Wax Microemulsions and Emulsions as Citrus Coatings

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Citrus fruit was coated with polyethylene wax, petroleum wax, synthetic petroleum wax, carnauba wax, and candelilla wax emulsified with fatty acids and other FDA-permitted ingredients. Weight losses were low with coatings that contained hydrocarbon wax and for those waxes emulsified with stearic or palmitic rather than oleic acid. Oranges coated with wax had less weight loss, lower internal CO_2, higher internal O_2, and better water resistance than fruit coated with shellac or resin. Coatings formed on polymer films had proportionally higher resistance to water vapor when made with wax microemulsions rather than with mixtures of wax with shellac or wood resin.

INTRODUCTION

It is the practice in the citrus industry to apply coatings to fresh citrus fruit to reduce weight loss from transpiration (Kaplan, 1986). The goal of the present research was to develop wax microemulsion coatings that reduce shrinkage rate of coated fruit to levels below that presently achieved, with attention to how these coatings affect respiration and appearance of the produce, using only FDA-approved ingredients.

MATERIALS AND METHODS

Carnauba wax grades 1 and 3 and candelilla wax were from Strahl & Pitsch (West Babylon, NY). Microcrystalline wax was Be Square 195 from Petrolite (Tulsa, OK). Synthetic petroleum wax was polywax 500, also from Petrolite. Petroleum wax was P161 from Exxon (Houston, TX). Oxidized polyethylene samples were E10 and E20 from Eastman Chemicals (Kingsport TN) and also AC629, AC680, and AC316 from Allied Signal Inc. (Morristown, NJ). Properties of the waxes are summarized in Table 1. The shellac was dewaxed and bleached (R-49 from Mantrose-Haeuser, Westport, CT). The wood resin was a modified maleic product (807A from Resinall, Stamford, CT). The oleic acid was of NF grade (Mallinckrodt Specialty Chemicals, Paris, KY). The lauric acid was of Sigma grade; the myristic, palmitic, and stearic acids were grade II (Sigma Chemical Co., St. Louis, MO); and the stearic–palmitic blend (C16/C18) contained 54% palmitic and 44% stearic acid (Mallinckrodt). The morpholine was 99+% (Aldrich Chemical Co., Milwaukee, WI). Polysorbate 60 was Capmul POE-S (Capital City Products, Columbus, OH). The sorbitan monostearate was Durtan 60K (Durkee Foods, Louisville, KY).

Emulsions. Three microemulsion-making methods were used: water-to-wax, wax-to-water, and pressure. The water-to-wax (or inversion) method requires that water be added to the molten wax (Prince, 1977). Typically, 60 g of carnauba wax and 12 g of fatty acid (the emulsifier) were melted in a boiling water bath and morpholine was stirred in. Hot water (95–99 °C) was added, initially in about 3-mL increments, as the mixture was stirred with a propeller mixer sufficiently that added water was dispersed before the next water addition. The viscosity gradually increased, then decreased as the inversion point was passed, after which time more hot water was added to attain total solids content of 25–30%. The agitated microemulsion was cooled to 50 °C in a cold water bath, filtered through glass wool, and stored at 25 °C in a closed container. This method—long in use for making carnauba microemulsions (Eaton and Hughes, 1950) even before the term "microemulsion" was coined in 1958 (Prince, 1977)—was used for all ammonia-free carnauba emulsions made in our laboratory and also for the nonionic petroleum wax preparations.

The wax-to-water method involves addition of molten wax to hot water and is also a standard method of making microemulsions (Eastman Kodak Co., 1990). Typically, 100 g of wax and 15 g of fatty acid were heated to 128 °C and 15 g of morpholine was stirred in, which lowered the temperature of the molten wax to about 115 °C. The mixture of wax and morpholine or KOH (now at about 115 °C) was immediately poured (at about 100 mL/min) into the vortex of 350 mL of water (90–95 °C) being stirred with a propeller mixer. Rate of pouring and speed of mixing were adjusted so that the molten wax did not puddle on the surface of the water; otherwise, mixing speed was not critical. The stirred mixture was cooled to 50 °C in a cold water bath, filtered through glass wool, and stored at 25 °C in a closed container. The wax-to-water method was used for oxidized polyethylene or candelilla wax, except for formulations containing ammonia. The preparation containing both carnauba wax and candelilla wax was made by mixing a water-to-wax carnauba preparation with a wax-to-water candelilla preparation.

