A New Palladium-Based Ethylene Scavenger to Control Ethylene-Induced Ripening of Climacteric Fruit

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A novel palladium-promoted zeolite material with a significant ethylene adsorption capacity at room temperature is described. It was characterised by diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) and transmission electron microscopy (TEM) to show palladium particles dispersed over the support. Initial measurements of the ethylene adsorption capacity were conducted with a synthetic gas stream at a higher ethylene concentration than would normally be encountered in fruit/vegetable storage, in order to obtain an accelerated testing protocol. Further laboratory-based trials on fruit samples show that the palladium-promoted zeolite material can be effective as an ethylene scavenger to prolong the shelf-life of fresh fruits.

Introduction

Ethylene is one of the simplest plant growth regulators and is known to play a role in many physiological processes in plants. Seed germination and growth, abscission, fruit ripening and senescence can all be affected by ethylene (1–4). It is believed that the ancient Egyptians and Chinese were aware of some of these effects and attempted to artificially control the ripening of figs and pears. More recently, in the nineteenth century, it was observed that street lighting (fuelled by town gas which contained low levels of ethylene) was causing stunting and other changes to nearby plant growth (2). Today, although it is well documented that almost all plants produce ethylene in varying amounts, understanding how ethylene interacts with each plant type and the effect this has on plant development is a very active area of scientific research.

The ability to control ripening is very important to the sale of many fresh produce types. Whilst consumers often want to purchase food that can be eaten straight away, suppliers may prefer to market fruit in need of further ripening, to avoid losses due to over-ripening in transit or storage. Therefore, the control of the ambient ethylene concentration is key to prolonging the shelf-life of many horticultural products.

Various methods of ethylene control are offered commercially, including several based on ethylene adsorption/oxidation. However, these technologies have seen limited uptake, especially in retail packaging and fresh produce transportation. In the present paper, we report on the development of a new palladium (Pd)-based ethylene adsorber, which works effectively both in synthetic gas streams and in laboratory-based trials using real fruit samples. We are now in a position to undertake 'real-world' testing.

Ethylene Production by Fresh Produce

Fresh fruit and vegetables are generally classified in one of two ways, depending upon the mechanism of ripening (5) and their capacity to produce ethylene (Table I). Climacteric items release a burst of ethylene during ripening, accom-

Table I			
Ethylene Production from Different Fresh Produce Types			
Low (< 1.0 ml kg^{-1} h^{-1})	Moderate (1–10 ml kg^{-1} h^{-1})	High (10–100 ml kg^{-1} h^{-1})	Very high (> 100 ml kg^{-1} h^{-1})
Pineapple, artichoke, cauliflower, broccoli, date, orange, rhubarb, spinach, beetroot, green asparagus, celery, lemon, onion	Banana, mango, plum, tomato	Apricot, nectarine, pear, peach	Apple, avocado, cherimoya, passion fruit

panied by an increase in respiration, whereas non-climacteric produce do not vary their rate of ethylene production in this fashion. Exogenous ethylene (typically < 0.1–1.0 µl l^{-1}) can initiate ripening in many climacteric fruit which then can lead to autocatalytic production of ethylene by the fruit. Climacteric fruits, which include bananas, avocados, nectarines and pears, ripen after harvest, typically by softening, changing colour and becoming sweeter (6). The latter class, including strawberries, grapes and pineapples do not ripen as dramatically after harvest, but rather senesce, leading to discolouration, unpleasant odour, shrinkage and general rotting. In such cases, the challenge to maintain product quality is paramount. Other fresh produce types are sensitive to ethylene even though their ethylene production is very low for example, potato tubers, bulb onions, broccoli and spring cabbage, along with some cut flowers.

To artificially reproduce the natural ripening process, ethylene can be introduced during storage. This process is used in the fruit industry on fresh produce such as bananas, avocadoes and mangoes (7). As well as such deliberate exogenous introduction, the level of ethylene can rise due to ethylene emission from stored fresh produce or from accidental sources such as forklift truck exhaust emissions. For this reason, the industry prefers to use electric vehicles in fresh food storage areas to minimise the risk of the fresh produce coming into contact with ethylene gas.

Excessive or uncontrolled levels of ethylene can result in a number of problems. For example, the premature ripening of fruits and vegetables, the fading and wilting of cut flowers and the loss of green colour and increase in bitterness of vegetables are common problems when ethylene levels have not been properly managed.

