CHAPTER TWO

CONTROLLED RELEASE PROPERTIES OF LIGNIN FOR TRIFLURALIN

ABSTRACT

Laboratory, greenhouse, and/or field studies were used to explore the potential of lignin as a controlled release material for trifluralin (α, α, α-trifluoro-2,6-dinitropropyl-p-toluidine) and other volatile herbicides. No controlled release properties were observed during preliminary studies with herbicides with vapor pressures greater than trifluralin (i.e. EPTC (S-ethyl dipropylthiocarbamate) and triallate (S-(2,3,3-trichloroallyl) diisopropyl thiocarbam-ate)). A commercially available emulsifiable concentrate of trifluralin (Treflan)[1] was mixed with various lignin fractions slurried in water. The slurried lignins and emulsifiable concentrate were tank-mixed with water just prior to application. Dry lignin fractions and technical grade trifluralin were mixed while grinding. Alterations in the formulation procedures and lignin to trifluralin ratios were tested. Bio-assays were used in the field and greenhouse tests to measure the phytotoxic effects of the herbicides. Barnyardgrass (<u>*Echinochloa crus-galli*</u> (L.) Beauv.) shoots or the roots of

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[1] Treflan is the trade name for the commercial emulsifiable concentrate marketed by Elanco.

corn (*Zea mays* L.) seedlings were used in the greenhouse studies. The weights of oat (*Avena sativa* L.) inter-cropped with soybeans (*Glycine max*) seedlings were used as the assay species in the field trial. Trifluralin displaced by a constant stream of N_2 was used to measure the control release properties in the laboratory studies. Under greenhouse conditions surface applications of various lignin-trifluralin fractions when surface applied provided extended weed control when compared to a similarly applied emulsifiable concentrate of trifluralin. In all experiments the duration of weed control provided by the non-soil-incorporated lignin-trifluralin formulations was shorter than the soil incorporated treatments. The extended weed control observed in greenhouse studies was not duplicated in the field or laboratory test. Controlled release properties of lignin with regard to trifluralin appear to be limited to physical encasement and not the result of chemical interaction between lignin and trifluralin.

INTRODUCTION

Numerous herbicides control emerging weed seedlings. Often these herbicides are applied prior to planting a crop (41, 42). Many pre-plant applied herbicides require soil incorporation within a prescribed period of time after application (3, 5, 27, 37, 50). Soil-incorporation for many herbicides is essential for weed control. Incorporation disperses the herbicides throughout the upper layer of soil. This provides better coverage and contact with emerging seedlings (2, 18, 28, 42). Soil-incorporation also reduces exposure to direct sunlight. This reduced exposure lowers the level of photodegradation for photosensitive herbicides (64). Off-site movement of volatile herbicides is reduced with soil-incorporation (10, 21, 22, 25, 30, 38, 45, 59, 64).

Reducing off-site movement of pesticides has recently been given a higher priority by the EPA (16, 38, 39, 61). Most point pollution sources have been identified and regulations implemented and refined. This has freed agency staff to focus on the more diffuse and regulatory challenging issue of non-point pollution sources. The EPA has identified agriculture as the nation's largest non-point polluting industry (1).

A primary form of agriculturally derived non-point pollution occurs with soil erosion. Minimizing or eliminating soil preparation for seed beds has developed as one possible solution for soil erosion. Reduced soil erosion holds promise as a means of limiting surface water contamination from pesticides and fertilizers.

Defining and solving the issue of ground water contamination is less clear. Soil properties and fauna are dominate factors impacting the movement of herbicides in soils. Increased activity of soil organisms in no-tillage operations appears to enhance the movement of some herbicides through the upper soil layers. Once a compound reaches the vadose zone continued movement becomes more dependent on the movement of soil water and specific properties of the herbicide and soil (12, 29, 30, 31, 32, 62). Questions remain regarding the positive or negative impact of no-tillage or minimum-tillage operations on ground-water contamination. Changes in tillage practices are unlikely to alter the compound specificity of the ground-water contamination problem (39).

Negative factors associated with no-tillage operations include the cost of new equipment, soil compaction, elimination of mechanical cultivation and lose of herbicides requiring soil-incorporation (4). Mechanical cultivation offers low cost, weed control practice that appears to be less environmentally insulting then many alternatives (1). Many soil-incorporated herbicides have historically provided a reliable low cost weed control.

With the increase of no-tillage farming and the loss of soil-incorporated herbicides growers have limited alternative weed control measures. Pre-emergence herbicides provide one alternative. Similar to soil-incorporated herbicides pre-emergence materials are used in a preventative manner. Use is based on predicted weed pressures verse actual evaluations. Unlike soil-incorporated herbicides pre-emergence materials require rainfall or irrigation (3). Efficacy is reduced or lost without adequate surface applications of water. A second alternative to soil-incorporated herbicides are the post-emergence herbicides. Their use is limited to responses to identified weed infestations. Post-emergence herbicides provide little or no residual weed control, allow weed crop competition to exist during the early growth of the crop, and at this time are a relatively expensive alternative to cultivation and most pre-plant soil-incorporated herbicides. In irrigated crops the delivery of some soil-incorporated herbicides provides a third alternative (37).