The direct pressure method is a commonly used industrial procedure for making microemulsions (Burns and Straus, 1965). Typically, 100 g of wax, 20 g of fatty acid, 12 g of 30% NH_3, and 270 g of water were heated to 135 °C for 15 min in a 1-L stainless steel cell (Parr Instrument Co.) with agitation at 200 rpm and then cooled by submerging the vessel in a water bath. This method was used for all preparations containing ammonia.

Four of the microemulsions used were from outside sources. An anionic carnauba microemulsion, M62125, was from Michelman Inc. (Cincinnati, OH). Three microemulsions containing oxidized polyethylene were from Allied Signal (Morristown, NJ) and Eastman Chemicals (Kingsport, TN) and are identified in the text.

Fruit. Purchased fruit used as industry control were from central Florida packinghouses. Other fruit were from central Florida groves maintained by the Florida Department of Agriculture. These were washed with rotating polyethylene brushes (type PSE, IBC, Eaton Park, FL), using a citrus cleaner

Table 1. Properties of the Wax Ingredients

identity and type of wax	melting or softening point (°C)	hydrocarbon content (%)	specific gravity[a]
carnauba[b]	82–86	1–3	1.00
candelilla[b]	66–71	40–60	0.98
P161 (petroleum wax)[c]	72	100	0.81
Polywax 500 (synthetic petroleum wax)[c]	88	100	0.93
Be Square 195 (microcrystalline wax)[c]	93	100	0.79
E10 (oxidized polyethylene)[c]	106	0	0.94
E20 (oxidized polyethylene)[c]	111	0	0.96
AC629 (oxidized polyethylene)[c]	102	0	0.93
AC680 (oxidized polyethylene)[c]	110	0	0.94
AC316 (oxidized polyethylene)[c]	140	0	0.98

[a] At 15–25 °C. [b] Data from Bennett (1975). [c] Data on melting point and specific gravity from manufacturer.

containing sodium o-phenylphenate (Freshgard 5, FMC Corp., Lakeland, FL). Coating (diluted to 20% nonvolatile total solids) was brushed onto fruit with a paint pad (Shur-Line, Lancaster, NY). Exception: more dilute formulations were used for Figure 2 data. The amount of coating (wet weight) applied to the fruit was determined from weight of the fruit within 10 s before and after application of the coating. Foam formation during application to fruit was controlled by addition of 50 mg/kg poly(dimethylsiloxane) (type FG-10, Dow Corning, Midland, MI).

Weight loss was determined with samples of 20 fruit per treatment, stored at randomized locations in a well-ventilated room that was maintained at 20 °C and 75% relative humidity (RH). The fruit were placed five each on four trays and weighed four times over a 1-week period.

Internal CO_2 and O_2 values were determined after 1 week of storage at 20 °C. A syringe needle was inserted into the blossom end of the fruit, and internal gas was withdrawn from fruit submerged in water. The CO_2 concentration was determined with a gas chromatograph (Model 5890A, Hewlett-Packard, Avondale, PA) fitted with a 30 m × 0.53 mm i.d. polystyrene column (type GSQ, J&W Scientific, Folsom, CA) and a thermal conductivity detector. Column and detector temperatures were 35 and 120 °C, respectively; The carrier gas flow was 7 mL/min. The O_2 concentration was measured by passing 4 mL of the gas through an InPack Model 507 O_2 analyzer (Wilmington, MA) modified to function as a flow-through cell by removal of the sampling syringe.

Coating fracture was visually determined from appearance of fruit submerged overnight (15–20 h) in water and then air-dried for 2–4 h. Fractured coating gave the fruit a whitish blush appearance.

Gloss was determined at 60° to the vertical with a reflectometer (Model Micro-Tri-gloss, BYK Gardner, Silver Spring, MD). The unit was calibrated on a standard surface, the lens opening was reduced to 18-mm length, and gloss units (GU) were measured directly on the fruit surface.

Ethanol content was determined after storage of the fruit for 1 week at 20 °C. Juice was extracted (two trials, seven fruit per trial) and distilled to obtain 20 mL of condensate/100 mL of juice. Ethanol was determined in duplicate with the Model 5890A gas chromatograph, using a 50 m × 0.32 mm FFAP column (Hewlett-Packard) and flame ionization detector. Column temperature was 60 °C, detector and injector were both 250 °C, and He column flow rate was 3 mL/min.

