HortScience 26(2):168-170. 1991.

Petroleum Spray Oil Effects on Net Gas Exchange of Grapefruit Leaves at Various Vapor Pressures

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Additional index words. Citrus paradisi, phytotoxicity, stomatal conductance, CO_2 assimilation

Abstract. **The effects of three highly refined petroleum spray oils and of ambient vapor pressure on net CO_2 assimilation (A) and stomatal conductance of water vapor (g_s) of single grapefruit *(Citrus paradisi* Macf.) leaves were investigated. Overall, g_s of various-aged leaves was decreased by a large leaf-to-air vapor pressure difference (VPD). In the first experiment, oils with midpoint distillation temperatures (50% DT) of 224, 235, and 247C were applied with a hand atomizer at concentrations of 0, 1%, and 4% oil emulsions in water and 100% oil, all with 0.82% surfactant (by volume). There was a tendency for oils of the two higher DT to decrease net gas exchange during a subsequent 12 days, but significant differences could not be attributed to oil DT. Both A and g_s were reduced by the two higher concentrations of oil mixtures. In the second experiment, a commercial airblast sprayer was used to apply the 224C oil at 4% or the 235C oil at 2% and 4% mixtures plus surfactant under field conditions. There were no significant effects of oil treatments on net gas exchange of leaves either measured under moderate VPD outdoors 1 day after spraying or under low VPD in the laboratory 2 days after spraying. No visible phytotoxic symptoms were observed in either experiment.**

Petroleum oils have been widely used for many years to control a variety of pests on citrus leaves and fruit (Riehl, 1981; Simanton and Trammel, 1966) in spite of potential phytotoxic responses (Beattie and Rippon, 1978; Beattie et al., 1989; Krenek and King, 1987). Phytotoxic responses in citrus, such as reduced fruit yield (Furness and Maelzer, 1981) and size (Dean et al., 1978); excessive leaf chlorosis, abscission, and fruit drop; reduced bloom and fruit set; poor fruit quality (Furness, 1981; Riehl et al., 1954); and increased susceptibility to cold-weather injury have been reported (Trammel and Simanton, 1966). However, phytotoxic responses are highly variable, depending on boiling point and viscosity of the oil (Beattie et al., 1989; Van Overbeek and Blondeau, 1954), percentage of unsulfonated residues (Trammel and Simanton, 1966), oil-pesticide interaction (Krenek and King, 1987), weather conditions (Dean et al., 1978), and method of application.

The mechanism of phytotoxicity seems to be associated with the physical presence of oil interfering with net gas exchange of CO_2 (Riehl and Wedding, 1959) and water vapor (Jones et al., 1983). Any reduction in evaporative cooling of leaves likely leads to elevated leaf temperatures. Oil tends to decrease net gas exchange of apple (Ferree et al., 1976) and pecan leaves (Wood and Payne, 1986). Similar responses may occur in citrus leaves, where the heaviest oils have been associated with the most acute phytotoxicity problems (Beattie et al., 1989). Our experiments were designed to test the hypothesis that spray oils can reduce net gas exchange of leaves under field conditions, depending on oil type and concentration.

We have recently reported that stomatal conductance of water vapor (g,), measured using porometry, could be reduced by applying relatively high concentrations (2% to 4%) of commonly used petroleum spray oils to single leaves of containerized trees (Salyani et al., 1990). However, we found no significant effects of spray oils on net CO_2 assimilation (A) or g_s when measured under moderate leaf temperature and high-humidity conditions in an open gas exchange system in the laboratory. Since g_s can respond to variations in ambient vapor pressure (VP) (Bunce, 1981; Vu et al., 1986), effects of spray oils on net gas exchange may vary with humidity. This study reports on the effects of VP and petroleum spray oils, applied to the entire canopy, on net gas exchange of grapefruit leaves measured outdoors.

All experiments were conducted on 2-year-old ‘Red Marsh’ grapefruit *(Citrus paradisi)* trees on *C. volkameriana* Ten. & Pasq. rootstock growing in 12-liter containers. Trees were well-watered, fertilized, and free of pests.