Future advances in application equipment, adjuvants or formulations may offer assistance in maintaining effective herbicides while adopting no-till farming practices (11, 16, 26, 35, 63). Considerable effort has been expended on the development of controlled release formulation of trifluralin using starch xanthides and related chemistries (9, 10, 24, 46, 51, 57). Much of the starch xanthate formulation work has been based on developing a granular formulation of trifluralin. Grower acceptance of a non-aqueous application technology is questionable. The ease, convenience and familiarity of the pesticide formulations applied in a water carrier will make convincing growers to change formulations difficult. In addition the limited soil movement of trifluralin leaves some question regarding the efficacy of granular formulations (9, 27, 28, 35, 46, 58).

Various synthetic and naturally occurring materials have been used to control or retard the release of pesticides or pheromones for decades (6, 7, 11, 13, 19, 36, 52, 54, 55).

Early aquatic controlled release materials included paints designed to retard the release molluscicides (6, 7, 19). More recently aquatic pest control materials have been release from various polymers utilizing a serious of shapes, sizes, and densities. Design changes can optimize efficacy by adjusting the placement and release rate for the targeted pest (7, 52, 53).

The success of controlled release aquatic pesticides is do in part to the relatively stable environment offered by the water. In contrast terrestrial pest controlled release materials operate in a more dynamic environment. Temperature, humidity, wind and other climatic conditions change drastically and frequently. Soil types and plant-back restrictions are additional considerations. These changes impact most controlled release materials

The success achieved in developing controlled release formulations for pheromones or growth regulators was in part a result of the low volume of material utilized (13, 60). Until recently, most herbicides have been applied at 0.25 kg/ha or greater. The pheromone controlled release materials can be composed out of more exacting and expensive materials.

The development of the No-Pest strip[2] by Shell Co. a polyvinyl chloride with appropriate plasticizer, stabilizers, and 20% dichlorvos (dimethyl-2,2-dichloro-vinyl phosphate) was the first long lasting controlled release material of commercial success (6, 7, 19). A series of similar protects designed to control ticks and fleas on dogs and cats were developed soon after the introduction of the No-Pest strip (7, 47).

Recently commercial formulations of microencapsulated herbicides have been introduced. Test on the microencapsulated formulation of metolachlor (2-chloro-*N*-(2-ethyl-6-methylphenyl)-*N*-(2-methoxy-1-methylethyl) acetamide) have revealed that the product provides questionable controlled release advantages (4, 63). The release cycle

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[2] The trade name for the dichlorvos formulation used to control houseflies developed by Shell Co.

appears to be strongly related to wetting-drying cycles. A larger release rate is observed on drying rather then wetting. This sequence fails to take advantage of the potential soil-incorporation and weed seedling germination occurring during or after a rainfall event.

The focus on developing synthetic polymeric controlled release materials may be ignoring a low cost substitute that has been used indirectly for years. The effect of soil organic matter on the retention and efficacy of various herbicides has been well established for decades (49, 56). Numerous herbicide labels describe limitations regarding use or adjustments in rates that are made as a result of soil organic matter levels.

The soil organic matter is composed primarily of two materials humic and fulvic acids (22, 50). Each has numerous similarities to the wood pulping by-product lignin (23, 44). Garbarini reports that the oxygen and carbon content of soil organic matter are more relevant than simple soil organic matter levels when attempting to predict the sorption properties of soils for various herbicides (19). This conclusion appears to collaborate the findings of Riggle and Penner(40), and Dellcolli (14). Each has reported that specific lignin fractions have a greater controlled release properties than others.

The studies were designed to explore the potential of lignin as a control release material for volatile herbicides.

MATERIALS AND METHODS

General greenhouse materials and methods. An air dried Spinks loamy sand soil was used as the growing media for the greenhouse studies. The Spinks loamy sand's organic matter was 0.8% and the pH 6.5. Soil was screened prior to each study. A soil sieve with 2 mm square openings was used to standardize soil structure from study to study. After herbicide application the pots were sub-irrigated until the soil surface moistened. The soil surface was maintained in a moisten state throughout the studies.

Herbicide applications consisted of three types: none treated controls, soil incorporated treatments, and none soil incorporated surface applications. One-liter plastic pots were used as growing containers. All herbicide applications were applied to the soil surface and not incorporated unless otherwise stated. After allowing for a prescribed period of time the indicator plants were sown on top of the treated soil and covered with 3 cm of non-treated soil.

Five hundred-milliliter pots containing 5 cm of soil were used when treating the soil that was to be incorporated. Each 500 ml pot was inverted ten times after the application to assure uniform incorporation of the herbicides. The treated soil was placed above non-treated soil in the 1 liter plastic pots.

Herbicides and herbicide-slurried lignin tank mixes were applied with an aqueous spray. The carrier and formulated products were applied at 375 L ha^{-1}. The spray was maintained at 10.25 kg cm^{-1}. Pots were placed in single or double rows under an SS8002E nozzle. A 50 mesh screen inserted prior to the nozzle prevented blockage of the orifice and a distortion of the spray pattern. The boom was attached to a motorized belt system and passed over the pots.

For the barnyardgrass bio-assays twenty seeds were sown per pot. Shoot lengths were measured when the shoot length of the controls were 20 to 25 cm long. The average shoot length of the barnyardgrass seedling per pot were used for the analysis of variance (ANOVA).

An ANOVA was run on the averages and differences between means was determined using a Duncan's Multiple Range test at the 5% level of significance.

