Adsorption, Mobility, and Filtration of Metolachlor in Container Media

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Abstract. Adsorption, mobility, and filtration ability of organic media toward metolachlor were evaluated in a series of laboratory experiments. Experimental variables included media type, metolachlor concentration, and equilibration time. Adsorption isotherms were determined by applying the log form of the Freundlich equation. Mobility was evaluated using glass columns filled with media, which were then surface spiked with metolachlor and then leached daily for 10 consecutive days. Peat, pine bark, combinations of these two media and a mixture of pine bark and sand adsorbed >90% of the ¹⁴C metolachlor. Freundlich sorption coefficients were 10.9, 18.2, 13.4, 14.2, and 11.0 for pine bark, peat, 5 pine bark : 1 peat, 3 pine bark : 1 peat, and 5 pine bark : 1 sand, respectively. In a timed exposure experiment using bark, minimum metolachlor adsorption (57%) was at 90 seconds and maximum adsorption (82%) required at least 1440 minutes. In column leaching studies, data for all media indicate that metolachlor is relatively immobile through these substrates. An initial pulse of metolachlor (<1.0 µg-liter⁻¹) was detected with each medium up to the third wetting event with a subsequent decline (>0.5 µg-liter⁻¹ for each medium) in the metolachlor recovered. Filtration efficiency of commercially formulated metolachlor from water passed through different lengths of pine bark filled filters was 0%, 17%, 20%, 22%, 23%, and 29% for filters 4, 20, 12, 8, 16, and 24 cm in length, respectively. These results support the contention that such filtration would be effective provided the residence time of water within the filter was sufficient for adsorption of the contaminant by the media to occur.

Nursery and landscape crops generally require frequent irrigation and pesticide application. Irrigation runoff from landscape plant production areas and landscape developments has been suggested as a source of pesticide contamination for local water supplies or surrounding bodies of water (Keese et al., 1992). Holding ponds can be used to reduce the amount of pesticide contaminated water from a container production area. However, where containment ponds are unavailable or not practical, other means of remediation of pesticide contaminated water may be required. Filtration of pesticide contaminated water using container production organic materials, such as pine bark, or peat, could be achieved using these materials as an adsorbate.

Adsorption of oxadiazon and oryzalin in both organic media and a mineral soil was evaluated in previous research (Wehtje et al., 1993, 1994). Averaged across all media and a mineral soil, with herbicide concentrations that ranged from 0.1 to 100 mg-liter⁻¹ (dry-weight basis), the proportion of oxadiazon and oryzalin adsorbed was 98% and 94%, respectively. Column leaching studies revealed that mobility was likewise limited. Neither herbicide was detected in either the leachate or deeper than 3 cm below the surface. It has been concluded that the detection of either oxadiazon or oryzalin in runoff water results from the material not reaching the media due to method of application (Gilliam et al., 1992). The generally high adsorptivity of horticultural media to herbicides has been observed by others (Moles and Whitcomb, 1976; Wilson et al., 1994). Among the herbicides registered for use in nursery crop

production, oryzalin and oxadiazon are not likely suspects for leaching. Neither material has been reported to be subject to leaching when used in field situations (Ahrens, 1994), and both are relatively insoluble (Camper et al., 1994). The water solubilities of oxadiazon and oryzalin are 0.1 and 2.6 mg-liter⁻¹, respectively (Ahrens, 1994).

The herbicide metolachlor is registered and commonly used in nursery production for the control of annual grasses (Bailey and Simmins, 1979). Metolachlor is nonionizable, has a vapor pressure of 1.7 mPa at 20°C (Worthing and Walker, 1983), water solubility of 550 mg-liter⁻¹ (Ahrens, 1994), and a sunlight photodegradation half-life in water of 28 days (Kochany and Maguire, 1994).

Peter and Weber (1985) evaluated the adsorptivity and mobility of metolachlor in nine distinct soils. Adsorption was determined with a batch equilibrium technique using ¹⁴C-metolachlor. The distribution coefficients (K_d) ranged from 0.48 to 10.9 with the proportion of metolachlor that remained in solution ranging from 9% to 68%. Regression analysis revealed that increasing metolachlor adsorption and K_d values correlated well to increasing organic matter and clay content, as well as cation exchange capacity (CEC).