Ethylene-Sensitive Plants

Plants, flowers and buds are also sensitive to ethylene (see Table II for examples) and some release ethylene gas when they are cut or damaged. Some of the most detrimental effects of ethylene on plants are:

– Partial or incomplete flower abortion;
– Retarded plant growth;
– Growth abnormalities such as excessive leafiness or the stimulated growth of daughter bulbs;
– Shortened vase lifespan of cut flowers (abscission of leaves and flower petals);
– Inhibited development of immature (unopened) flower buds;
– Accelerated senescence of all types of plants;
– Susceptibility to disease.

Ethylene Removal Technologies

In many instances, ethylene concentrations can be controlled by ventilation of the area containing

Table II		
Different Degrees of Ethylene Sensitivity amongst Cut Flower Species		
Low Sensitivity	Moderate Sensitivity	High Sensitivity
Tulip, daffodil	Lily, freesia, agapanthus, alstroemeria, anemone, dahlia	Carnation, Geraldton waxflower

the fresh produce. This can be aided by container designs that allow for air circulation. However, ventilation is not always appropriate; for example, it cannot be used in sealed environments, such as controlled atmosphere storage or retail packaging. There are a number of ethylene removal technologies available:

– *Catalysts.* Often based on platinum/alumina, these operate at elevated temperature (> 200ºC) and catalytically oxidise ethylene to carbon dioxide (CO_2) and water (8). There are also reports of the use of photocatalytic oxidation of ethylene using titanium dioxide (TiO_2), which can occur at room temperature (9).

– *Stoichiometric oxidising agents.* Mostly based on potassium permanganate ($KMnO_4$), which again oxidises ethylene and is itself reduced. Though some CO_2 and water is produced, some partially oxidised species such as carboxylic acids may also be formed.

– *Sorbents.* These materials work by sorption of the ethylene and are often based on high surface area materials, including activated carbon, clays and zeolites.

Ethylene Blocking Technologies

A different approach is to inhibit ethylene action in the produce itself, which can in turn reduce the amount of ethylene released by the produce into the container or storage area. Several chemicals have been shown to act as ethylene inhibitors, including both volatile and aqueous treatments:

– *1-Methylcyclopropene (1-MCP).* 1-MCP is the most widely used commercial volatile ethylene inhibitor, which blocks ethylene binding sites. It is applied exogenously as a gas and has been widely applied to fruit (particularly apples) and flowers. 1-MCP is sold commercially as SmartFresh[SM] (10) into the fresh produce industry and as EthylBlock[TM] into the floral industry.

– *Silver thiosulfate (STS).* The use of this material is largely restricted to cut flowers and it is sold commercially under the trade name Chrysal AVB[®]. It is applied by putting the cut flower stems in a solution containing the STS.

– *Aminoethoxyvinylglycine (AVG).* This material is sold commercially as ReTain[®] and acts as a plant growth regulator by blocking the production of ethylene in the plant tissue. It is sprayed onto the fruit, usually 1 to 3 weeks prior to harvesting.

New Palladium-Based Ethylene Scavenging Technology

In this paper, we report on the discovery by Johnson Matthey scientists of a novel palladium-promoted material with a significant ethylene adsorption capacity at room temperature (11). A wide range of materials were synthesised and screened for activity. Pd gave by far the best performance of the promoter metals tested. The material is a palladium-impregnated zeolite giving finely dispersed palladium particles.

Initial testing was conducted with a synthetic gas stream at a higher ethylene concentration than would normally be encountered in fruit/vegetable storage, in order to obtain an accelerated testing protocol for measuring ethylene adsorption capacity. Ethylene adsorption capacity measurements were carried out at room temperature (21ºC) in a plug flow reactor using 0.1 g of active Pd-based material with a gas composition of 200 µl l⁻¹ ethylene, 10% (v/v) oxygen balanced with helium, at a flow rate of 50 ml min⁻¹, with and without *ca.* 100% relative humidity (RH). Reactor outlet gas concentrations were analysed using a Thermo Onix ProLab mass spectrometer (Thermo Onix, Houston, Texas, U.S.A.). A mass:charge ratio of either 26 or 27 was used for ethylene, as the use of nitrogen as a diluent gas leads to the presence of a large peak at $m/z = 28$. A value of $m/z = 44$ was used for CO_2. Ethylene uptake capacity was measured using a simple 'breakthrough' measurement, in which the total integrated ethylene removal was determined after the outlet ethylene concentration from the reactor had reached the inlet ethylene concentration, showing that the adsorber was saturated with ethylene (12).