EPTC (Table 1). EPTC (S-ethyl dipropylthiocarbamate) and individual lignin fractions slurried in water were tank-mixed at a 1:3 ratio of, 1 part dry weight lignin and 3 parts active herbicidal ingredient. The active material was applied at a rate of 1.7 kg ha^{-1}. Twenty hours after EPTC application barnyardgrass seeds were sown.

Trifluralin study using low levels of slurried lignins (Table 2). Trifluralin and various slurried lignin fractions were tank mixed at a 1:3 ratio, 1 parts dry weight lignin and 3 parts active herbicidal ingredient. The active material was applied at 0.84 kg ha^{-1}. Zero, 17 and 36 days after herbicide application barnyardgrass seeds were sown.

Direct verses greenhouse filtered sunlight. (Table 3). An emulsifiable concentrate of trifluralin and various slurried lignin fractions were tank mixed with equal parts of lignin and active herbicide. The active material was applied at 0.84 Kg ha^{-1}. The following lignin fractions blocked the 50 mesh screen: PC951, PC951C, PC955A, PC955B, PC955C and PC950. All six of these trifluralin lignin fraction solutions are passed through four layers of cotton fiber. A marked loss of lignin was noted in fraction PC950.

The tank mix combinations were replicated eight times. Four replications were placed under direct sunlight for 12 hour. After 12 hours of direct sunlight the pots were moved into the greenhouse. Four replications were retained in the greenhouse throughout the study. Weather conditions on the day of direct exposure were: RH 34%, wind 9 MPH, temperature high and low 22 C and 1 C respectively.

Bean and beet farm field study (Table 4). Field studies established near Saginaw, Michigan measured the efficacy of various lignin slurry solutions tank mixed with either triallate (S-(2,3,3-trichloroallyl)diisopropylthiocarbamate) or the trifluralin. Applications were made using a four nozzle boom attached to a backpack sprayer. Triallate was applied at 1.12 kg ha^{-1} and trifluralin at 0.86 kg ha^{-1}. Tank mixes were made at a 3:1 ratio of lignin to active ingredient. Treatments were replicated four times on 1.8 by 6 m-plots. Treatments consisted of non-treated control, trifluralin, triallate and one of the preceding herbicides mixed with slurries of PC950W, PC940 or REAX. The commercial formulation treatments were replicated eight times with four replications having the treatments incorporated and four unincorporated.

After application the herbicides were incorporated with a springtime harrow. Oats and soybeans were seeded into all plots. Thirty days after treatment and planting quadrants were randomly placed within each plot. The oat foliage within the quadrant was harvested and weighed. The weights from the treated plots were divided by the average weight from the non-treated control to give a percent of control value.

Barnyardgrass coleoptile node bio-assays (Tables 5 and 6). The studies were designed to allow multiple planting dates each spaced at varying intervals from a single application. The bottoms of 500 ml plastic pots were removed and replaced with a double layer of cheese cloth. The smaller pots were filled with air dried and screened Spinks sandy loam soil. Applications were made to the smaller pots which were then placed on top of 1-L pots filled with the same soil.

By assuring good contact between the cheese cloth and the soil of both pots the surface of the smaller pot was moistened by sub-irrigation. Seeds were sown by lifting the 500 ml pots and placing 20 seeds on the surface of the lower pot. The barnyardgrass seedlings were then allowed to grow through the treated soil.

The study was carried out in the greenhouse under natural lighting supplemented from high pressure sodium lights. Supplemental lighting provided 500 uE m^{-2} s^{-1} of light and combined with natural lighting would reach 1200 uE m^{-2} s^{-1}. Supplemental lighting was run for 12 hrs from 6 AM until 6 PM.

The lignin-trifluralin formulations were made by combining technical grade trifluralin with dry lignin fractions. The dried lignins were ground with the herbicide, heated to temperatures ranging from 70 to 80 degrees Celsius for 30 to 50 minutes and ground again. All lignin-trifluralin treatments used 0.84 kg ha^{-1} of the herbicide and a 2.52 kg ha^{-1} rate of lignin.

All lignin and lignin-herbicide applications were made by spreading 13 mg of the formulation across the soil surface.

The commercial emulsifiable concentrate of trifluralin was applied with a aqueous carrier. Application parameters were as stated in the general methods.

Barnyardgrass seedlings were sown on four different dates. Treatments were replicated four times in a completely randomized design. Evaluations were taken approximately 3 weeks after the seeding and entailed measuring the length of the shoots.

The impact of cross-linking, and oxidizing on control release properties of the lignins was explored by testing the fractions 5528-60 C, D, 5528-61 A, B and C (Table 4). The fractions were altered prior to the addition of trifluralin.

The lignin fractions utilized in study results presented in table 5 include lignins derived from peat, hardwood, pine or altered by methylation or cross-linking.

Corn bio-assays (Tables 7 through 13). All corn bioassay studies used Pioneer 3320 as the assay species. Four seeds were planted per pot directly on the treated soil surface. The seed was planted with the radical facing the center of the pot and the embryo facing up. For all applications involving lignin formulations and commercial formulations applied to the soil surface the seeds were placed directly on top of the treated soil, then covered with non-treated soil. Corn seeds were planted in a 3 to 4 cm bed of treated soil placed above none treated soil for the incorporated studies. Applications were made to dry soils but soils were sub-irrigated after application moistening the surface within 1 to 2 hours of application. The herbicide applications were made on dry soil in study presented in Table 13. In a deviation from the previous studies the soils in study 13 were left dry until seeding.

