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Mill Residue and Byproduct Utilization Project

1996 Annual Report

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and
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Second Annual Report
January 1997

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Summary

Support for the project remained strong during 1996 with direct industrial funding totaling $105,000. This support was augmented by substantial in-kind contributions. State support for the project, awarded on July 1, 1996, totaled $101,105 to UGA and $ 39,446 to Georgia Tech. This was $83,000 less than requested and resulted in postponement of some field and analytical activities.

Initial sampling of mill residue streams from 11 participating mills was completed in early 1996. Eight of these mills were re-sampled this year. A total of 47 residue streams have been characterized for nutrients, metals and Toxicity Characteristic Leaching Procedure (TCLP) metals, organics and pesticides. Soxhlet extractions of organics residues have not been completed. These analyses will be rescheduled for 1997. Characterization data indicate that inorganic residues (ash, lime mud, slaker grits and green liquor dregs) have characteristics that make them valuable as agricultural lime substitutes. Calcium carbonate equivalencies are high and concentrations of metals or leachable contaminants that may restrict use of these materials have been found in only two samples. Some sources of ash and slaker grit are also high in P and K and could serve as substitutes for these fertilizers. Both primary and secondary mill biosolids are highly variable in composition. Most secondary biosolids have N and P contents high enough to warrant use as N and P sources in forest plantations. As with inorganic residues, concentrations of metals and leachable contaminants are low in these materials. One mixed secondary sludge had high Pb concentrations.

One of two greenhouse studies of pine seedling response to residue addition was completed in 1996. A second pine greenhouse study was installed in June and will be completed in early 1997. Addition of inorganic mill residues was found to have little effect (positive or negative) on seedling growth in the first experiment. Growth response and soil conditions following addition of lime mud, slaker grits and green liquor dregs were generally similar to those associated with addition of agricultural lime. In addition to increased Ca, soil analyses indicate that measurable increases in K and Mg concentrations can be expected following addition of ash. Soil sodium concentrations did not generally differ among residue treatments and the untreated control or agricultural lime-treated soils.

In contrast to inorganic residue additions, addition of secondary and mixed mill biosolids generally improved seedling growth above untreated controls or soils treated with commercially available soil amendments. Much of this benefit appears due to increased N availability, but foliar concentrations of several other nutrients were also improved by organic residue addition.

Installation of forestry field trials was completed at two sites in 1996: an Upper Coastal Plain site near Oglethorpe, GA and a Lower Coastal Plain site near Rincon, GA. At each of these sites, selected inorganic and organic residues were applied at two rates to both established mid-rotation stands and to harvested stands that were site prepared and planted following residue addition. Residues were spread on the Oglethorpe site in mid-February, and on the Rincon site in October. The Oglethorpe site was planted in late February but prolonged April drought and heavy weed re-growth reduced survival below acceptable levels. This site was replanted in January 1997. The Rincon site was planted in late November 1996. An experimental use permit application for the Savannah River field trial site was submitted to SC DHEC in June in anticipation of site installation during January 1997. We have not yet received permit approval and installation of this site is currently on hold. Preparations for

installation of the Lower Coastal Plain flatwoods site are proceeding on schedule. This site will be installed in 1997.

Two greenhouse studies and three field studies of crop growth response to residue additions were also completed in 1996. Use of mill residues as lime substitutes was studied both in the greenhouse and field. Results indicate that pH adjustment can be achieved using mill residues; however, soil pH adjustment may occur more slowly with residues than with agricultural lime. In a two-month greenhouse study, soil pH at the end of the experiment was lower in pots treated with residues applied at equivalent rates than in pots treated with agricultural lime. In contrast, in field trials that lasted a full growing season, target soil pH was achieved equally well using either agricultural lime or mill residues. In the field study, all inorganic mill residue treatments were associated with wheat growth equal to, or exceeding, growth on plots treated with agricultural lime.