Koncal et al. (1981) examined metolachlor displacement in media following application to 12.5-cm-deep pots. Rate of application was 10.0 and 40.0 kg-ha⁻¹ for a granular and tablet formulation of metolachlor, respectively. The authors concluded that the rate of metolachlor release from the tablet formulation was adequate for weed control, yet slow enough so as to prolong activity. Release from the granular formulation was too rapid rendering weed control limited in duration.

Mahnken et al. (1994) examined the leaching of metolachlor through various combinations of bark (redwood, pine, and hardwood), sand, and Yolo loam. Maximum adsorption of metolachlor occurred with hardwood plus pine bark (1:1 v/v), and pine bark plus sand (3:1 v/v). Minimum adsorption occurred with redwood bark plus sand (3:1 v/v), comparable leaching recoveries were 560 µg/day per container, or 2.5% of amount applied. While the amount of metolachlor recovered in leachate was a function of

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media composition, it never exceeded 5% of the amount applied.

It is apparent from these studies that horticultural media are highly adsorptive toward a variety of herbicides. Horticultural media may offer a means of cleansing pesticide-contaminated water. This may be accomplished by simply allowing pesticide-contaminated water to drain through a medium-filled filter. Metolachlor, with its relatively high water solubility would represent a fairly difficult challenge for removal by filtration. The first objective of this study was to evaluate both the adsorptivity and mobility of metolachlor in container media. In previous studies, which established the high adsorptivity of media toward herbicides, at least a day was allowed for the adsorption to occur. However for removal by filtration to be effective, adsorption would have to occur in a much shorter period. Consequently, time was included as an experimental variable in the adsorption studies. The second objective was to evaluate the feasibility of cleansing metolachlor-contaminated water by allowing it to drain passively through pine bark filters.

Materials and Methods

General procedures. Two media components and three blended media, which are typical of those used in container nursery production in the southeastern United States, were used in the adsorption and mobility studies. The two components were pine bark (PB) and peat (PT). The blended media were 3 PB:1 PT, 5 PB:1 PT, and 5 PB:1 sand (S), v/v. Media were characterized by standard analytical procedures as to their organic matter content, CEC, bulk density, and water holding capacity (Table 1). The gravimetric water holding capacity, more commonly referred to as field capacity (FC), and the water holding capacity at 30 kPa were determined (Gardner, 1986).

Metolachlor radioisotope and liquid scintillation spectrometry (LSS) were used to facilitate metolachlor detection in both the adsorption and mobility studies. Appropriate amounts of formulated metolachlor and ring labelled ^{14}C -metolachlor [1×10^6 disintegrations per minute (dpm) specific activity of $9.7 \text{ Mbq} \cdot \text{mg}^{-1}$] were used. Counting efficiency averaged 94%. Evaluation of the removal-by-filtration concept required that large quantities of water be spiked with metolachlor, which rendered the radiological techniques for metolachlor impractical. Consequently, metolachlor detection in this part of the study was accomplished through enzyme-linked immunoabsorbent assay (ELISA). This technique for pesticide residue detection and quantification in water and substrates has been reviewed (Hutchinson et al., 1993; Lawruk et al., 1992, 1994; Van Emon et al., 1987, 1989). The range of metolachlor detection for this study was 0.05 to $5.0 \mu\text{g} \cdot \text{liter}^{-1}$ (Van Emon et al., 1987).

Table 1. Physical properties of media used in metolachlor sorption studies.

Media (v/v) ^x	Physical properties				
	CEC ^z	Bulk	Ignition	Water-holding	
	(meq/100 g)	Density ($\text{g} \cdot \text{cm}^{-3}$)	wt loss ^y (%)	Gravimetric	30 kPa
PB	45	0.27	71	120	68
PT	62	0.14	98	388	109
3 PB:1 PT	35	0.25	89	179	69
5 PB:1 PT	49	0.28	85	150	60
5 PB:1 S	30	0.58	64	69	33

^zCEC = cation exchange capacity.

^yA determination of organic matter.