Lignin, sand or rosins were combined with the technical grade herbicides in a 3:1 ratio (matrix to herbicide) unless specified differently in the table. Lignin-trifluralin formulations used in the study presented in Table 13 examined the shelf life of the lignin-trifluralin formulation. The older formulation was formed on 10/8/85, the treatment titled new was formulated on 10/31/85.

The dried lignin or sand was ground with the herbicide, heated to temperatures ranging from 70 to 80 degrees C for 30 to 50 min and ground again. The results provided in Table 11 describe the effects of combining technical grade trifluralin with lignins without heating the mixture. Treatments designated by a lignin fraction description but followed by data collected on a single date were tests designed to identify the phytotoxicity of the lignin fraction.

All lignin studies used a 0.84 kg ha^{-1} rate of trifluralin or ethalfluralin and a 2.52 kg ha^{-1} rate of lignin. All lignin and lignin-herbicide applications were made by evenly spreading 13 mg of the formulation across the surface of the pot. Concentrations varied depending on the ratio of inert material to herbicide, for mixtures requiring less than 13 mg, talc was added to facilitate handling. The commercial formulation of trifluralin was applied as stated in the general methods.

Micro column test. A glass wool plug was placed at the base of a Pasteur pipet to hold 50 mg of 3:1 mixture of lignin-trifluralin or sand-trifluralin added to the pipet. A steady stream of N_2 was passed through a water bath maintained at room temperature and then through the column. The Pasteur pipets were kept in a growth chamber maintained at 38 C. The flow rate of the N_2 was maintained at 100 ml min^{-1} as possible. The concentration of trifluralin released was linear in this system over a range of 60 to 240 ml min^{-1}. The flow rates were measured at the beginning and end of each time period. The average time was used to determine the amount of trifluralin released per 100 ml N_2 min^{-1}.

The tapered end of the Pasteur pipet was passed though a polyurethane plug. The plug was placed in the neck of a scintillation vial. The N_2 vented into the vial was collected in the polyurethane. The polyurethane plug was removed from the vial and placed in a glass tube. Three 5 ml aliquots of acetone were passed through the tube and each collected separately. Preliminary studies demonstrated that the trifluralin was completely removed after the second 5-ml aliquot.

The acetone solution was injected into an HPLC system equipped with a 25 cm ODC column The column was run under isocratic conditions with an acetonitrile:water (80:20) mobile phase set at 1 ml min^{-1}. Data was reported as ug trifluralin (100 ml N_2 min^{-1})$^{-1}$.

RESULTS AND DISCUSSION

The surface application of the lignin fraction PC955A tank mixed with EPTC reduced barnyardgrass shoot length to a greater degree than the similarly applied non-tank mixed EPTC (Table 1). All non-incorporated applications of EPTC either tank mixed with lignins or not, gave poor barnyardgrass control. As a result of the large gap in phytotoxicity between the best lignin-EPTC surface and the incorporated EPTC application future studies focused on a pesticides with lower vapor pressures.

Differences in phytotoxicity appeared to be attributable to the lignin fractions in the initial lignin trifluralin study (Table 2). The first planting was done the same day as the application and in 100 percent control for all treatments (data not shown). Two surface applied lignin-trifluralin treatments (PC950 and PC58C) yielded results comparable to the incorporated treatments when seeds were sown 17 days after treatment (DAT). At the conclusion of the third planting incorporated treatments were providing a superior control when compared to all non-incorporated treatments. Differences between surface treatments not noted at the 17 DAT planting developed at the conclusion of the third planting. Lignin trifluralin formulations; PC952, and 58C reduced barnyardgrass shoot growth more than the similarly applied trifluralin treatment.

The third study was designed to continue exploring the controlled release properties of lignin and to determine if the lignins might enhance the photodegradation of trifluralin (Table 3). Ten of the 1. Eleven of the 20 least effective treatment were left in the greenhouse throughout the study and ten of the 19 most effective treatment were left in the greenhouse throughout the study. The exposure to the sun did not impact the efficacy of trifluralin. Though, differences were observed between lignin fractions the spread from the greenhouse control and the surface treatment yielding the greatest control (PC952) was 3.2 cm. In contrast, the spread form the best surface treatment (PC952) to the incorporated trifluralin was 4.5 cm. The probability of any given lignin yielding dissimilar results appear about even. Comparing direct and indirect light exposed treatments to one another 10 of 19 were significantly different. Under these conditions the observed differences between lignins though significant fail to demonstrate consistency or a comparable degree of efficacy to the incorporated treatments.

The field study confirmed that the slurried lignins failed to yield weed control comparable to the incorporated treatments (Table 4). The response of triallate to tank mixes was similar to that observed for EPTC and trifluralin. In contrast to previous studies no absolutely no differences could be distinguished between the lignins used (PC940 and PC950W).

By allowing the barnyardgrass to grow through, rather than on top of, treated soil the next set of studies more closely imitated actual field conditions (Table 5 and 6). It was assumed that talc would demonstrate little or no adsorptive properties. Thus the trifluralin formulated with talc provided a comparison between the application techniques. The commercial formulation of trifluralin was applied with water as the carrier. The lignin and talc formulations were applied as a dry powder by hand. The phytotoxicity resulting from the talc formulation was statistically identical to that caused by surface applied commercial trifluralin formulation. The lignin appeared to extend the effectiveness of the trifluralin through 8 days. Certain lignins were more effective than others but at no time did the lignin formulations approach the effectiveness of the incorporated treatments.