Four special projects were initiated as graduate student thesis or dissertation research during 1996. The first of these is a study of P availability in wood ash and the use of wood ash as a P fertilizer substitute in young loblolly pine plantations. This research is being completed by Catherine Merz as her MS thesis. Catherine's initial results indicate that P in ash is readily available, even at the higher soil pH associated with ash addition. Alanna Conley, an MS student working with Dr. Nutter, is evaluating soil conditions and metal movement at the Oglethorpe study site. Herwig Goldemund, a Ph.D. student working with Drs. Morris and Pennell, is conducting research on the movement of inorganic and organic compounds in residue-amended sites. Herwig will be evaluating movements of selected inorganic compounds in the laboratory and using the LEACHM model to predict how soil and climatic condition will affect movement at the Oglethorpe and Rincon field sites. A fourth project is being conducted by Rachel Cochran as her MS thesis research with Dr. Miller. Rachel is evaluating the effects of pulp and paper mill residues on soil physical properties. Initial results from Rachel's research have shown that additions of organic mill residues lead to improved aggregate stability and reduced erosion potential.

The 1997 project budget assumes annual support from each industrial member will remain at $15,000 and that State support will total $74,000. State support is not expected past 1997. Additional membership will be required to maintain project activity at current levels after 1997.

Characterization of Mill Residues

Initial sampling of 47 residue streams from 11 mills was completed by project personnel in early 1996. Eight of these mills were re-sampled by company personnel between March and December using protocols established during the initial sampling. Nutrient, metal and Toxicity Characteristic Leaching Procedure (TCLP) analyses have been completed for the initial sampling.

Sampling Methods

Samples were collected from each selected residue stream and sampled according to EPA protocols. Depending upon the nature of the residue and process, samples were collected using either temporal or spatial sampling. For temporal sampling, each process stream was sampled 17 times over a one week period. Three samples were collected on each of 4 days and 5 samples collected on one day. Daily samples were combined in a weekly composite sample. Spatial sampling was utilized to collect samples from ASB lagoons, compost piles and storage areas. Generally, these areas were stratified and samples collected along randomly located transect lines. EPA approved and certified clean glass bottles (8 oz) were used for daily sampling. Weekly composites were made from these and stored in 16 oz bottles. An additional certified clean glass bottle was used to store archive samples. Bulk samples were collected in five gallon plastic buckets for greenhouse studies. Samples were brought to the University of Georgia, School of Forest Resources and stored in a locked, refrigerated room at 4 °C. Weekly composites of each mill's residues were split and transported to the participating laboratories for testing. Chain-of-custody records have been maintained on each split sample.

During the sampling period at each mill, a separate sample of the mill's primary residue stream was composited. This residue could be either the highest volume category generated by the mill, or the residue which the management considered the highest priority for land application. This residue sample was signed over to the designated contact person at each mill and sent for dioxin testing.

Laboratory Analyses

General Characterization: Reaction (pH) was determined on subsamples of the weekly composite in a deionized water paste. Samples were characterized for water content, volatile solids and ash by loss-on-ignition at 110 °C, 175 °C and 500 °C, respectively. Electrical conductivity (EC) was determined in filtrates from a 1:5 sample:deionized water paste following a 24 hour incubation. A set of splits from the weekly composites were digested in HNO_3 (EPA Method 3050) to prepare them for nutrient and metal analyses. These analyses included 40 CFR 503 listed metal ions as well as additional metals of interest. Subsamples from weekly composites were also sent to Dr. Shuman's fertility lab in Griffin for lime equivalency evaluation. Analyses for total Kjeldahl nitrogen and total phosphorus and plant nutrients were completed colorimetrically following digestion in sulfuric-selenous acid.

TCLP metals, pesticides and organics: An additional set of splits from the weekly composite samples were analyzed at the Cooperative Extension Services Laboratory in Athens, GA where they were extracted using EPA Toxicity Characteristic Leaching Procedure (TCLP, EPA Method 1311). This procedure was used to evaluate and classify mill residues for volatile organics, metals, and pesticides.

Soxhlet extractions: Additional soxhlet extractions and screening for nonvolatile and semivolatile organic compounds are planned for 1997.