Water vapor permeance of coatings applied to cellulose acetate was measured with the Permatran W1A water vapor permeability tester (Modern Controls, Minneapolis, MN). As previously described (Hagenmaier and Shaw, 1992) coating permeance was calculated from resistance of coated and uncoated film, three trials per coating. Mean coating thickness was 0.008 mm, and permeance was measured at 20 °C with 0% RH on the uncoated side, 85% RH on the coated side. The cellulose acetate was coated with a proprietary carnauba microemulsion (M62125); with resin, shellac, and oxidized polyethylene formulations containing 8%, 7%, and 15% oleic acid, dry basis, respectively; and with mixtures of these formulations. Results for each coating are based on permeance measurements of three samples of coated film.

RESULTS AND DISCUSSION

The wax formulations used in this study, except where indicated, were apparently microemulsions rather than oil-in-water emulsions because (a) the already-described procedures did not employ sufficient intensity of agitation to form microemulsions and (b), except where noted, the formulations were translucent to clear, with amber to brown color—rather than white and opaque as expected of oil-in-water emulsions (Prince, 1977).

The water vapor resistance (r) of a carnauba wax coating formed on plastic film was markedly higher than that of shellac, wood resin, or oxidized polyethylene or of mixtures of these substances with carnauba wax (Figure 1). Resistances of shellac–wax resin–wax, and polyethylene–wax coatings were low in part because these mixtures did not make efficient use of the carnauba wax; resistances were only about 60% of the values expected from one layer of carnauba wax and a separate layer of shellac, resin, or oxidized polyethylene (Table 2).

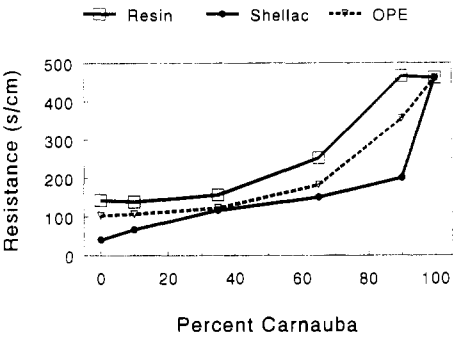

Figure 1. Resistance to water vapor of coatings made from carnauba wax alone (100% carnauba) or mixed with wood resin (squares), shellac (circles), or oxidized polyethylene (triangles). SE = 22 s/cm.

Table 2. Water Vapor Resistance of Coatings with Carnauba Wax as Ingredient of Mix or as Separate Layer

mix formulation		r (s/cm)		
% carnauba[a]	type of adjunct	mix[b]	layer[c]	ratio r(mix)/r(layer)
35	resin	157	255	0.62
65	resin	253	350	0.72
35	shellac	117	189	0.62
65	shellac	151	314	0.48
35	OPE[d]	123	229	0.54
65	OPE	183	336	0.54

[a] The coating contained this % carnauba wax and the remainder as resin, shellac, or OPE adjunct (see also Figure 1). [b] Measured water vapor resistance (r) per 0.0076-mm-thick film of this mixture. [c] Calculated resistance for the separate layers, based on values of 462, 143, 41, and 103 s/cm for 0.0076-mm films of carnauba, resin, shellac, and oxidized polyethylene, respectively; e.g., r of 65 resin, 35% carnauba is r = (0.65 × 143 + 0.35 × 462). [d] Oxidized polyethylene.

Table 3. Weight Loss and Internal CO_2 of Hamlin and Pineapple Oranges Coated with Oxidized Polyethylene, Carnauba Wax, Shellac, and Wood Resin[a]

coating code	Hamlin		Pineapple	
	wt loss[b] (%)	CO_2[c] (%)	wt loss (%)	CO_2 (%)
OPE[d]	58	3.0	58	5.5
M62125[e]	34	5.2	37	6.9
shellac[f]	55	15.0	52	15.2
RS[g]	49	10.7	50	12.1
control	100	2.3	100	5.9

[a] Coating application at 0.4 mL per fruit. Mean fruit weight was 160 g for both types of oranges. [b] Weight loss at 20 °C, 50% RH relative to washed fruit. SE = 2.7%. [c] Internal CO_2 after 1 week, four fruit per treatment. SE = 1.0%. [d] An oxidized polyethylene microemulsion supplied by Allied Signal with 16% AC316, 2.8% oleic acid, 2.1% morpholine, and 0.4% NH_3. [e] Carnauba formulation M62125. [f] Composition: 18.7% shellac, 1.3% oleic acid, and 3.7% morpholine. [g] Composition: 9.3% wood resin, 9.4% shellac, 1.3% oleic acid, and 4.7% morpholine.