In the next 8 studies corn was used as the bioassay species. Lignin's lack of phytotoxicity is verified in the first study (Table 7). No differences between the lignin formulations and the commercial formulation were observed when applied in an identical manner.

The controlled release properties of PC950W on ethalfluralin were explored in the next study (Table 8). At 14 days after application all four treatments were statistically distinct with PC950W providing extended control over the comparably applied commercial ethalfluralin formulation.

The controlled release properties observed in PC950W resulted from the formulation process (Table 9). The slurried PC950W when tank mixed with ethalfluralin produce a negative impact on the efficacy of the surface application. The newly formulated lignin ethalfluralin slightly improved the efficacy of the surface application.

The extended efficacy noted in the last experiment was not duplicated when trifluralin was substituted for ethalfluralin (Table 10). Altering the ratio of PC950W to trifluralin failed to consistently extended weed control (Table 11). Liquefying the trifluralin in the presence of PC950W by applying heat failed to improve the controlled release properties of the formulation. The relatively low melting point of trifluralin and the ability of the liquified technical grade material to dissolve most lignin fractions aided the formulation processes. The lignin trifluralin formulation treatments in this study once again demonstrated a slight extension of the efficacy of trifluralin. Once again no lignin trifluralin formulation approached the efficacy provided by the incorporated treatments. Differences between the surface applications were not observable when the loamy sand was replaced with a clay soil (Table 12). Differences were not observed when a new preparation of PC950W was compared to a month old formulation (Table 13).

The lignins appeared to provide an extension of the efficacy of trifluralin in the greenhouse studies. The extension could not be linked to a specific lignin, the intensity varied from weak to negligible, never approaching the results yielded by incorporated treatments. The controlled release properties noted were not apparent in reproducible form in the tank mixes involving slurried lignin and herbicide. Greater consistency was observed when technical grade herbicide was formulated with dry lignins. Attempts to extend the efficacy by increasing the concentration of lignin or the formulation process under these conditions failed.

Results of the corn bioassay indicate that mixing rosin with technical grade trifluralin results in complete or nearly complete retention of the herbicide (Table 14). All lignins examined to date originated from the Kraft pulping process. The lignin identified as BEC was derived from a pulping process using organic solvents. BEC when formulated with trifluralin yielded results similar to the Kraft lignins.

The laboratory study partially confirmed the greenhouse results. A sand trifluralin mixture yield a similar level of the herbicide as a three lignin trifluralin formulations. The laboratory test lacked the sensitivity of the greenhouse studies but confirmed that the degree of controlled release was at best slight.

Table 1. Controlled release properties of lignins tank mixed with EPTC.

	Barnyardgrass
Treatment (lignin)	----------Shoot length---------- ---(cm)---
Nontreated control	10.1 A[1]
PC951A[2]	9.9 A
37D	9.7 AB
PC953	9.6 AB
PC949	9.2 ABC
EPTC[3](surface)[4]	8.8 ABC
PC951	8.7 ABC
PC955B	8.7 ABC
PC954	8.4 BC
PC951C	8.3 BCD
PC950	8.3 BCD
PC952	8.3 BCD
PC955C	7.9 CD
PC955A	6.9 D
EPTC (incorporated)	0 E

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[1] Means followed by the same letter are not significantly different according to Duncan's multiple range test at the 0.05 level.

[2] All lignin fractions were tank mixed with the EPTC formulation at a 1:3 ratio lignin dry weight to active ingredient weight of the herbicide.

[3] Trade name for the ICI Americas emulsifiable concentrate of EPTC was EPTAM.

[4] EPTC applications were either incorporated in the soil immediately after application or surface applied without incorporation as were the lignin applications.

Table 2. Controlled release properties of lignin tank mixed with trifluralin[1].

Treatment (lignin)	17 DAT Planting --Shoot length-- ---(cm)---	Treatment (lignin)	36 DAT Planting --Shoot length-- ---(cm)---
Nontreated cont.	9.6 A	Nontreated cont.	9.5 A
22D	5.0 B	37DSL	9.1 AB
PC949	4.8 B	19	8.5 ABC
PC954	4.1 BC	PC949	8.2 BCD
PC957	3.7 CD	Treflan (surf.)	8.2 BCD
PC922	3.0 DE	PC956	7.9 CDE
PC954	2.9 DE	22D	7.8 CDE
PC956	2.8 DE	58A	7.7 CDE
58B	2.7 EF	37D	7.6 CDE
Trifluralin(surf.)[3]	2.6 EFG	PC922	7.6 CDE
PC925	2.5 EFG	PC957	7.6 DEF
37DSL	2.4 EFGH	PC953	7.1 DEF
37D	2.3 EFGH	58B	6.9 EFG
58A	2.2 EFGHI	PC954	6.8 FGH

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1. Trade name for the DowElanco emulsifiable concentrate of trifluralin.
2. Average values followed by the same letter are not significantly different according to Duncan's multiple range test at the 0.05 level. The data is analyzed using a two way ANOVA comparisions between dates are not intended.
3. All lignin fractions were tank mixed with the Treflan formulation at a 1:3 ratio lignin dry weight to active ingredient weight of the pesticide.
4. EPTC applications were either incorporated in the soil immediately after application or surface applied without incorporation as were the lignin applications.

Table 2 continued. Controlled release properties of lignin tank mixed with Trifluralin[1].