^xPB = pine bark; PT = peat; S = sand.

Experiments using ^{14}C -metolachlor had four replications for the adsorption and timing studies and 8 for the mobility study. Experiments using ELISA procedures were replicated three times. Data from each experiment were subjected to analysis of variance and treatment means compared using LSD at the 5% probability level for the ^{14}C -metolachlor studies and 10% probability level for the ELISA studies.

Experiment 1. Metolachlor adsorption was evaluated using a soil solution technique (Patterson et al., 1982). Water used to bring media to FC had been spiked with a combination of formulated and ^{14}C -labeled metolachlor. Sufficient water was added to media samples (1.2 kg) to bring each to FC and achieve concentrations of 0.01, 0.10, 1.00, and $10.0 \text{ mg} \cdot \text{kg}^{-1}$ (dry-weight basis). After the metolachlor-containing water had been added to and mixed thoroughly with the medium, samples were stored in glass beakers, covered with aluminum foil to prevent evaporation, and allowed to equilibrate for 48 h at 30°C. This equilibration time is commonly used in soil solution work (Adams et al., 1982). Previous trials by the authors with thin-layer chromatography (unpublished) indicated that no detectable metolachlor degradation occurred within this time. After 48 h equilibration, samples were divided into four soil solution extraction cups and compacted by applying pressure (275 kPa) with a piston attached to a modified drill press. Extraction cups were constructed of plexiglass pipe with an inside diameter of 8 cm. The bottom plate was perforated allowing the solution to escape. Solution was collected in a catch cup fitted below the perforated plate. Samples were centrifuged for 3 h at 2500 rpm. Subsamples (1 ml) of the extracted solution were assayed for ^{14}C -metolachlor using LSS. The fraction of metolachlor in solution was determined by multiplying the amount of radioactivity in the 1 ml sample by the total amount of water that had been added to bring the sample to FC, and dividing by the total amount of radioactivity originally added to the sample. The difference between the amount in solution and the total amount which had been added to the sample was adsorbed.

Data were plotted using the logarithmic form of the Freundlich equation (Bailey and White, 1970):

$$\log S = \log K + (1/n) \log C \quad [1]$$

where S = adsorbed-phase concentration, C = equilibrium solution-phase concentration, and K and $1/n$ are the Freundlich adsorption constants.

Statistical analysis was used to determine if the slopes of the isotherms were different from zero and from each other as determined by t tests (5% level). Significant differences among intercepts were determined using the confidence intervals (5% level) around the isotherm regression lines. The Freundlich K value was determined from the intercepts of the regression lines (Barnes et al., 1989).

Experiment 2. Adsorption of ^{14}C -metolachlor at a single concentration ($1 \text{ mg} \cdot \text{kg}^{-1}$) as a function of time was measured using PB medium. The equilibration time progressed downward in a geometric manner from 24 h to 1 min 30 sec. A relationship between equilibration time and adsorption was determined. At equilibration times of 75 min or less, the standard centrifuge-based soil solution extraction procedure was deemed inappropriate due to the time necessary for centrifuge acceleration. For these equilibration periods, the soil solution was extracted via pressure. Media at FC were placed in the extraction cups and pressed with a piston. While this technique does allow for the recovery of some of the solution, the amount recovered is not as great as with centrifugation and it is damaging to the cups.

Experiment 3. Metolachlor mobility was evaluated for the media combinations using a column leaching technique. Columns consisted of glass pipe with a 3.4-cm inside diameter held in a

vertical position. The pipe was open at the top, and closed at the base except for a 3-mm exit port, which allowed leachate to escape into a beaker. Glass wool was placed over the exit port and the column was filled with medium to a depth of 25-cm and lightly packed only to remove air pockets and not compressed. This simulated horticultural media depth in a 25-cm container. A separatory funnel held above the columns served as a reservoir. Media-filled columns were preconditioned by allowing 1 liter of water to percolate through the column before spiking the media with metolachlor. The surface of the media was spiked with 40 μg of commercially formulated metolachlor and a known amount of ^{14}C -metolachlor. Metolachlor was allowed to equilibrate for 24 h before leaching.