The Pd-based material typically removed all measurable ethylene until breakthrough occurred. A typical example of an ethylene breakthrough

measurement under humid conditions is shown in Figure 1. Under these conditions the Pd-promoted material was found to have an ethylene adsorption of 4162 µl g^{-1} under *ca.* 100 % RH. This performance was increased to 45,600 µl g^{-1} under dry conditions.

Further experiments were carried out at room temperature in a sealed, unstirred batch reactor (0.86 l) with 0.1 g active Pd-based material and an initial gas composition of 550 µl l^{-1} ethylene, 40% (v/v) air balanced with argon. Selected gas concentrations were measured at hourly intervals with a Varian CP-4900 Micro-GC (Varian, Inc, Palo Alto, California, U.S.A.). Gas samples (40 ms duration) were taken *via* an automated recirculating sampling system. Column and injector temperatures were set at 60ºC and 70ºC, respectively. The 0.15 mm diameter, 10 m long column was packed with PoraPLOT (porous layer open tubular) Q stationary phase. Ethylene and CO_2 were calibrated against 10 µl l^{-1} ethylene balanced with air and 5% (v/v) CO_2 balanced with Ar (Air Products Europe, Surrey, U.K.). A thermal conductivity detector was used with He carrier gas at 276 kPa inlet pressure. Peak integration was carried out within the Varian STAR software.

Under these conditions, CO_2 and ethane production were observed, as plotted in Figure 2. The ethane is likely to be produced by the hydrogenation of adsorbed ethylene, the hydrogen being generated by the partial dissociation of ethylene.

The selectivity to ethane varies with experimental conditions but is typically no more than the maximum value of ~ 10% observed under the conditions tested here. Ethane can be produced by plants in response to stress (13) and has not previously been reported to be detrimental to plants in the concentration range reported here. It is clear, however, that the palladium-based material is acting largely as an adsorber rather than as a catalyst. The mechanism of reaction is discussed further in this article by interpretation of diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) data.

Characterisation of the palladium-based material was carried out to determine the Pd distribution through the support. Transmission electron microscopy (TEM) analysis (Figure 3) showed that the palladium particles (bright particles) are dispersed over the support material. The average size of the palladium particles in Figure 3 was calculated to be 1.7 nm. Scanning electron microscopy (SEM) analysis (not shown) also identified some larger palladium particles with diameters around 20 to 40 nm. These results correspond well with CO chemisorption data which gave a metal dispersion of ~ 15 %. From this dispersion value, a slightly larger average size of the palladium particles would be expected. This is consistent with the observation of many small and some larger particles by TEM (Figures 3 and 4) and SEM.
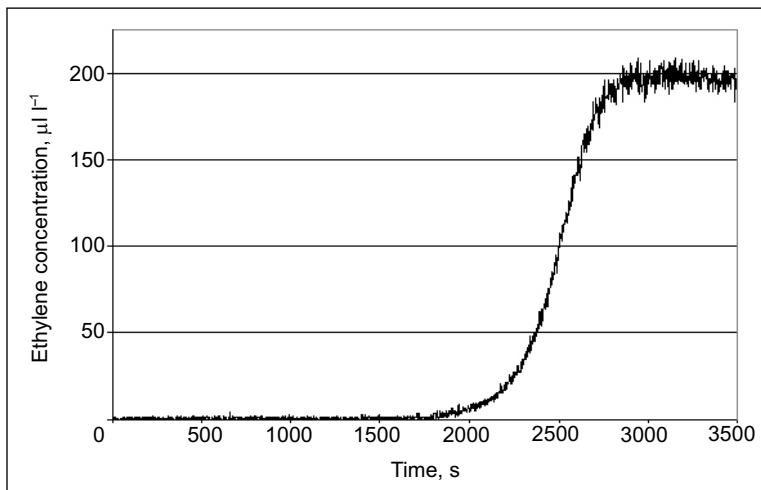