Application with hand atomizer (Expt. 1). Three horticultural spray oils with different midpoint distillation temperatures (50% DT), Sunspray 7N, Sunspray 9N (Sun Refining and Marketing Co., Philadelphia), and VOLCK Supreme Spray (Chevron Chemical Co., Memphis, Term.) (Table 1), were used to prepare oil and water mixtures of 0, 1%, and 4% (v/v) and 100’% oil. An emulsifier, T-MULZ-FCO (Hacros Chemicals, Kansas City, Kan.), was mixed with oils at 0.82% of oil volume before mixing with deionized water (Berndt, 1987). Nine spray mixtures (three oils × three concentrations) and a water-only treatment (0%) were applied with a constantly agitated hand atomizer in the spring (April) to each of 10 trees. The entire canopy of each tree was sprayed. All measured leaves were mature, 4 to 6 months old, and were sprayed thoroughly on both sides. A high uniformity of spray distribution on the leaves was verified by using a fluorescent dye and an ultraviolet light (data not shown). Trees were left outdoors for 2 months, watered every 2nd day, and fertilized weekly.

Net gas exchange was measured on single leaves using a LI-COR (Lincoln, Neb.) 6200 portable photosynthesis system with a l-liter cuvette. The ‘system was modified with the addition of an external flow switch (LI-COR, 1988) and appropriate software to allow steady-state measurements in an open system. All measurements were made outdoors between 0900 to 1200 HR to minimize variations in environmental conditions during the day. To reduce heat load during measurements, four layers of cheesecloth shaded the cuvette. Photosynthetic photon flux (PPF) within the cuvette was always greater than that required for saturation (600 $\mu mol \cdot s^{-1} \cdot m^{-2}$), and leaves were at 33 ± 3C, and CO_2 concentration was 350 ± 10 $\mu l \cdot liter^{-1}$. Each leaf was enclosed for 6 to 10 min while the continuously monitored CO_2 concentration and calculated g_s reached steady-state. Incoming vapor pressure and CO, concentration were noted, and cuvette VP and CO_2 were then recorded over three 10-sec periods. Calculations of A and transpiration (E) fluxes (von Caemmerer and Farquhar, 1981) were made using the absolute differences between incoming CO_2 and VP concentrations, assumed to be constant

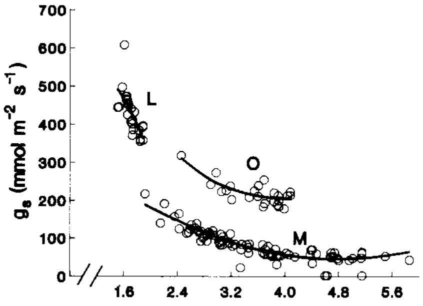

Fig. 1. Effect of leaf-to-air vapor pressure difference (VPD) on stomatal conductance (g_s) of recently expanded leaves measured under laboratory conditions (L) or outdoors (O) in Expt. 2 and of mature leaves (M) in Expt. 1. g_s (L) = 159 + 669 V – 297 $(V)^2$, $r^2 = 0.50$; g_s (O) = 1029-428 + 55 $(V)^2$, $r^2 = 0.63$; g_s (M) = 450-170 V + 18 $(V)^2$, $r^2 = 0.82$.

Received for publication 18 Apr. 1990. Florida Agricultural Experiment Station Journal Series no. R-00655. The cost of publishing this paper was defrayed in part by the payment of page charges. Under postal regulations, this paper therefore must be hereby marked *advertisement* solely to indicate this fact.

Table 1. Specifications of the petroleum spray oils used in experiments (from Salyani et al., 1990).

Oil properties	ASTM[z] standard method	Spray oils Sunspray[y] 7N	Sunspray 9N	VOLCK supreme spray[x]
Hydrocarbon composition (%)	D-2140			
Paraffins		59	65	68
Naphthenes		40	33	32
Aromatics		1	2	0
Distillation temperature at 10 mm Hg (°C)	D-1160			
10%		206	227	231
50%		224	235	247
90%		243	260	278
10%–90% (maximum)		44	44	47
Unsulfonated residue (vol %, minimum)	D-483	92	92	92
API[z] gravity at 15.5C	D-287	32.5	33.6	34.8
SUS[z] viscosity at 37.8C (sec)	D-2161	85	98	105
Pour point (°C)	D-97	−18	−12	−12

[z] ASTM, American Society for Testing and Materials; API, American Petroleum Institute; SUS, Saybolt Universal Seconds.

