compared the performance of three different e-nose instruments, including the PEN3 metal-oxide (MOS) e-nose, the LibraNose quartz microbalance (QMB) e-nose, and the Aromascan A32S CP e-nose to effectively discriminate between different healthy wood types and wood decayed by various wood-rot fungi.

Aroma data profiles from e-nose analyses that provide some indications of chemical relatedness between plant species may be a new tool and means for studying chemotaxonomic relationships between woody plants based on their wood volatiles as well as relationships between non-woody plants based on leaf, stem, or floral volatiles.

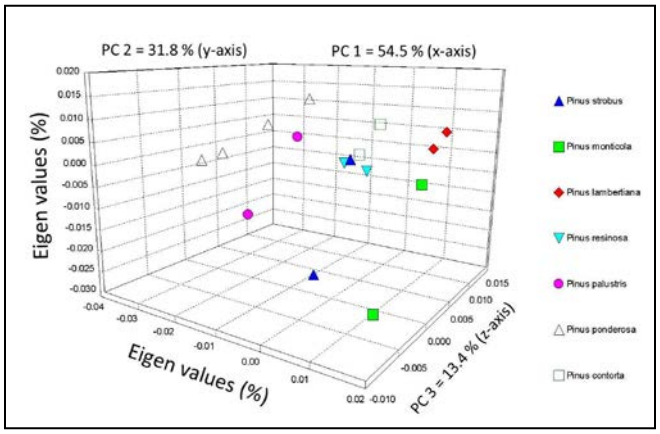

Figure 2. Aroma map showing the relatedness of wood volatiles from seven *Pinus* spp. using conductive polymer analysis (CPA).

Some possible reasons why the *P. menziesii* wood type could not be identified may include insufficient wood volatiles for analysis, the lack of sufficient principal components in adequate quantities to make up a representative aroma signature profile for this particular wood type, or the presence of particular wood VOCs to which the A32S e-nose sensors were not sufficiently sensitive, thus unable to generate a distinctive pattern of sensory outputs. This problem would no doubt be resolved with a fresher sample, but applying moisture to the sample surface and building headspace volatiles longer might help generate enough wood VOCs for analysis.

This study has demonstrated that a CP e-nose has the capability of identifying and discriminating wood types even when wood samples are aged in storage for long periods of time. The critical treatment of aged woods during sample preparation that made e-nose analysis possible was to wet the surface of the wood for a minimum of 15 min (followed by blotting) to facilitate the release of wood volatiles. Without wetting the wood sample surfaces, insufficient volatiles were generated to build headspace for an effective e-nose analysis. However, the wetting procedure should have no effect on the accuracy of results as long as sufficient volatiles are released to produce a sensory output (pattern) from the sensor array.

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