Results

General Characterization: Results of the general characterization, total metal and TCLP analyses for the initial characterization sampling are presented in tables 1-4. In these tables, organic residues that did not contain a source of partially digested fiber were classified as primary biosolids. Some of these primary biosolids contained mixtures of inorganic materials not considered to be a source of N (e.g. lime mud). The secondary and mixed biosolid category included all residues that contained some portion of activated or partially digested fiber. With the exception of one secondary and mixed biosolid and wood flume grit, all residue materials were alkaline with pH ranging from 7.0-13.4. (Table 1). Lime equivalencies of inorganic residues, particularly lime mud, approached that of pure calcium carbonate. Nitrogen concentrations of primary materials ranged from 0.07% to 0.83%. Secondary and mixed biosolid N concentrations ranged from 0.24% to 1.98%. Phosphorus concentrations of several secondary and mixed biosolids were as high as 0.16% (1600 ppm) indicating that these materials may also be a suitable P source when applied at high rates. Several ash, lime mud and slaker grit samples had P concentrations greater than 0.3% (3000 ppm).

Metal concentrations were generally low in mill residues (Table 2). Generally, maximum concentrations for metals were lower in all residues than the pollutant limit established for land application of biosolids from municipal treatment facilities under 40 CFR Part 503. One ash exceeded these limits for As and Zn. This ash was from a boiler that burned tires as well as wood. Mo concentrations were exceeded in one unwashed slaker grit sample and Pb in one secondary and mixed biosolid. All of these residue streams have been re-sampled to determine if these higher concentrations are typical.

TCLP metals, pesticides and organics: With the exception of barium, which was detected at low concentrations in most samples, metals were below detection limits in most mill residue leachates. Low concentrations of As, Cd, Pb and Se were detected in one to three samples (Table 3). In each case, these were well below EPA limits established for hazardous materials. Volatiles, and semivolatile organics and pesticides of leachates were low for all samples (Table 4).

Table 1. Range in characteristics of 47 organic and inorganic mill residues collected from 11 mills during initial characterization sampling.

Residue Type	Sub-Type	n	Moisture Content	Volatiles[2]	Ash[2]	$CaCO_3$ Equiv.	pH	N	P
			%	-%-	-%-	-%-	-s.u.-	-%-	-ppm-
Inorganic Residues									
Ash	Mixed	8	2-85[1]	0-5.6	11.9-86.2	4.6-60.4	7.9-12.1	0.03-0.26	619-3213
	Bottom	1	18	0	3.6	23.6	13.4	0.05	992
	Fly	3	41-97	0-32.9	14.3-50.9	23.1-25.2	8.9-12.0	0.13-0.18	1085-2464
Lime Mud		7	19-41	0-1.6	1.5-5.6	95.8-100	10.4-12.4	0-0.2	465-5300
Slaker Grit	Unwashed	5	20-69	0-1.0	0-10.3	78.0-99.9	12.8-13.4	0.010.08	450-5000
	Washed	2	14-19	0.5-0.6	1.1-22.0	100	11.1-11.6	0.01	840-1349
Green Liquor Dregs		3	40-58	0-3.5	6.7-17.1	82.3-96.4	12.7-13.3	0.04-0.11	472-1937
Organic Residues									
Primary Biosolids		6	68-84	0.9-36.9	11.6-61.0	20.2-75.7	6.8-9.0	0.07-0.83	342-999
Secondary and Mixed Biosolids	Mixed	6	62-96	5.4-38.3	14.1-80.0	2.8-46.7	6.5-8.1	0.24-1.98	1054-1674
	Composted	1	67	7.6	37.7	41.8	7.4	0.58	1302
	ASB	1	70	3.28	28.8	50.5	7.6	0.36	1612
Knot Rejects	Washed	1	63	13.5	81.3	2.1	9.2	0.05	379
	Unwashed	1	62	23.7	37.9	7.2	8.9	0.09	425
	Composted	1	70	4.9	56.9	1.0	7.9	0.14	395
Wood Flume Grit		1	61	1.9	2.0		5.3	0.06	1029

[1] Ranges are for 11 mills.

[2] Volatiles expressed as % dry wgt of oven-dry (100°C); ash expressed as % dry wgt of volatile-free sample (175°C)

Table 2. Range in total metal concentrations of 47 inorganic and organic mill residues collectd during intial mill sampling.