[y] Information supplied by Sun Refining and Marketing Co.

[x] Information supplied by Chevron Chemical Co.

Table 2. Effects of spray oil midpoint distillation temperatures (50% DT) and their concentration (%) on mean CO_2 assimilation (A), stomatal conductance (g_s), and water use efficiency (WUE = A/transpiration) of mature grapefruit leaves in Expt. 1 measured over a wide range of VPD (M, Fig. 1).[z]

Oil 50% DT (°C)	n[y]	A (μmol·s^{-1}·m^{-2})	g_s (μmol·s^{-1}·m^{-2})	WUE (μmol·mmol^{-1})
Water	8	5.09 (0.46)	105.4 (21.4)	1.55 (0.14)
224	25	4.46 (0.55)	84.2 (8.6)	1.28 (0.51)
235	25	3.42 (0.24)	79.1 (6.4)	1.32 (0.11)
247	25	4.00 (0.53)	73.0 (6.0)	1.50 (0.20)
Concn (%)				
1	39	4.58 (0.39)	92.6 (5.7)	1.35 (0.32)
4	18	3.67 (0.43)	73.4 (7.4)	1.40 (0.17)
100	18	2.90 (0.52)	54.2 (5.0)	1.36 (0.30)
Main effects				
Oil DT		NS	NS	NS
Concn		$P < 0.03$	$P < 0.001$	NS
Oil × concn		NS	NS	NS

[z] Numbers in parentheses are standard error.

[y] n = Number of leaves evaluated.

[NS] Nonsignificant at $P \geq 0.10$.

Table 3. Effects of various spray oils (see Table 1) and their concentration (%) on mean (n = eight leaves) net CO_2 assimilation (A), stomatal conductance (g_s), and water use efficiency (WUE = A/transpiration) of 2-month-old grapefruit leaves in Expt. 2. Data from day 1 were measured under a moderate range of VPD outdoors (O, Fig. 1) and those from day 2 were measured under relatively small VPD in the laboratory (L, Fig. 1).[z]

Oil 50% DT (°C)	Concn (%)	A (μmol·s^{-1}·m^{-2})	g_s (μmol·s^{-1}·m^{-2})	WUE (μmol·mmol^{-1})
		Day 1 (outdoors)		
224	4	9.4 (1.0)	209 (10)	1.2 (0.1)
235	2	9.6 (1.0)	240 (13)	1.2 (0.1)
235	4	8.6 (1.1)	208 (7)	1.1 (0.1)
		Day 2 (laboratory)		
224	4	9.7 (1.1)	432 (13)	1.4 (0.1)
235	2	7.3 (1.3)	429 (17)	1.1 (0.2)
235	4	8.7 (1.0)	425 (29)	1.2 (0.2)

[z] Numbers in parentheses are standard error.

during the typical 30-sec measurement period, and the CO_2 and VP concentrations within the cuvette, respectively. Sequential measurements showed that incoming CO_2 concentrations varied <0.5 μl·liter^{-1} and incoming VP varied <0.01 kPa during this short period. Leaf VP was assumed to be saturated at leaf temperature and was used to calculate leaf-to-air vapor pressure difference (VPD) within the cuvette. Water use efficiency (WUE) was calculated as A/E.

We could only evaluate 18 to 30 individual leaves per measurement day. On day 1 after spray treatments, we evaluated all 10 treatments, with three replicates of each treatment. Thereafter, we measured only four to six treatments per day, with at least four replicate leaves each on each of four clear measurement days during the 12 days following spraying. The experimental design was a three oil DT × three concentration factorial plus the water control (10 treatments) in a completely random design. A one-way analysis of variance was used to test for significance of the main effects; a 3 *x* 3 factorial tested for significant interactions.