Carnauba wax applied to fruit gave better protection against weight loss than shellac or polyethylene (Table 3). Thus, for this comparison the coating with highest resistance to water vapor (Figure 1) was also the most effective in reducing weight loss, caused in most part by loss of water vapor. However, water vapor resistance of coatings on plastic film may not always correlate to fruit weight loss, which is partly determined by a coating's tendency to plug pores in the fruit peel (Hagenmaier and Baker, 1993). For this reason, direct measurement of weight loss is preferred to measurement of resistance values for evaluation of fruit coatings.

Table 4. Weight Loss of Valencia Oranges Coated with Various Oxidized Polyethylene Formulations[a]

coating code	type OPE[b]	fatty acid	ratio FA/OPE	wt loss[c] (%)
K107C	E10	oleic	0.18	56
K109C	E20	oleic	0.18	52
K121A	AC680	oleic	0.12	53
K109E	E10	oleic	0.10	54
K113C	E10	lauric	0.18	66
K121E	AC680	lauric	0.12	52
K117A	E10	lauric	0.10	56
K121D	AC680	myristic	0.12	44
K121C	AC680	palmitic	0.12	41
K107B	E10	stearic	0.18	31
K121B	AC680	stearic	0.12	39
control				100
field[c]				80

[a] Morpholine content was 15% of wax ingredient. Mean fruit weight was 200 g. [b] Oxidized polyethlene. [c] Weight loss relative to washed control. SE = 4%. [c] Unwashed control.

Table 5. Shrinkage of Fruit Coated with Carnauba and Candelilla Wax[a]

coating code	type wax	fatty acid	ratio FA/wax	wt loss[b] (%) Valencia	wt loss[b] (%) Marsh
K90A	carnauba 3	oleic	0.22	41	
K111D	carnauba 3	oleic	0.15	40	50
M15B	carnauba 3	C16/C18[c]	0.20	22	28
M15A	carnauba 1	C16/C18	0.20	20	25
M9A	candelilla	oleic	0.20	21	27
K159B	candelilla	palmitic	0.21	22	25
M21D	C3/cand[d]	oleic	0.18	29	30
	control	none		100	100
	field	none		80	83

[a] Morpholine content was 20% of wax for K90A, 15–16% for the rest. Mean fruit weight: oranges, 200 g; grapefruit, 340 g. [b] Relative to fruit cleaned with type PSE brushes. SE = 4%. [c] Mixture of 54% palmitic acid and 44% stearic acid. [d] 50/50 mixture of carnauba 3 and candelilla wax.

The weight loss of citrus fruit coated with oxidized polyethylene or carnauba wax was influenced by the type and amount of fatty acid in the formulation (Tables 4 and 5). Formulations containing stearic and palmitic acid gave lower weight loss than those containing the shorter-chain lauric acid or those with oleic acid. The relative effectiveness of the different fatty acids thus agrees with previous findings that monolayers of short-chain or unsaturated fatty acids reduced water evaporation less than monolayers of saturated long-chain fatty acids (Jarvis et al., 1962).

Use of stearic or palmitic acid in the formulation did, however, give the disadvantage that when the coating was wetted, approximately 70% of the surface of the fruit was covered with whitish, fractured coating, about the same fraction as that observed for fruit coated with shellac- and resin-based commercial waxes. For formulations with oleic acid, only about 2% of the surface was so affected, with lauric acid about 5%. Thus, coatings made with palmitic or stearic acid would probably not be acceptable for fruit that is refrigerated, because condensation generally forms on the surface after fruit is transferred out of cold storage.

Emulsions that contained a 30–50% added hydrocarbon wax were especially effective in reducing weight loss (Table 6). The results were similar to those for candelilla wax, which contains 40–60% natural hydrocarbons (Table 1). Candelilla wax emulsions were previously shown to be effective in controlling weight loss of citrus (Gassner et al., 1969; Paredes-López et al., 1974).