Twenty-three milliliters of water (equivalent to a 2.54 cm irrigation) was used for each of 10 leaching events. Water was added in four equal volumes over a 1-h period. Between 85%–90% of the applied water was collected within 4 h after the final application had ended. Volume of this sample was measured and a 1 ml sample taken for analysis. Columns were allowed to continue draining before the next leaching. Amount of water collected in this period generally was 2–5 ml and was discarded because of possible evaporational enrichment.

Experiment 4. Metolachlor removal from water by filtration with PB filled filters was tested. To simulate nursery water conduits, 1-m long conduits were constructed of 15.2-cm-diameter PVC pipe that had been split in half. Filters 4, 8, 12, 16, 20, and 24 cm in length were also constructed from 15.2-cm-diameter PVC pipe and filled with PB medium. Pipe sections of the appropriate length were cut open longitudinally and 21 mm of the wall removed. When reclosed and held with a band clamp, these reduced-circumference sections fitted tightly within the conduit sections. PB for the filters was pre-sifted through a 3-mm round-holed screen with that passing through the screen discarded. Filter sections were filled with a known volume of PB. Both ends of the filter section were closed with circular pieces of shade cloth, which were glued in place. Filter sections were positioned within the conduit, and caulking was used to prevent water from seeping between the filter-conduit union. Conduits with filter apparatus were positioned on a greenhouse bench at a 10% slope. Plastic water storage containers (19-liter), which served as reservoirs, were positioned such that the valve directed water into the upper edge of the conduit. Water draining through the conduit with filter was collected in a 19-liter pail. The maximum flow rate of each filter size was determined in preliminary trials. This was accomplished by allowing water to drain from the reservoir into the conduit at progressively greater flow rates. The flow rate at which the depth of the water in front of the filter remained constant was deemed the maximum flow rate. From this value the area, and in turn the filter volume occupied by the flowing water, could be determined (Amer. Concrete Pipe Association, 1987). The flow rate was used in subsequent trials with metolachlor-spiked water. For these trials, the reservoir water was spiked with commercially formulated metolachlor at 1 $\text{mg}\cdot\text{kg}^{-1}$. Metolachlor spiked water was allowed to flow through the filter at the appropriate flow rate. Water leaving the conduit was collected and a sample taken for metolachlor quantification. Immediately after the completion of the filtration process, an additional 19 liters of water (nonspiked) was passed through the filter, and a sample collected for metolachlor quantification. The purpose of this flush was to determine if any metolachlor held by the filter was subsequently released into this flush.

Results and Discussion

Experiment 1. Metolachlor adsorption was influenced (prob-

ability <0.01) by concentration and type of media (Table 2). Interaction of these two variables was not significant. By applying equation 1, the adsorption isotherms for these five media best fit an L type isotherm classification (Giles et al., 1960). Statistical regression analysis techniques described by Dao et al. (1982) indicated that the slopes ($1/n$) did not differ significantly within each medium. Each slope is presented separately with the intercepts (K) for the five media (Table 2). The value K is the index of the quantity adsorbed per gram of media in an equilibrium solution (Barnes et al., 1989). Applying the log form of the Freundlich equation results in an empirical, linear equation in which sorption is dependent on solution concentration but never reaches a sorption maximum. This method of determining herbicide adsorption was recently reviewed by Harper (1994) and is applied in other herbicide soil studies (Gan et al., 1994).

PT was the most adsorptive medium, with 92% adsorption averaged over concentrations (i.e., maximum amount in solution was 7.6%). PT had the highest CEC and K value, 62 $\text{meq}/100\text{ g}$ and 18.2, respectively. PB, PB: PT combinations, and 5 PB: 1 S mixture were less sorptive for metolachlor than PT, but adsorption was still 88.5% for all concentrations. K values corresponded with the amount of PT in the media, ranging from 14.2 to 13.4 for 3 PB:1 PT, and 5 PB:1 PT, respectively. Where media contained no PT, K was 11 and 10.9 for 5 PB:1 S and PB, respectively. Overall, these data indicate the highly adsorptive nature of any medium for metolachlor. Pusino et al. (1992), who evaluated metolachlor adsorption across a series of field soils, noted that adsorption was strongly correlated with organic

Table 2. Experiment 1, adsorption of ^{14}C -metolachlor and Freundlich constants in five different media.