Residue Type	Sub-Type	[illegible]	Ag	Al	As	Ba	Be	Cd	Co	Cr
			ppm							
40 CFR 503 Limits					*41*			*39*		*1200*
Inorganic Residues										
Ash	Mixed	8	bdl[1]-0.9	3017-19655	1.5-55.5	162-1544	bdl-4.8	bdl-2.6	bdl-29.5	bdl-47.3
	Bottom	1	bdl	11155	2.5	992	bdl	bdl	bdl	95.9
	Fly	2	bdl	5939-17780	9.8-11.7	71-1041	1.7-3.7	1.1	1.6-9.2	20.4-33.9
Lime Mud		7	bdl-0.1	591-1801	2.1-9.4	50-414	bdl-0.2	bdl-0.5	bdl-2.0	bdl-36.3
Slaker Grit	Unwashed	5	bdl-3.7	2001-9499	1.4-5.9	209-899	bdl	bdl	bdl	bdl-90.9
	Washed	2	bdl-0.3	752-4239	3.6-5.4	230-499	bdl-0.2	bdl-0.1	bdl-1.4	bdl-36.
Green Liq. Dregs		3	0.8-1.2	2769-5532	1.3-2.0	415-664	bdl	1.5-2.1	0.6-5.7	32.0-35.5
Organic Residues										
Primary Biosolids		5	bdl-0.7	604-7753	bdl-4.2	92-226	bdl-0.4	bdl-0.3	bdl-3.5	bdl-39.6
Secondary and Mixed Biosolds	Mixed	7	bdl-1.0	5353-18510	1.5-10.9	82-506	bdl-3.1	bdl-1.4	bdl-4.2	bdl-71
	Composted	1	bdl	6340	1.3	297	bdl	bdl	bdl	32.0
	ASB	1	3.2	12130	2.6	268	bdl	bdl	bdl	31.1
Knot Rejects	Washed	1	bdl	108	2.0	6.3	bdl	bdl	bdl	bdl
	Unwashed	1	bdl	1949	1.7	19.8	bdl	bdl	bdl	1.2
	Composted	1	bdl	652	1.6	14.3	bdl	bdl	bdl	bdl
Wood Flume Grit		1	0.5	730	4.3	6.4	0.2	0.1	0.6	27.9

[1] bdl = below detection limits.

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Table 2. continued.

Residue Type	Sub-Type	n	Cu	Mn	Mo	Ni	Pb	Sb	Se	Zn
			ppm							
40-CFR-503			*1500*		*18*	*420*	*300*		*36*	*2800*
Inorganic Residues										
Ash	Mixed	8	15-101	559-4317	0.2-5.5	0.5-128.4	0.8-45.9	bdl-1.6	bdl-8.9	48-**3010**
	Bottom	1	21	2861	11.4	8.6	94.2	0.8	bdl	46
	Fly	2	54-102	283-2506		29.6-54.8	26.0-30.7	bdl	5.0	149-170
Lime Mud		7	3-66	135-836	bdl-0.1	3.3-70.9	bdl	13.5	bdl-7.6	4-93
Slaker Grit	Unwashed	5	1-197	42-8533	bdl-**19.7**	5.8-84.4	bdl-25.5	bdl	bdl	8-742
	Washed	2	1-3	112-253	bdl-0.3	10.2-52.1	3.3-5.2	bdl-0.1	bdl-0.7	10-28
Green Liq. Dregs		3	78-169	2292-9467	1.5	34.3-155.8	5.7-30.2	bdl	4.9-6.1	440-709
Organic Residues										
Primary Biosolids		5	10-109	295-1327	bdl-1.9	1.9-36.4	1.9-13.2	bdl-0.5	bdl-0.8	54-237
Secondary and Mixed Biosolds	Mixed	7	15-124	330-1338	bdl-3.4	3.2-161.3	6.0-**492.2**	bdl	bdl	147-517
	Composted	1	37	1110	3.0	57.2	12.6	bdl	bdl	168
	ASB	1	45	1559	0.2	12.2	18.4	bdl	6.3	408
Knot Rejects	Washed	1	bdl	120.7	bdl	bdl	29.3	bdl	2.9	23
	Unwashed	1	5	84.2	1.1	2.5	112.7	bdl	1.1	23
	Composted	1	bdl	79	bdl	bdl	0.4	bdl	0.0	14
Wood Flume Grit		1	3	50.1	2.1	28.2	3.4	0.2	1.3	5.0

[1] bdl = below detection limits.