The formulations containing natural or added hydrocarbons all reduced weight loss to 21–35% that of washed fruit when applied to oranges at the rate of 0.4 mL per fruit whether made with oleic, stearic, palmitic, or lauric acid or whether the formulation was clear like a microemulsion or (in the case of mixture K179B) had sufficiently large particle size to have a milky appearance (Tables 5 and 6). At such low values of weight loss it is possible that permeation through the cuticle was no longer the main pathway for water vapor loss, but rather movement through open pores (Hagenmaier and Baker, 1993). The optimum amount of added hydrocarbon wax seems to be about 50% of total wax for mixtures with oxidized polyethylene and 30% for mixtures with carnauba wax. Lower levels of hydrocarbon wax would increase the cost, at least if petroleum wax is the source of hydrocarbon. Higher levels were not emulsified, presumably because the oxidized polyethylene or carnauba acted as a secondary emulsifier that could not indefinitely be reduced in amount.

Table 6. Shrinkage Rate at 20 °C of Valencia Oranges Coated with Formulations[a] Containing Oxidized Polyethylene or Carnauba Wax with Added Hydrocarbon Wax (HCW)

coating code	type wax	type HCW[b]	HCW as % of total wax	fatty acid	ratio FA/wax	wt loss[c] (%)
M43C	AC629	P161	50	oleic	0.16	34
K123B	E20	P161	50	oleic	0.18	28
K127D	E20	P161	50	stearic	0.18	30
K179B[e]	AC629	P161	50	lauric	0.18	27
PE40[f]	AC680	BS195	40	oleic	0.18	35
K127C	E20	BS195	50	stearic	0.18	31
K175B	AC629	BS195	50	C16/C18[d]	0.14	32
K173C	AC629	BS195	25	C16/C18	0.12	31
K179A	AC629	PW500	50	oleic	0.15	31
M91B	C#3	P161	30	oleic	0.16	23

[a] Morpholine content wax 9–15% of wax ingredient. Fruit weight was 200 g. [b] Parvan 161, Be Square 195, and Polywax 500. [c] At 60% RH, relative to fruit washed with PSE brushes. SE = 4%. [d] A mixture of 54% palmitic and 44% stearic acid. [e] This formulation was milky white, the others were translucent. [f] Supplied by Allied Signal (Morristown, NJ).

Other wax formulations that did not contain morpholine or ammonia were also tested. The weight loss was 92% that of washed fruit for Marsh grapefruit coated with a milky-white emulsion containing 16% P161, 1.9% polysorbate 60, and 1.4% sorbitan monostearate; this coating was unacceptable because it became slimy when wetted. The weight loss was 79% or 86% of washed fruit, respectively, for carnauba wax or oxidized polyethylene, using formulations made up of 16.7% wax, 3.3% oleic acid, and 5% KOH; these coatings were unacceptable because they also were very susceptible to water damage.

The values of internal O_2 and CO_2 for wax-coated citrus fruit were virtually the same for different types of waxes; the internal O_2 was roughtly twice the CO_2 level (Table 7). For fruit with high-gloss coatings the O_2 was only about half the internal CO_2 value. Because fruit coated with wax had higher O_2, it tended to have less ethanol than fruit with high-gloss coatings (Table 8) and therefore less tendency to develop off-flavor (Davis and Hoffman, 1973; Cohen et al., 1990; Ahmad and Khan, 1987).

The gloss of oranges and grapefruit coated with wax was higher than that of uncoated fruit but lower than that of fruit coated with high-gloss commercial coatings (Table 9). However, after the coated fruit was wetted with water and dried, the waxy coatings maintained their appearance better than the resin and shellac coatings. Further, the relatively low weight less of waxed fruit resulted in little shriveling of the skin, thus giving the fruit peel a smooth appearance, even for fruit stored at ambient conditions for 6 weeks.