Fig. 1 Ethylene breakthrough graph of the Pd-based material in humid gas conditions. Ethylene was monitored using the mass 26 signal on a mass spectrometer (mass 26 and 27 gave very similar breakthrough profiles)
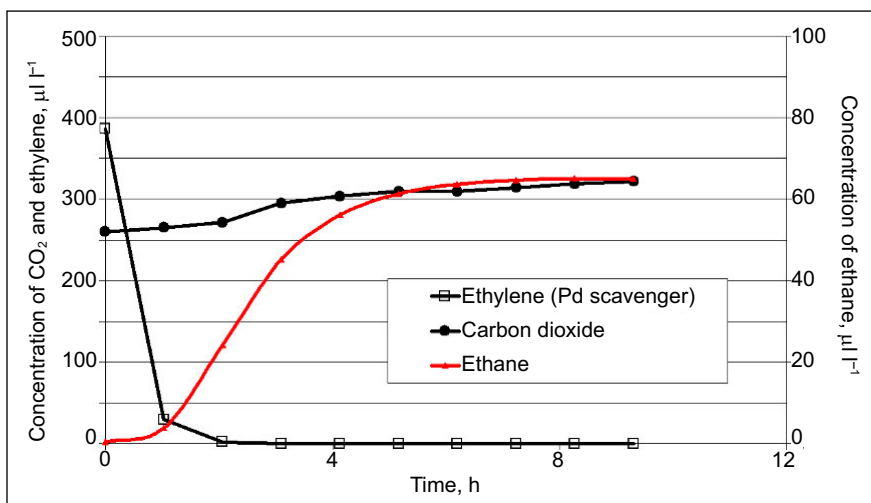
Ethylene–Metal Interaction *via* DRIFTS Analysis

In order to gain further information regarding the processes involved in ethylene removal, a vibrational spectroscopic study was performed to probe the interactions between the ethylene and the Pd-promoted scavenger. DRIFTS was used to characterise the species adsorbed on the ethylene scavenger after exposure to ethylene, in the presence and absence of oxygen and water vapour.

DRIFTS allows spectra to be obtained of powdered samples in the presence of gaseous atmospheres. It produces infrared spectra of the diffusely scattered radiation in the reflectance mode (Figure 5), which are analogous to those collected in the more conventional transmittance mode. Disadvantages of this method include the appearance of artefacts resulting from the collection of specularly reflected radiation. It is also difficult to perform quantitative studies, since both scattering and absorption coefficients must be considered. By contrast, only the latter is required to quantify adsorbed species when spectra are collected in the transmittance mode and converted to absorbance (14). The key advantages of this method are that no sample preparation is necessary and that the cells can be operated in plug flow mode with the reactant gases being forced to travel through the bed of powdered scavenger material.

Commercial DRIFTS cells are readily available which allow the collection of spectra of powdered samples at ambient or elevated temperatures, while controlling the composition of the gaseous atmosphere. In the study performed here, the outlet port of the DRIFTS cell was coupled to a quadrupole mass spectrometer (QMS) *via* a heated, glass-lined capillary. This permitted continual monitoring of ethane ($m/z = 30$), carbon dioxide ($m/z = 44$) and ethylene (a value of $m/z = 27$ was selected for this experiment) during exposure to ethylene and during subsequent temperature-programmed desorption (TPD) measurements. Experiments were performed by exposing samples
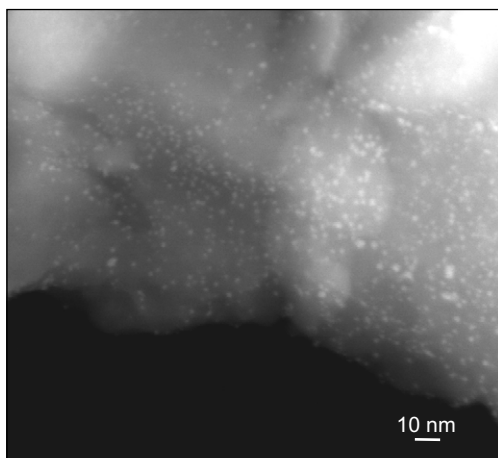

Fig. 3 TEM image of the Pd-promoted zeolite material showing nanometre size palladium particles (bright areas) on the zeolite support
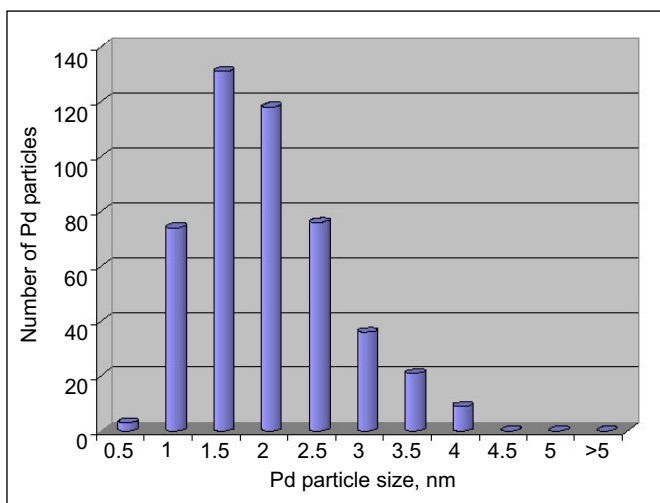
with and without Pd, at ambient temperature, to a flow of ethylene in nitrogen with the addition of either no other gases, air or air and water. After a period of exposure to these gaseous atmospheres, the ethylene stream was switched off and a TPD measurement was performed in the presence of the remaining gases. Spectra were also obtained of samples that had been exposed to ripened fruit.