Treatment (lignin)	17 DAT Planting --Shoot length-- ---(cm)---	Treatment (lignin)	36 DAT Planting --Shoot length-- ---(cm)---
37DSL	2.4 EFGH	PC953	7.1 DEF
37D	2.3 EFGH	58B	6.9 EFG
58A	2.2 EFGHI	PC954	6.8 FGH
37DSL (incorp.)	1.7 FGHI	PC952	5.9 GH
PC825 (incorp.)	1.6 GHI	Treflan (incorp.)	1.6 I
Treflan (incorp.)	1.4 HIJ	37DSL (incorp.)	1.5 I
PC58C	1.4 HIJ	PC954 (incorp.)	1.3 I
PC950	1.3 IJ		
PC954 (incorp.)	0.6 J		

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3. All lignin fractions were tank mixed with the Treflan formulation at a 1:3 ratio lignin dry weight to active ingredient weight of the pesticide.
4. EPTC applications were either incorporated in the soil immediately after application or surface applied without incorporation as were the lignin applications.

Table 3. Controlled release properties of lignin as impacted by direct sunlight.

Treatment (lignin)	--Shoot length-- ---(cm)---	Treatment (lignin)	--Shoot length-- ---(cm)---
Control[2] G[3]	9.4 A	PC922HW O	7.3 G-O
58C O[4]	9.3 A	PC950N O	7.3 G-O
PC955C G	8.8 AB	PC955B G	7.3 G-O
PC922HW G	8.7 AB	PC955B O	7.3 G-O
PC949 O	8.5 BC	PC951A G	7.3 G-O
PC955C O	8.2 B-E	PC951B G	7.3 G-O
PC940C G	8.1 B-F	PC952 O	7.2 H-O
5528 60A G	8.0 C-G	PC922H O	7.1 I-O
PC950 G	7.9 C-H	PC940C O	7.1 J-O
58C G	7.9 C-H	PC950 O	7.1 J-O
PC949W O	7.9 C-I	5528 60A O	7.1 J-O
PC922H G	7.8 C-J	PC949 G	7.0 K-O
Treflan (surf.) O	7.8 D-J	PC950W G	7.0 L-O
PC951B O	7.7 E-K	PC922L O	6.9 L-O

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3. All lignin fractions were tank mixed with the Treflan formulation at a 1:1 ratio lignin dry weight to active ingredient weight of the pesticide.
4. EPTC applications were either incorporated in the soil immediately after application or surface applied without incorporation as were the lignin applications.

Table 3 continued. Controlled release properties of lignin as impacted by direct sunlight.

Treatment (lignin)	--Shoot length-- ---(cm)---	Treatment (lignin)	--Shoot length-- ---(cm)---
Control O	7.6 E-L	PC955A G	6.8 L-O
PC951C O	7.5 E-L	PC949N G	6.8 M-P
PC922LW O	7.5 F-M	PC922L G	6.7 NOP
Treflan (surf.) G	7.4 G-N	PC922LW G	6.6 OP
PC949W G	7.4 G-N	PC952 G	6.2 Q
PC951C G	7.4 G-O	Treflan (inc.)	1.7 R
		Treflan (inc.)	1.5 R

1. Trade name for the DowElanco emulsifiable concentrate of trifluralin.
2. Average values followed by the same letter are not significantly different according to Duncan's multiple range test at the 0.05 level.
3. All lignin fractions were tank mixed with the Treflan formulation at a 1:1 ratio lignin dry weight to active ingredient weight of the pesticide.
4. EPTC applications were either incorporated in the soil immediately after application or surface applied without incorporation as were the lignin applications.

Table 4. Oat-field study testing the controlled release properties of tank mixes lignin with triallate or trifluralin.

Treatment	Shoot weight (% of control)
PC040:Treflan	92[3]A
PC950W:Treflan	92 A
Treflan[4](surface)	91 A
REAX:Treflan	86 A
Far Go (surface)	82 A
PC950WP:Far Go	71 A
PC940:Far Go	71 A
Treflan (incorporated)	34 B
Far Go (incorporated)	9 B

1. Triallate was applied or added in tank mixes with the lignins as the commercial product Far Go an emulsifiable concentrate marketed by Monsanto.

2. Trifluralin was applied or added in tank mixes with the lignins as the commercial product Treflan an emulsifiable concentrate marketed by DowElanco

3. Average values followed by the same letter are not significantly different according to Duncan's multiple range test at the 0.05 level.

4. Lignin herbicide tank mixes were not soil incorporated.

Table 5. Release rate of trifluralin form crosslinked (5528 - 60 B-E) and oxidized (5528-61 A-B) Kraft lignins.

	----- Shoot length (cm/plant) -----				
Treatment	2 DAT	5 DAT	8 DAT	14 DAT	24 DAT
Nontreated control	10.0A[1]	14.8A	14.9A	14.0A	26.9A
Trifluralin (surf)	1.2AB	2.2BC	6.8AB	11.8AB	17.8A
Talc:trifluralin	4.0AB	2.6B	5.2AB	10.7AB	11.7ABC
5528-60 C	0.2CD	0.7DE	0.8D	3.2AB	5.2C
5528-60 D	0.7BC	1.1BCD	1.6CD	4.7AB	11.7ABC
5528-60 E	2.0AB	1.1BCD	1.3CD	0.1C	17.0AB
5528-61 A	2.6AB	0.4DE	0.6D	2.1AB	9.8ABC
5528-61 B	3.6AB	0.7CDE	3.0BC	8.1AB	5.9BC
5528-61 C	0.1D	0.3E	0.5D	1.2B	13.8A
Trifluralin (inc.)	0.0E	0.0F	0.0E	0.0D	0.0D

[1] Values with a single column (DAT) are comparable. Means followed by the same letter are not significantly different according to Duncan's multiple range test at the 0.05 level.

