DETECTING PLASTICS IN SEEDCOTTON

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Abstract

To increase global market share and value the US cotton industry needs to supply cotton lint that is free of contamination. Removing plastic contamination first requires developing a means to detect plastics in seedcotton. This study was conducted to validate a custom Ion Mobility Spectrometer (IMS) that will be used to find small amounts of plastic in a background of heated seedcotton. The custom IMS analyzer with membrane inlet sampled air displaced from a drying oven containing seedcotton alone or seedcotton and plastic samples. Vapors from plastics associated with cotton contamination were detected with the custom IMS analyzer. One to two minutes were required to achieve the maximum plastic signal in seed cotton; it took much longer for the signal to completely dissipate. IMS detection of plastics vapors in seedcotton is feasible in the laboratory. Developing an IMS system for commercial cotton gins may require additional seedcotton handling equipment.

Introduction

Ion mobility spectrometry (IMS) has been developed over the last thirty-five years to detect trace amounts of chemicals in air (Eiceman and Stone, 2004). Its principal advantage is that it works at atmospheric pressures and ambient temperatures. IMS trace analytical detectors using established principles of ion mobility in differential electrical fields have proven themselves in life-critical applications such as screening passengers' baggage for explosives at airports and detecting warfare chemicals on battlefields (Eiceman and Karpas, 2005). IMS variants have also been used to detect specific insect pheromones (Eiceman et al., 2001a) and to discriminate between smokes from various combustible materials found in a cotton warehouse (Eiceman et al., 2003).

Several IMS configurations exist; a typical analyzer draws sample air into the instrument where it is mixed with gaseous ions (formed by exposing analyzer internal atmosphere to beta particles, photons or corona discharge, Figure 1).

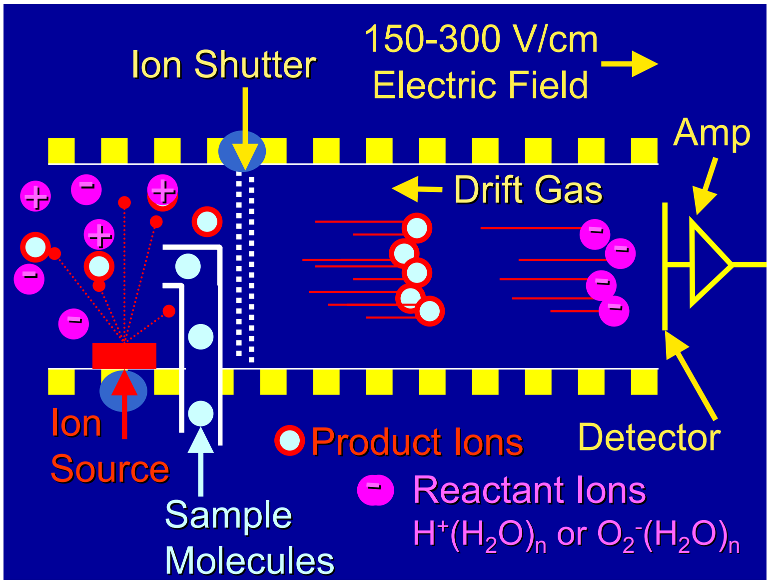

Figure 1. Basic operating principles of a typical IMS: Ionization of water vapor forms reactant ions which attach to sample molecules to form product ions. Ion clusters enter the drift tube through the shutter and move toward the detector plate under the influence of an electric field. Ions are neutralized when they exchange electrons with the

detector and are then swept from the drift tube by clean dry drift gas. Detector plate current is amplified and plotted against drift time (elapsed from shutter's opening) resulting in a spectra characteristic of ion size and shape (drift time; x-axis) and ion abundance (voltage; y-axis).

Associations between vapors and reactant ions result in product ions that are correlated with the chemical composition of the sample (Equation 1).
Equation 1: Ionization chemistry based on negative ions:

M	+	$O_2^-(H_2O)_n$	→	$M\ O_2^-(H_2O)_{n-x}$	+	xH_2O	(1)
Molecule from sample		Reactant anion		Product ion		water	

Product ions are introduced into the drift tube's electric field when the electric shutter opens (typically for about 200 to 300 microseconds) and then characterized on the basis of their drift speed. Electrons are exchanged when reactant and product ions collide with a detector plate at the far end of the drift tube. An amplifier converts the resulting electric current into a voltage representing ion abundance. This is plotted against the time elapsed between the shutter's opening and ion detection to form a mobility spectrum. The mobility spectrum characterizes a substance based on its product ions' terminal velocity in a gas. Terminal velocity is a function of product ion size and shape (wind resistance) as well as charge. A spectrum can be completed in ten to twenty milliseconds. Several spectra are accumulated for digital signal processing. The total time to make a measurement can be one second or less. Comparison to a library of spectra from known substances means chemical "fingerprints" can be identified even when sample molecule concentrations are as low as 100 to 10 parts per billion (Eiceman and Karpas, 2005). The custom IMS analyzer of this trial used these basic principles.