The Fourier transform infrared (FTIR) spectrum of ethylene in nitrogen flowing through the cell, in the absence of scavenger, gave main features at 3112 cm^{-1} (ν_9, νCH asym.), 2992 cm^{-1} (ν_{11}, νCH sym.), 2048 cm^{-1} ($2 \times \nu_{10}$, γCH$_2$ rocking), 1889 cm^{-1} ($2 \times \nu_7$, δCH$_2$ out-of-plane) and 1446 cm^{-1} (ν_{12}, δCH$_2$ in-plane). The absence of bands for the IR inactive (totally symmetrical) modes at 1623 cm^{-1} (ν_2, νC=C) and 1343 cm^{-1}

(ν_3, δCH$_2$ in-plane) should be noted, although it is expected that any interaction with the adsorbent might permit these modes to be detected as the symmetry is lifted. These interactions might also be expected to shift the frequencies away from the gas phase values listed above.

FTIR spectra of a sample exposed to fruit (Figure 6(a)) showed features at 1467, 1460, 1434 and 1382 cm^{-1}. FTIR spectra of the Pd-free zeolite showed only very weak features, including a band at 1439 cm^{-1} which could be assigned to the presence of adsorbates resulting from exposure to ethylene (Figure 6(c)). However, these features were absent following heating to 50°C in air. The narrow shape of the feature at 1439 cm^{-1}, and the appearance of a single rather than a double component (as in the gas phase), confirms that this feature can be assigned to an adsorbed state, although the limited shift ($\Delta\nu = 7$ cm^{-1}) with respect to the gas phase feature (due to the ν_{12}, δCH$_2$ in-plane) would suggest a very weakly bound state. This was confirmed by the disappearance of bands at this frequency when the sample was heated in air to 50°C. Note that this ease of removal was not the case for the sample containing Pd (Figure 6(b)). This, along with the detection of other infrared bands (at 1467 and 1382 cm^{-1}) when Pd was present, would indicate that the metal played a significant role in the retention of the adsorbate and in the dominant adsorbed state. The relative intensities of the features indicate that the
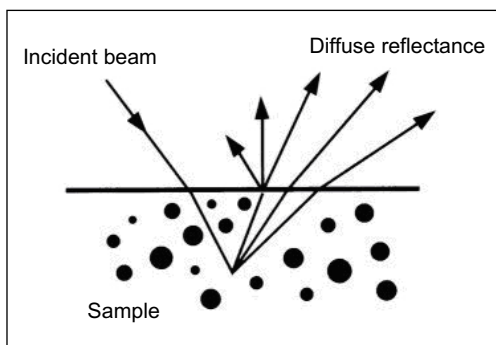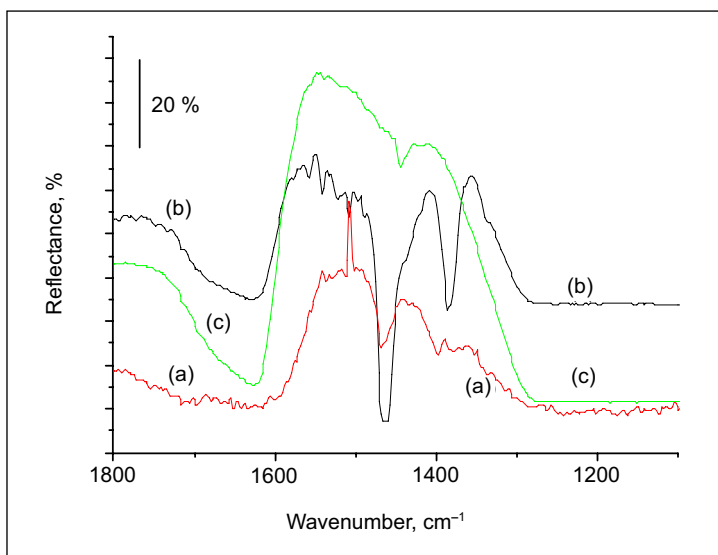
Fig. 6 DRIFT spectra of:
(a) the Pd-promoted zeolite material following exposure to fruit,
(b) the Pd-promoted zeolite material exposed to a flow of ethylene/nitrogen at 25ºC,
(c) Pd-free zeolite exposed to ethylene in nitrogen at 25ºC