Table 6. The release rate of trifluralin form lignin series 531-75 (A-E) and Indulin W.

	----- Shoot length (cm/plant) -----				
Treatment	4 DAT	8 DAT	12 DAT	16 DAT	26 DAT
Nontreated control	17.2A[1]	11.3A	11.2A	18.7A	31.6A
PC951A	1.3E	1.3C	1.2E	1.8D	24.5AB
Trifluralin(surface)	1.4E	1.6B	8.6AB	12.1AB	29.5A
Talc:trifluralin	7.7B	1.6B	3.4BCD	7.4ABC	23.4AB
5528-60 A	1.4E	1.4B	3.1CD	7.5ABC	26.9AB
Indulin W	5.8BC	1.6B	3.8BCD	7.5ABC	18.6AB
5531-75 A	1.6DE	1.3B	4.9ABC	2.9CD	18.2AB
5531-75 B	6.3BC	1.8B	3.0CD	7.0ABC	18.2AB
5531-75 C	3.5CD	1.7B	2.8CD	4.1CD	24.0AB
5531-75 D	5.9BC	1.2B	4.9ABC	5.7BC	22.4AB
5531-75 E	2.4DE	1.1B	1.5DE	3.2CD	-----
Trifluralin(incorp.)	0.0F	0.0D	0.0F	0.0E	0.0B

1. Values with a single column (DAT) are comparable. Numbers followed by the same letter are not significantly different according to Duncan's multiple range test at the 0.05 level.

Table 7. Phytotoxic properties of PC940, PC922, PC940:trifluralin, PC922:trifluralin as evaluated by corn root assay.

	----- Root length in cm -----					
Treatment	1 DAT	2 DAT	4 DAT	5 DAT	7 DAT	11 DAT
Nontreated control	16.7A[1]	27.13A	24.6A	19.4A	21.1A	24.9A
PC 940[2]	24.5A					
PC 922	23.2A					
PC922:trifluralin	3.9B	3.4BC	3.5B	9.1B	9.2B	14.6C
PC940:trifluralin	2.8B	6.0B	3.7B	6.0BC	8.5B	17.8BC
Treflan[3](surface)	2.3B	3.5BC	3.4B	5.3BC	8.2B	19.1B
Treflan (incorporated)	2.3B	2.3C	2.4C	3.0C	3.1C	3.5D

1. All analysis done on the log transformed root length data. Values with a single column (DAT) are comparable. Means followed by the same letter are not significantly different according to Duncan's multiple range test at the 0.05 level.

2. PC950W and PC940 applications made without trifluralin to evaluate the lignin phytotoxicity

3. Trade name for the DowElanco emulsifiable concentrate of trifluralin

Table 8. The rate of release of ethalfluralin from the Kraft lignin fraction PC950W.

	Root length					
		Time				
	0 DAT	3 DAT	5 DAT	8 DAT	14 DAT	21 DAT
Treatment		(cm)				
Control (nontreated)	15.4[1]A	12.5 A	13.3 A	20.4 A	15.0 A	17.7 A
PC950W: ethalfluralin	2.0 B	2.6 C	4.2 B	5.1 B	4.9 C	5.9 C
Sonalan (surface)	1.7 B	2.4 C	6.0 B	5.4 B	6.9 B	11.8 B
Sonalan (incorporated)	1.2 B	3.9 B	3.2 B	3.3 C	3.1 D	3.7 D

1. Values with a single column days after treatment (DAT) are comparable. Numbers followed by the same letter are not significantly different according to Duncan's multiple range test at the 0.05 level.

2. Trade name for the DowElanco emulsifiable concentrate of ethalfluralin.

Table 9. Controlled release properties of dry and slurried lignins for ethalfluralin.

	Root length	
	Time	
Treatment (formulation)	1 DAT	2 DAT
Non-treated control	21.6[3]A	15.0 A
PC950W S[1]:ethalfluralin	10.8 B	14.5 A
PC950W D[2]:ethalfluralin	3.7 C	3.1 C
Ethalfluralin[4](surface)	2.6 CD	4.5 B
Ethalfluralin(incorporated)	2.0 D	1.9 D

1. Lignin is mixed as a slurry. The slurry was mixed at a ratio that provides a 3:1 ratio of lignin (dry weight) to ethalfluralin.

2. Lignin is mixed as a dry material to ethalfluralin by grinding, heating and grinding.

3. Values with a single column (DAT) are comparable. Numbers followed by the same letter are not significantly different according to Duncan's multiple range test at the 0.05 level.

4. Trade name for the DowElanco emulsifiable concentrate of ethalfluralin was Sonalan.

Table 10. Controlled release properties of dry and slurried lignins for ethalfluralin.