In a first exploratory study (Eiceman et al., 2002), the specific volatile organic chemicals (VOC) emitted by polypropylene-based tarp and seedcotton at 100 and 150°C in air and helium were determined using gas chromatography-mass spectrometry (5890A gas chromatograph, 5971A mass selective detector (Hewlerr-Packard, Palo Alto, CA) and 30m x 0.25 mm dia. capillary column, 50% phenyl, 50% methypolysiloxane df 0.25 µm (Restek, Bellefonte, PA)). The VOC emitted during polypropylene heating were primarily alkanes and alkenes with carbon numbers from 14 to 19. Specific species positively identified were: tetradecane ($C_{14}H_{30}$), hexadecane ($C_{16}H_{34}$), hexadecane ($C_{16}H_{32}$) heptadecane ($C_{17}H_{36}$), octadecane ($C_{18}H_{38}$), octadecene ($C_{18}H_{36}$), nonadecane ($C_{19}H_{40}$), and two unidentified aromatic hydrocarbons, all emitting from 0.7 to 9.3 µg g^{-1}. These appeared to arise both from simple thermal desorption and from oxidative decomposition. The VOC emitted during heating of seedcotton were eicosane ($C_{20}H_{42}$), henicosane ($C_{21}H_{44}$) and four unidentified hydrocarbons, all emitting from 0.21 to 0.31 µg g^{-1}. Thus polypropylene VOC differed from seedcotton VOC and were formed in quantities typically an order of magnitude greater, suggesting that an IMS vapor sensor approach to detecting plastics in seedcotton is feasible.

Objectives

Validate a custom Ion Mobility Spectrometer (IMS) that will be used to find small amounts of plastic in a matrix of heated seedcotton. Determine limits of detection, sensor response time and time required for signal to clear.

Materials and Methods

Instrument

A custom research ion mobility spectrometer was fabricated with a large (7 in^2 (46 cm^2)) polymethylsilicone membrane inlet to allow greater numbers of sample molecules to diffuse into the ionization region while excluding most water molecules. Ionization was accomplished with 10 mCi ^{63}Ni foil. The ¾ inch (19 mm) diameter by 2 inch (51 mm) long drift tube had an electric ion shutter at one end and a Faraday plate detector at the other. It was operated with the electric field strength approximately 200 V cm^{-1}. Clean dry air from a Whatman International (Maidstone, Kent, UK) model 76-803 Zero Air Generator was used for drift gas. Drift gas flow was maintained at 0.325 l min^{-1} by a Fathom Technologies (Round Rock, TX) GR116 mass flow controller. The drift tube was heated to 270°F (132°C) to prevent condensation of sample molecules on exposed surfaces; drift gas temperature was 212°F (100°C). A slight vacuum assisted with sample molecule diffusion across the membrane.

Supporting electronics were provided in three boxes containing shutter control, high voltage power supply and the 10^{11} current amplifier, all interfaced through a National Instruments (Austin, TX) PCI 6024 E data acquisition card to a personal computer running National Instruments (Austin, TX) LabView 6.0 software customized for this application. More details including dimensions of key components and electric field potentials are available in Eiceman et al. (2001b).

The custom IMS analyzer sampled air diffusing through the top vent of an 0.11 m^3 insulated stainless steel drying oven (Fisher Isotemp model 338F) maintained at 220°F (104°C) containing 1 kg seedcotton alone or 1 kg seedcotton and up to 10 g plastic samples. Thirty IMS spectra (1.45 seconds of data) were averaged by LabView's digital signal processing software to reduce noise. Each average spectrum was recorded continuously for ten minutes, resulting in over 400 average spectra per trial. Figure 2 shows the IMS and most of its supporting hardware.