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Table 3. Toxicity Characteristic Leaching Procedure (TCLP) metal concentrations for organic and inorganic mill residues collected during initial characterization sampling[1]

Residue Type	Sub-Type	n	As	Ba	Cd	Cr	Pb	Hg	Se	Ag
			ppb							
Regulatory Limit			*5000*	*100,000*	*1000*	*5000*	*5000*	*200*	*1000*	*5000*
<u>Inorganic Residues</u>										
Ash	Mixed	8	bdl[1]-144.0	460-8700	bdl-14.0	bdl	bdl	bdl	bdl-69.3	bdl
	Bottom	1	bdl	2760	bdl	bdl	bdl	bdl	bdl	bdl
	Fly	2	bdl-5.9	320-3650	bdl	bdl	bdl	bdl	bdl	bdl
Lime Mud		7	bdl	130-883	bdl	bdl	bdl	bdl	bdl-18.0	bdl
Slaker Grit	Unwashed	5	bdl	440-1490	bdl	bdl	bdl	bdl	bdl-17.2	bdl
	Washed	2	bdl-28.7	930-944	bdl	bdl	bdl	bdl	bdl-5.5	bdl
Green Liquor Dregs		3	bdl	445-1070	bdl	bdl	bdl	bdl	bdl-8.3	bdl
<u>Organic Residues</u>										
Primary Biosolids		5	bdl-7.5	480-1380	bdl	bdl	bdl	bdl	bdl-22.1	bdl
Secondary and Mixed Biosolids	Mixed	7	bdl-3.0	270-2330	bdl	bdl	bdl-95.0	bdl	bdl-9.4	bdl
	Composted	1	bdl	2130	bdl	bdl	bdl	bdl	bdl	bdl
	ASB	1	bdl	1170	bdl	bdl	bdl	bdl	bdl	bdl
Knot Rejects	Washed	1	bdl	170	bdl	bdl	bdl	bdl	bdl	bdl
	Unwashed	1	bdl	734	bdl	bdl	bdl	bdl	bdl	bdl
	Composted	1	bdl	512	14.0	bdl	bdl	bdl	bdl	bdl
Wood Flume Grit		1	bdl	430	bdl	bdl	bdl	bdl	bdl	bdl

[1] bdl = Below detection limits.

Table 4. Toxicity Characteristic Leaching Procedure (TCLP) pesticides, volatiles and semi-volatiles for organic and inorganic pulp mill residues collected during the initial characterization sampling.

Pesticides	
Chlordane	All non-detects
2,4-D	All non-detects
Endrin	All non-detects
Heptachlor & its epoxide	All non-detects
Lindane	All non-detects
Methoxychlor	All non-detect
Toxaphene	All non-detects
2,4,5-TP (Silvex)	All non-detects
Volatiles	
Methyl Ethyl Ketone	All non-detects
Vinyl Chloride	All non-detects
1,1 Dichloroethylene	All non-detects
Chloroform	1.7 ppb in one primary sludge mix with slaker grit, 12.9 ppb in one secondary and mixed sludge
1,2 Dichloroethane	All non-detects
2-Butanone	420 ppb in fresh knot rejects, 20 ppb in one washed slaker grit, 18 ppb in one lime mud 20 ppb in one washed slaker grit
Trichlororethene	All non-detects
Benzene	Substituted benzenes in fresh knot rejects, 20 ppb in one washed slaker grit, 18 ppb in one lime mud
Tetrachloroethylene	All non-detects
Chlorobenzene	All non-detects
Carbon tetrachloride	All non-detects
Semi-volatiles	
b-Cresol	All non-detects
m-Cresol	All non-detects
o-Cresol	9.7 ppb in one primary sludge
p-Cresol	All non-detects
Cresol	All non-detects
1,4-Dinitrochlorobenzene	All non-detects
2,4-Hexachlorobutadiene	All non-detects
Hexachloroethane	All non-detects
Nitrobenzene	All non-detects
Pentachlorophenol	All non-detects
Pyridine	All non-detects
2,4,5-Trichlorophenol	All non-detects
2,4,6-Trichlorophenol	All non-detects