presence of Pd was essential in order to achieve the 45,600 µl g^{-1} adsorption capacity obtained under dry conditions. The similarities between the features resulting from exposing the sample to fruit (Figure 6(a)) and to exogenous ethylene, in particular the features at 1467 and 1382 cm^{-1}, are strong evidence that similar adsorbed species exist in both cases. Under these conditions, the C–H stretching region gave two dominant bands at 2969 and 2865 cm^{-1}, which are consistent with expectations for CH$_3$ species (Figure 7).

The transformation from C$_2$H$_4$ to adsorbed species containing CH$_3$ would be consistent with the development of ethylidyne (CCH$_3$) species (15, 16). In these species, the carbon forms three bonds to the surface metal atoms (Figure 8(c)), probably on three-fold hollow sites of Pd(111) type facets (17). Such an assignment would be consistent with the detection of the 1467 and 1382 cm^{-1} band pair (Figure 6) arising from the corresponding CH$_3$ deformation modes. The formation of a hydrogen atom for each ethylidyne species generated would
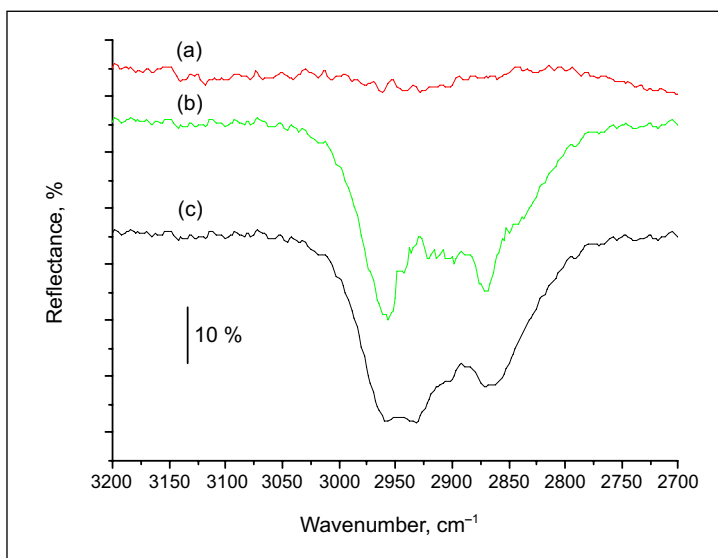

Fig. 7 DRIFT spectra of the Pd-promoted ethylene scavenger:
(a) before exposure to ethylene,
(b) after exposure to a flow of ethylene in nitrogen at 25ºC,
(c) after exposure to a flow of ethylene in air at 25ºC

explain the formation of ethane following initial exposure of the scavenger to ethylene (Figure 2), which was observed in the mass spectrometry (MS) trace ($m/z = 30$) recorded during the FTIR measurements. The surface selection rule states that vibrations parallel to a metal surface should not be detected, due to the creation of an opposing image dipole on the metal. This means that the νCH_3 asymmetric and δCH_3 asymmetric modes at 2969 and 1467 cm^{-1} should be absent from the spectrum. However, this rule will be relaxed for very small metal crystallites of the order of 2 nm and below and where extended flat facets are absent due to the presence of steps and edges.

Such a description of the morphology is consistent with the TEM image presented (Figures 3 and 4). This assignment cannot be confirmed from the expected absorption at around 1130 cm^{-1} ($\nu C-C$), due to absorption by the support in this region. Bands due to ethylidyne were diminished in spectra recorded after heating the sample in nitrogen to 150°C. If air was present during this thermal treatment, then CO_2 was detected by MS as a complete oxidation product. Below this temperature, FTIR evidence for partial oxidation included the detection of carboxylate type species and a band at 2125 cm^{-1} due to CO adsorbed at surface oxidised Pd sites. The latter was detected in spectra of the sample recorded at 100°C, indicating that the onset of oxidation took place well below the temperature of complete oxidation.