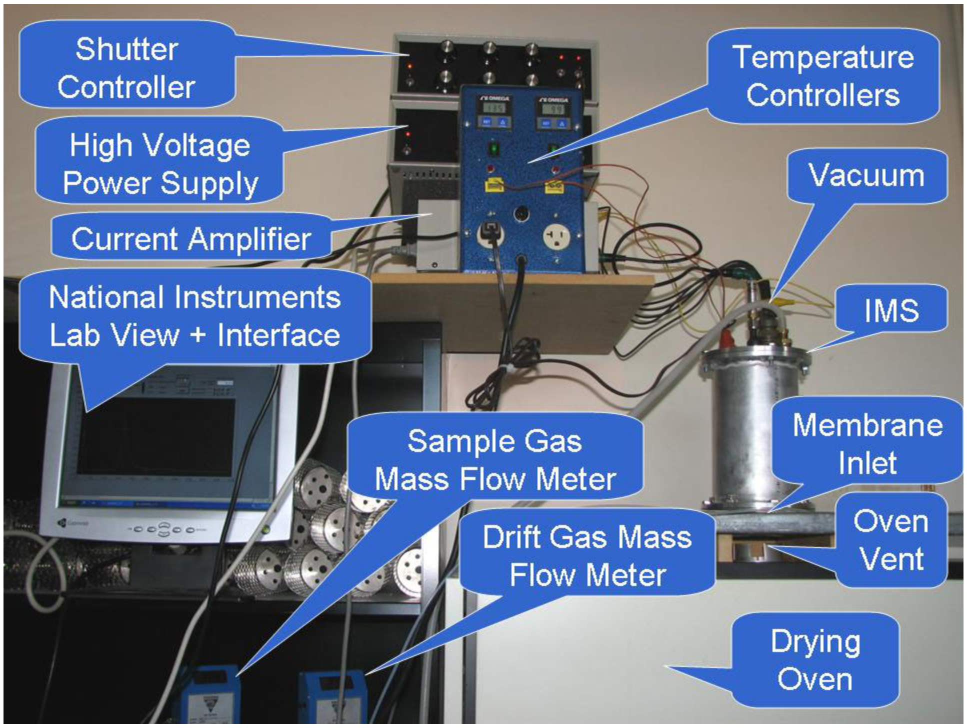

Figure 2. Experiment apparatus: Drying oven to warm seedcotton and plastics that it may contain; Ion Mobility Spectrometer in stainless steel canister with electronics to control temperature, shutter opening and drift field voltage; mass flow meters to supply clean dry air for drift tube and sample molecule conveyance; and current amplifier and computer interface to display and record results.

Results and Conclusions

Vapors from plastics associated with cotton contamination were detected at 7.6 and 9.2 mS drift time (Figure 3) though one to two minutes were required to achieve maximum ion intensity for the plastic signal in seed cotton. Product ions persisted at 7.6 mS for over an hour, but dissipated rapidly at 9.2 mS.

IMS detection of plastics vapors in seedcotton is feasible in the laboratory. Developing an IMS system for commercial cotton gins may require additional seedcotton handling equipment. An accumulator such as a steady flow device downstream from the seedcotton dryer may provide enough dwell time for signal acquisition and processing to take place on-line in a commercial gin.

Acknowledgement

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Disclaimer

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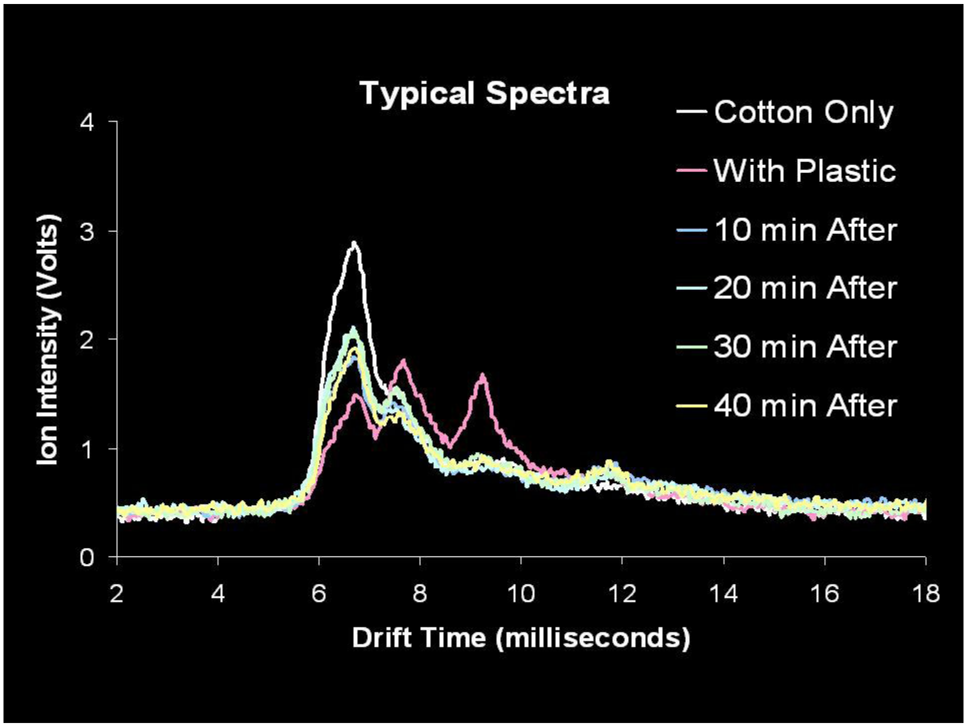

Figure 3. The reactant ion peak (6.7 mS) remained depressed long after removal of the plastic sample. Reactant ions continued to form product ions with molecules from the plastic sample that lingered in the drying oven (6.7 and 11.8 mS). However, a distinctive peak for plastic at 9.2 mS drift time returned to previous levels immediately.

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