Day-to-day variations in VP caused VPD to range from 1.9 to 5.8 kPa, which accounted for a significant amount of variation in g_s of mature leaves (M, Fig. 1), regardless of oil treatment. There was a tendency for oil to reduce net gas exchange, but oil DT did not have a significant effect on A, g_s, or WUE measured under these-conditions (Table 2). However, the two higher oil concentrations significantly reduced both A and g_s. Both A and E responded similarly, as there were no trends in WUE. No leaf chlorosis or abscission was observed during the subsequent 2 months, by which time oils had apparently weathered off leaves.

Application with commercial airblast sprayer (Expt. 2). To reduce potential variation attributable to differences in leaf age, populations of uniform-aged leaves were grown after decapitating 12 additional trees 60 cm above the container in April. Regrowth leaves reached full expansion outdoors over 2 months (May–June). The trees were placed within rows of mature grapefruit trees in the field. A 4% oil (plus surfactant, as in Expt. 1) and water mixture of 224C oil was applied at 2350 liters·ha^{-1}, and 2% and 4% mixtures of 235C oil were applied at 4700 and 2350 liters·ha^{-1}, respectively, using a commercial airblast sprayer (South Wind, Winter Garden, Fla.) under field conditions in July. The experimental design was a randomized block of four replicate trees. Visual observation of leaves revealed that oil was deposited on all leaf surfaces, although droplet size and distribution was not as homogeneous as in Expt. 1.

Gas exchange measurements were made outdoors, as in Expt. 1, on two leaves from each of the four replicate trees. The first day after spraying (day 1), cuvette leaves were at 37 ± 3C, VPD was 3.3 ± 0.8 kpa, and CO_2 concentration was 342 ± 8 μl·liter^{-1}. Trees were moved into the laboratory on day 2, and gas exchange was evaluated using a tungsten-halogen projector lamp filtered through 6 cm of water as a light source. PPF was still 600 μmol·s^{-1}·m^{-2}, but cuvette leaves were at 30 ± 1C, VPD was 1.7 ± 0.2 kPa, and CO_2 concentration was 352 ± 12 μl·liter^{-1}.

There was no significant difference among the effects of oil treatments on gas exchange measurements on either day (Table 3). We did not evaluate net gas exchange after 2 days, but it is unlikely that these oil treatments had any significant effect on later dates, as no visible phytotoxic symptoms were observed. Net gas exchange rates of this younger population. of leaves were higher than in the previous experiment. Higher g_s occurred at the lower temperature and VPD in the laboratory than outdoors (L and O, respectively, Fig. 1).

Decreases in g_s, as young leaves age, support previous studies (Syvertsen, 1985) that show the cuticular water loss can be high in young leaves. Negative relationships between citrus g_s and VPD have been described (Lloyd and Howie, 1989; Vu et al., 1986). This report describes such responses in citrus as affected by leaf age. overall, effects of VPD on g_s were more dramatic than those attributable to oil treatment. It is important to note that all gas exchange measurements were made under a reduced radiation load in the cuvette in an attempt to moderate elevations of leaf temperature during measurement, Thus, measured leaf temperature may not reflect actual temperatures that likely play a role in phytotoxic responses to oil.

Recommended concentrations of oils are 0.5% to 1.0% for control of many insect and fungal pests (Knapp et al., 1989). There were no phytotoxic symptoms observed in either experiment in this or previous studies (Salyani et al., 1990), even after oil concentrations of 4% and 100% decreased net gas exchange. Short-term reductions in stomatal conductance of grapefruit leaves do occur over broad ranges of VPD when oils are applied at excessively high concentrations. Such reductions do not necessarily precede phytotoxicity symptoms however, at least in these well-watered containerized trees.

All oils used in this study met the recommended specifications for unsulfonated residues (Trammel and Simanton, 1966) thought to be associated with phytotoxicity problems. It is possible that the relatively thick cuticle of citrus leaves renders them less sensitive than deciduous species (Ferree et al., 1976; Wood and Payne, 1986) to oil-induced decreases in net gas exchange. Seasonal studies that will investigate interactions between oil types, oil concentrations, leaf temperatures, and ambient vapor pressures as they relate to potential phytotoxic problems in citrus merit attention.

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