Media	Metolachlor concn (mg.kg ⁻¹)					Freundlich		
	0.01	0.1	1	10	LSD ^z	constants		
	(v/v) ^y					K	1/n	r ²
(% in solution)								
PB	11.5	9.1	8.6	8.6	NS	10.9	1.03	0.995
PT	7.6	5.3	5.8	5.6	0.7	18.2	1.04	0.997
3 PB:1 PT	7.0	6.6	8.2	5.9	0.9	14.2	1.01	0.999
5 PB:1 PT	8.1	6.7	8.1	6.7	NS	13.4	1.05	0.999
5 PB:1 S	9.3	7.5	7.0	9.2	NS	11	0.99	0.994

^zLSD 0.05 between any individual concentrations.

^yPB = pine bark; PT = peat; S = sand.

Fig. 1. Experiment 2, ^{14}C -metolachlor percent adsorption in pine bark measured from 1 min 30 sec to 24 h.

Table 3. Experiment 2, recovery of metolachlor in leachate after 10 consecutive leachings with 23 ml of water each. Metolachlor (40 ug) had been applied to the surface.

Media (v/v) ²	Leachate recovered		Total amount of metolachlor recovered in leachate		Avg metolachlor in leachate (ng-liter ⁻¹)
	(ml)	(% of applied)	(ng)	(% of applied)	
PB	199	86	93	0.23	467
PT	216	94	94	0.23	435
3 PB: 1 PT	203	87	100	0.25	492
5 PB: 1 PT	202	88	103	0.26	510
5 PB: 1 S	207	90	99	0.25	478

²LSD 0.05 between any individual concentrations.

³PB = pine bark; PT = peat; S = sand.

Fig. 2. Experiment 3, ¹⁴C-metolachlor mobility through columns filled with five different media, leached over 10 consecutive days.

matter. In that study, the most adsorptive soil was a Macomer sandy loam which contained 9.3% organic matter. The resulting K value from the Freundlich isotherm was 16.8, which is in agreement with the values obtained with container media. Conversely, Carpi clay, a soil low in organic matter (1.0%), had a K value of only 0.98. These data concur with previous research with oxadiazon and oryzalin which indicated the adsorptive nature of container media to these

herbicides (Wehtje et al., 1993, 1994).

Experiment 2. Adsorption of metolachlor over time by PB was >50% at the initial measurement, 1 min 30 sec (Fig. 1). Adsorption increased to >70% after only 24 min. After 24 min, the rate of adsorption increased at a decreasing rate, reaching 80% after 12.5 h and >80% after 24 h. Thus the equilibration time of 48 h, as used in the previously described adsorption study, is more than adequate if not excessive (Table 2).

Experiment 3. For mobility, metolachlor was recovered in leachate at a rate that was equivalent across all media (Table 3). Total metolachlor recovered after 10 leaching events ranged from 93 to 103 ng, which corresponds to 0.23% to 0.26% of the amount applied, 40 µg. Similarity in both the rate and total amount recovered is probably a reflection of comparable adsorption for all of the media. Within each medium, metolachlor appeared in the leachate between the second and third leaching event (Fig. 2). Similar peaks for each medium indicated an initial surge of metolachlor at 0.6 to 1.2 ng·ml⁻¹. Generally, after the seventh leaching event, the amount of metolachlor recovered was 0.06 ng·ml⁻¹ and continued to decrease. Of the metolachlor surface applied to the column, >99% was adsorbed and thus immobile.

Peter and Weber (1985) reported similar results for metolachlor mobility within a Norfolk sand, organic matter 0.5%, using a column leaching technique. Columns were leached daily with 150 ml water (2.2 cm/day) for 24 consecutive days. The total amount of metolachlor recovered during the experiment in the leachate was 11.1% of the amount applied. Metolachlor concentration within leachate peaked at 110 mg·liter⁻¹, and then fell to a constant level of 40 mg·liter⁻¹. Greater than 88% of the metolachlor applied was adsorbed. As indicated by this and other research (Obrigawitch et al., 1981; Wietersen et al., 1993; Pignatello and Haug, 1991), metolachlor adsorption is affected by organic matter content.