An MS trace was recorded during the collection of spectra with exposure to ethylene. This showed a breakthrough shape which was strongly dependent on the composition of the gas phase, with the greatest removal of ethylene occurring during treatment in nitrogen. The presence of air or air and water reduced the adsorption capacity. Additionally, the presence of air or air and water modified the predominant modes of adsorption, with four bands now appearing in the C–H stretching region. In addition to the pair at 2957 and 2865 cm^{-1} which have already been assigned to the CH_3 modes of ethylidyne in the absence of air or water, a further pair at 2934 and 2853 cm^{-1} were detected, although these were very weak when water was present. These features are consistent with expectations for CH_2 groups, although the vibrational frequencies are relatively low for vinyl species. Di-σ species of adsorbed ethylene (Figure 8(b)) give lower frequency CH modes than π-C_2H_4 (18) (Figure 8(a)), although the former are less favoured in the presence of adsorbed oxygen (18). The presence of oxygen in the system also led to the appearance of a species giving a maximum at 1514 cm^{-1}, which was absent for the air-free system.

A similar feature was found at 1510 cm^{-1} in electron energy loss spectroscopy (EELS) spectra of ethylene on oxygen covered Pd(100). This was assigned to δCH_2, so it would be tempting to assign the surface species to vinyl intermediates such as $HCCH_2$ (17). However, an alternative assignment, consistent with the known stepwise dehydrogenation of ethylene (19), is that the additional maxima at 2934 and 2853 cm^{-1} represent vibrations due to the CH_3 groups of ethylidene ($CHCH_3$), where the expected full conversion to ethylidyne, observed in the absence of air and water (18), is hindered due to the presence of the co-adsorbates which limit the activation and dissociation of the C–H bond. The lesser extent of C–H dissociation at room temperature, and subsequently the lesser population of the surface by adsorbed hydrogen, would explain the reduced
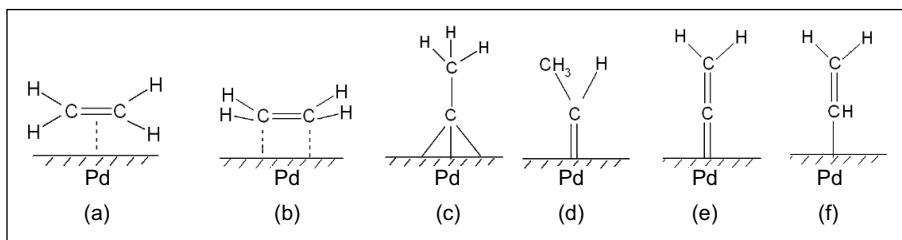

Fig. 8 Potential adsorbed species following exposure of the Pd-containing scavenger to ethylene: (a) π-bonded, (b) di-σ bonded, (c) ethylidyne, (d) ethylidene, (e) vinylidene and (f) vinyl

ability of the surface to liberate ethane resulting from ethylene hydrogenation during the initial exposure stages.

In summary, the surface speciation is somewhat dependent on the gas mix and the moisture content. However, the DRIFTS study has identified the key role of the palladium–ethylene interaction and the benefit that this has on binding ethylene to the zeolite surface.

Fresh Produce Research

The use of platinum group metals including palladium in different forms and on different supports for ethylene removal from fruit and vegetables has been investigated by other authors in the past (20, 21). Bailén *et al.* at University Miguel Hernández have recently published results on delaying tomato decay using a combination of controlled atmosphere packaging and a granular-activated carbon (GAC) or GAC impregnated with a palladium-based catalyst (22). Martínez-Romero *et al.* at the same University have also reported the use of a cartridge heater device (optimally running at 175ºC) joined to activated carbon containing palladium for ethylene removal above room temperature (23).

In contrast, the new material described here consists of a specific combination of a precious metal with a zeolite and removes significant amounts of ethylene at low temperature (5ºC) and room temperature. In order to test the material under realistic conditions, the active material has been evaluated in collaboration with the Plant Science Laboratory at Cranfield University, U.K. Bananas were included in the initial studies, as the role of ethylene in initiating ripening in these fruits

is well documented (24, 25). Initial findings, now published (2), have demonstrated for the first time that the presence of a palladium-based scavenger was effective at removing ethylene to below physiologically active levels for preclimacteric green bananas and green avocado fruits.

Reduced CO_2 production and control of the colour change from green to yellow was observed for the preclimacteric bananas (Figure 9). The palladium-promoted ethylene scavenger was also found to be far superior to a $KMnO_4$-based ethylene adsorber when used in low amounts at high relative humidity. No adverse effects on fruit quality or subsequent ripening were observed after removal of the ethylene scavenger material.