Table 7. Internal Gases of Fruit Stored for 1 Week at 20 °C and 75% Relative Humidity

coating code[a]	internal gas concn[b]			
	Valencia		Marsh	
	O_2	CO_2	O_2	CO_2
K111D	11.6	5.5	14.1	4.9
M21D	11.2	7.0	11.4	5.9
M9A	11.2	6.6	11.0	5.8
K109C	14.2	4.9	13.9	4.5
K123B	12.1	6.3	11.6	5.5
RS[c]	4.4	10.3	8.0	8.5
high gloss[d]	4.9	11.3		
control	16.6	4.1	18.4	2.5
field	18.7	2.1	20.0	1.1

[a] The wax microemulsions contained oleic acid and morpholine; application 0.4 mL per fruit, unknown for purchased control. [b] SE = 2% O_2, 1% CO_2. [c] Contains 9.4% shellac, 9.3% wood resin, 1.3% oleic acid, and 4.7% morpholine. [d] High gloss shellac and wood resin waxes applied in packinghouses; mean values for oranges from six central Florida packinghouses.

Table 8. Ethanol Content of Valencia Orange Juice from Fruit Stored for 1 Week at 20 °C

coating code	ethanol[a] (ppm)	coating code	ethanol[a] (ppm)
K123B	896	resin/shellac	1323
K179BC	1036	high gloss[b]	2010
M21D	1179	none (unwashed)	609
M9A	1194	none (washed)	695

[a] Each sample comprised juice from seven pooled fruit. For coatings applied in our lab results are based on four trials per treatment; SE = 60 ppm. [b] The high-gloss coatings (made from wood resin and shellac) were as applied in packinghouses; mean value for fruit from six packinghouses, two trials each.

Table 9. Gloss of Valencia Oranges with Wax and Resinous Coatings, Application Rate 0.4 mL per Fruit

coating	GU[a]	coating	GU[a]
control	4.4	K123B[b]	5.8
		resins[c]	6.8

[a] Gloss units, measured within 3 days of coating the fruit, 4 trials, 6 fruit/trial, 10 measurements per fruit. lsd = 1.3 GU. [b] For composition see Table 6. [c] Shellac and wood resin containing commercial waxes of proprietary composition, applied in the laboratory.

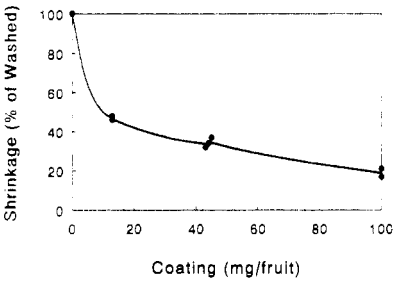

Figure 2. Weight loss of Valencia oranges at 20 °C as influenced by amount of K123B coating applied, dry basis. Mean fruit weight was 190 g.

The application rate for the data of Tables 3–9 was 0.4 mL of coating formulation with 20% total solids, which amounts to 80 mg of wax, dry basis. At this application rate the amount of wax is not critical for oranges (Figure 2). The relatively higher weight loss of coated grapefruit may indicate that 0.4 mL was not optimum for that fruit.

The ingredients used for the wax emulsions and microemulsions are approved by the FDA (Table 10). Regulations in some other countries do not permit petroleum-based waxes as coating ingredients, which may require that any hydrocarbon components in coatings for those markets be part of natural waxes or be made from raw materials other than petroleum.

Table 10. FDA Regulations for Wax Microemulsion Components as Coatings for Fruits and Vegetables

component	CFR ref[a]	limits on usage[b]
oxidized polyethylene	172.260	30 named fruits and vegetables
petroleum wax	172.886	none
synthetic petroleum wax	172.888	none
carnauba wax	184.1978	none
candelilla wax	184.1976	none
fatty acid	172.860	component of additive
fatty acid	172.210	component of coating
morpholine	172.235	salt of fatty acid
poly(dimethylsiloxane)	173.340	10 ppm as consumed
polysorbate 60	172.836	coating emulsifier
sorbitan monostearate	172.842	coating emulsifier

[a] Title 21, *Code of Federal Regulations*, revised April 1, 1990. [b] Other than good manufacturing practice.

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Received for review October 28, 1993. Accepted January 28, 1994.[⊗] All programs and services of the U.S. Department of Agriculture are offered on a nondiscriminatory basis without regard to race, color, national origin, religion, sex, age, marital status, or handicap. Mention of a trademark or proprietary product is for identification only and does not imply a warranty or guarantee of the product by the U.S. Department of Agriculture over other products that may be suitable.

[⊗] Abstract published in *Advance ACS Abstracts*, March 1, 1994.