Treatments	1 DAT	2 DAT	3 DAT	5 DAT	14 DAT	21 DAT
	Root length in cm					
Non-treated control	14.9A[1]	21.7A	18.0A	15.0A	19.8A	20.5A
PC950W	12.5A[2]	----	----	----	----	----
PC950W:trifluralin	3.1B	3.6B	3.2C	3.4B	9.5B	12.6B
Treflan[3](surface)	2.6B	3.6B	4.5B	3.2B	8.8B	10.8B
Treflan (incorporated)	2.1C	3.1B	2.1D	2.1B	3.4C	3.9C

1. All analysis done on the log transformed root length date. Values with a single column (DAT) are comparable. Numbers followed by the same letter are not significantly different according to Duncan's multiple range test at the 0.05 level.

2. PC950W application made without trifluralin to evaluate the lignin phytotoxicity

3. Trade name for the DowElanco emulsifiable concentrate of trifluralin.

Table 11. Controlled release properties of lignin for trifluralin as effected by ratio and formulation procedures.

Treatment (formulation)	5 DAT	6 DAT	11 DAT	12 DAT
	----------- Root length on cm -------------			
Nontreated control	20.0[1]A	21.6 A	16.7 A	17.6 A
PC 950W Trifluralin heated 6:1	9.0 BC	4.0 BC	10.4 BC	15.0 AB
PC 950W Trifluralin not heated 3:1	7.7 BCD	5.5 B	12.1 B	15.7 AB
PC 950W Trifluralin not heated 6:1	6.1 CDE	3.4 C	8.7 BC	11.1 B
PC950W Trifluralin heated 3:1	4.8 DE	3.5 C	7.8 C	12.1 B
Treflan[2](surface)	11.4 B	6.0 B	17.3 A	14.5 AB
Treflan (incorporated)	3.9 E	3.5 C	3.6 D	3.5 C

1. All analysis done on the log transformed root length data. Values with a single column (DAT) are comparable. Numbers followed by the same letter are not significantly different according to Duncan's multiple range test at the 0.05 level.

2. Trade name for the DowElanco emulsifiable concentrate of trifluralin.

Table 12. Controlled release properties of lignin for trifluralin on a moist clay surface.

Treatments (formulation)	3 DAT	6 DAT	10 DAT	15 DAT
	----- Root length in cm -----			
Nontreated control	21.1 A[1]	21.8 A	25.5 A	24.8 A
PC950W:trifluralin	7.3 C	6.9 B	13.4 B	13.8 B
Treflan[2](surface)	12.0 B	5.3 B	16.4 B	12.6 B
Treflan (incorporated)	4.3 C	4.1 B	4.2 C	4.6 C

1. All analysis done on the log transformed root length data. Values with a single column (DAT) are comparable. Numbers followed by the same letter are not significantly different according to Duncan's multiple range test at the 0.05 level.

2. Trade name for the DowElanco emulsifiable concentrate of trifluralin.

Table 13. Shelf life of lignin trifluralin formulations.

Treatment	Root length 3DAT (cm/plant)	7DAT	14DAT	19DAT
Non-treated control	24.1A[1]	27.9A	17.8A	25.6A
PC950W:trifluralin old[2]	2.6C	2.2C	2.2B	2.2B
PC950W:trifluralin new[3]	2.5C	2.4C	2.7B	2.3B
Treflan surface	2.7C	2.3C	2.5B	2.4B
Treflan incorporated	3.6B	3.4B	3.2B	4.0B

[1] All analysis done on the log transformed root length data, values with a single column (DAT) are comparable. Numbers followed by the same letter are not significantly different according to Duncan's multiple range test at the 0.05 level.

[2] Formulation prepared on 10-8-85.

[3] Formulation prepared on 10-31-86.

Table 14. Release rates of trifluralin from lignins (Kraft and organosolvent derived) and other inert materials.

	----- Root length -----			
	9 DAT	13 DAT	17 DAT	22DAT
Treatment	----- (cm/plant) -----			
Non-treated control	11.2A[1]	10.2A	9.1A	9.1A
Rosin:trifluralin 2:1	11.7AB	11.7A	9.8A	10.0A
Rosin:trifluralin 1000:1	12.6A	9.5A	9.5A	9.3A
Rosin:trifluralin 10:1	10.7AB	9.5A	9.1A	8.5A
BEC:trifluralin 1:1	0.9D	2.4BC	5.5B	8.7A
BEC:trifluralin 3:1	1.1CD	1.1C	5.2B	8.5A
Talc:trifluralin 3:1	3.3BC	4.1B	5.2BC	8.5A
PC951:trifluralin 3:1	0.9D	2.9B	3.2C	7.8A
Treflan (surface)	11.0AB	9.8A	9.5A	8.9A

1. All values with a single column (DAT) are comparable. Numbers followed by the same letter are not significantly different according to Duncan's multiple range test at the 0.05 level.

Table 15. The release rate of trifluralin from lignin and other inert materials as measured by a laboratory assay.

Treatment	1-13	13-25	Time (min) 25-37	37-49	49-56	Total Released
	-----------		ug/(100/ml N_2/min)	-------------		--(%)--
Sand[1]	36(7)[2]	48(3)	44(4)	40(6)	37(2)	76.3
PC950W	34(1)	45(1)	46(1)	45(1)	48(2)	74.9
PC940	34(3)	41(2)	41(4)	38(1)	41(5)	68.8
PC922	34(2)	49(3)	47(2)	48(2)	55(3)	78.9

1. Sand and lignins are prepared by mixing at a 3:1 ratio inert matrix to trifluralin.

2. The average value of three replicates is followed by the standard deviation of the three values.

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