Similar experiments were also conducted on avocados. Results showed that exogenous and endogenous ethylene concentration was reduced significantly with increasing amounts of the Pd-promoted material. In the presence of Pd-promoted material, ethylene was removed to below physiologically active levels. The effect of ethylene on the colour of avocado cv. Hass fruits in the presence or absence of the palladium material is shown in Figure 10. Fruit held in the presence of 100 mg or 1000 mg of the Pd-promoted material for three days were generally greener, and thus less ripe, than control fruit after seven to ten days (Figure 10).

Furthermore, when avocados were treated with ethylene and then subsequently held in the presence of the Pd-promoted material (1000 mg) after day 1, ethylene was removed to below physiologically active levels. Despite the climacteric phase having been initiated for these fruits, the
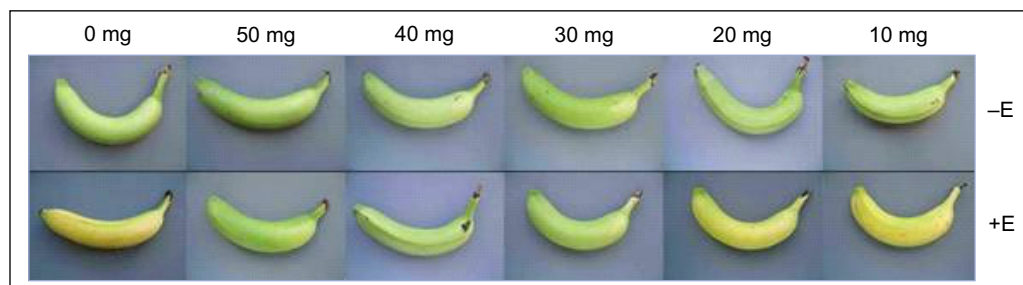

Fig. 9 Colour of a five-day-old banana cv. Cavendish fruit previously held for three days at 16ºC in 3 l sealed jars containing the Pd-promoted ethylene scavenger material (0–50 mg) and previously treated with (+E) or without (–E) 100 µl l⁻¹ ethylene when at the preclimacteric stage (i.e. green) at day 0
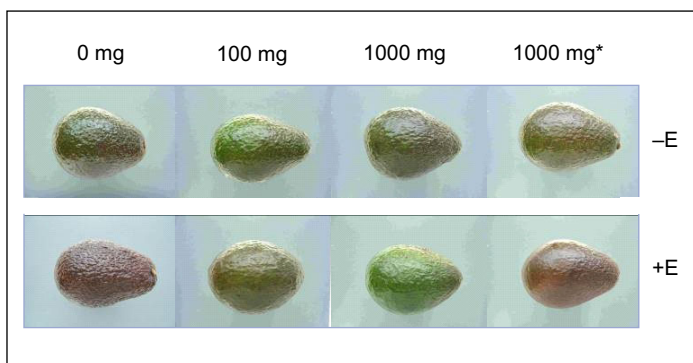
Fig. 10 Colour of a seven-day-old avocado cv. Hass fruit previously held for three days at 12ºC in 3 l sealed jars containing the Pd-promoted ethylene scavenger material (0, 100 or 1000 mg) and previously treated with (+E) or without (–E) 100 µl l⁻¹ ethylene when at the preclimacteric stage (i.e. green) at day 0

* The Pd-promoted material (1000 mg) was put into the jars after day 1, following treatment of the fruit with or without 100 µl l⁻¹ ethylene

subsequent total removal of ethylene resulted in better maintenance of fruit firmness as compared to controls. This suggests that the normal and expected climacteric respiratory rise has been disrupted. Therefore, for the first time an ethylene scavenger has been shown to be capable of extending shelf-life even when the climacteric respiratory rise has already been initiated.

Current work is focused on comparing the efficacy of the Pd-promoted ethylene scavenger to 1-MCP for the control of ripening in avocado fruit, and the resultant effect on non-structural carbohydrates and fatty acid methyl esters.

Conclusions

The results from this study demonstrate that the Pd-promoted ethylene scavenger described here is effective for the control of ethylene to prolong the shelf-life of climacteric fresh produce such as bananas and avocados. The material has the potential to be used commercially, as an alternative and/or supplemental treatment to 1-MCP. The technology does not require elevated temperature to remove ethylene, and the choice of zeolite support material makes it suitable for most fresh produce and floral applications under conditions of high humidity and low or room temperature. Future research will elucidate further uses of the Pd-promoted ethylene scavenger.

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