Experiment 4. Nurseries having sloped conduits that direct drainage water away from container areas and towards holding ponds are common in the Southeastern United States. Water may leave the property directly from the nursery area, or if captured in a holding pond, used for irrigation. The most logical site for positioning a water filter would be in these conduits.

Filters were placed in the 1-m PVC conduits and metolachlor spiked water allowed to pass through. Metolachlor removal by the PB-filled filter was generally proportional to filter length (Table 4). While no metolachlor was removed by the 4-cm filter, filters 8–20 cm in length provided 17%–23% total retention. The 24-cm filter exhibited the highest removal (29%). With this filter, 36% of metolachlor contained within the 1.0 mg·kg⁻¹ was retained, 7% of

Table 4. Experiment 4, ability of pine bark-containing filters to cleanse metolachlor-spiked water².

Filter length (cm)	Amount of PB (g)	Maximum flow rate (liters·h ⁻¹)	Water		Volume of filter occupied by water (cm ³)	Residence time of water in filter (sec)	Filtering ability		
			depth at filter entrance (cm)	Area contacted with water at filter entrance (cm ²)			Metolachlor recovered during initial filtration (%)	Metolachlor recovered in subsequent wash (%)	Total amount retained (%)
0	0	---	0	0	0	0	100 a ^y	0 c	≈0 b
4	140	610	2	17	68	0	77 ab	23 a	≈0 b
8	280	580	3	25	200	1	64 b	14 b	22 ab
12	420	420	4	37	444	4	70 b	10 b	20 ab
16	560	375	5	50	800	8	69 b	8 ab	23 ab
20	700	325	6	58	1160	13	74 ab	9 b	17 ab
24	840	250	7	81	1944	27	64 b	7 bc	29 a

²Nineteen liters of metolachlor spiked water (1 mg·kg⁻¹) was passed through each filter, collected and analyzed. Subsequently, an additional 19 liters of nonspiked water (wash) was passed, collected, and analyzed.

^yMeans within a column followed by the same letter are not significantly at LSD 0.01.

the initial was recovered in the subsequent wash with clean water. Thus the net removal was 29%. This relatively modest amount of metolachlor that was recovered in this subsequent washing indicates the retention of metolachlor was predominately true adsorption and not temporary filtration.

Within the filters evaluated, a relationship clearly exists between length, residence time, and the ability to remove metolachlor. Furthermore, these data are in general agreement with the adsorption-time exposure study. Metolachlor removal from water by filtration probably reflects the same phenomenon as that observed in the adsorption studies, only the time involved is much shorter. The residence time of 27 sec, as provided by the 24-cm filter, resulted in 29% removal of metolachlor. Metolachlor adsorption in the timing study after a 90-sec exposure to PB was 57%. These data indicate that to achieve water cleansing values of >70% will probably require filter residence time of 6 min. Filters with this residence time will have to be either very long or constructed with internal baffles to increase exposure time.

The practical implications of Freundlich isotherms, adsorption, and mobility data show that media are very adsorptive. These data confirm the suspicion that contaminant in nursery runoff is largely attributable to a granular herbicide not reaching pots during the application. Conversely, media such as PB and PT are so adsorptive that they may offer merit as filter materials to remediate pesticide-contaminated water.

Successful use of this technique will require that the filtering process have a sufficiently long residence time for adsorption to occur. Potentially, 25%–30% filtration of water containing 1.0 mg-liter⁻¹ metolachlor could be achieved by simply passing contaminated water through a 24-cm PB-filled filter. To increase filtration ability, filter residence time could be increased with either slower flow rates, different media, greater filter length, or filters fitted with internal baffles. However, these modifications may also render water flow rate unacceptably low.

While complete filtration of contaminants in runoff water may not be achievable, the potential for use of existing grower materials, such as peat and pine bark, to reduce off-site movement does exist. Development of filtering systems that are easily constructed and maintained by container nursery producers is achievable.

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