Greenhouse Evaluations of Residue Utilization

Tree Seedling Studies

Characterization data indicated that the majority of mill residues have potential to improve productivity when applied to forest and crop lands in appropriate amounts. As part of residue characterization, two greenhouse studies of tree seedling response to residue addition have been conducted. The first study evaluated 18 residues from the first eight mills sampled during this project and was conducted between July 1995 and February 1996. The second study was installed in June of this year and included 12 residues collected from mills 9-11. It will be completed in January 1997.

Experiment 1

The residues selected for evaluation in this study encompassed the range of plant nutrient availability, physical characteristics, metal and salt concentrations that occurred in residues from mills 1-8. These residues were applied at low and high rates to surface soils from two soil series. Inorganic residues (ash, lime mud, slaker grit and green liquor dregs) were applied on the basis of calcium carbonate equivalence (CCE) at rates designed to raise the soil pH to 6.5 (low rate) or 7.5 (high rate). The high rate was considered to be near the maximum pH for acceptable loblolly pine growth. Organic residues (primary sludge, secondary and activated sludges, mixed sludges, composted sludges, knot rejects, woodyard grit and flume grits) were applied at low and high rates equivalent to 10 and 60 dry tons·ac^{-1} (2.25.10^{3} kg·ha^{-1} and 1.35 10^{3} kg·ha^{-1}). In addition to a non-amended soil control, three commercial soil amendments were incorporated into the greenhouse experiments: commercial lime, peat moss and a cow manure-based organic amendment (Black Kow®).

The two soils selected for use in the greenhouse study were from the Leon (Aeric Alaquod), chosen to represent a typical Lower Coastal Plain wet mineral soil and the Orangeburg (Typic Kandiudult), chosen to represent a well drained Upper Coastal Plain soil. Low and high rates of each residue were applied to pots in factorial combination with soil type and replicated in four family blocks. The four families of loblolly pine, selected and provided by Union Camp Corporation, were: 0235-10-1027, 0225-07-1056, 2035-11-1066, and 0235-11-1057. Two seedlings were planted per pot.

Initial seedling heights and a subsample of diameters were measured at planting. Five seedlings per family were destructively sampled to determine initial biomass and nutrient concentrations. Heights and diameters were measured at least monthly during the course of the study. At the conclusion of the experiment, seedlings were harvested, dried to constant weight at 65 °C and above- and below-ground biomass determined. Dried samples were ground and analyzed for macronutrient and metal concentrations. Soil was collected from each pot and pH and nutrient and metal concentrations determined.

Seedling size, as indexed by height and diameter, and foliar nutrient concentrations at the conclusion of the experiment are presented in tables 5-8. Addition of inorganic residues did not have a significant impact on seedling height or diameter growth at either the high or low levels of addition (Table 5). Seedlings grown in Orangeburg surface soil were larger than seedlings grown in Leon surface soil. Individual residues had slightly different effects on the two soil types but the interaction was not significant ($p=0.09$).

Table 5. Growth response of pine seedlings to addition of inorganic mill residues averaged across target pH rates (*Experiment 1*).

Residue Type	Mill Code	Soil pH[1]	Height	Ground Line Dia.
			--cm--	--mm--
		Leon Series		
Control		6.1	39.7	9.3
Ag. Lime		7.7	40.1	9.7
Ash	M2	8.0	48.1	10.3
	M5	7.9	41.8	10.4
Lime Mud	M4	7.9	42.3	9.3
	M7	7.4	44.9	10.5
Slaker Grit	M1	7.6	47.9	10.0
	M6	7.9	43.0	9.6
Dregs	M1	7.8	44.2	9.6
		Orangeburg Series		
Control		6.1	49.9	11.7
Ag. Lime		7.9	48.6	11.4
Ash	M2	8.1	42.4	10.1
	M5	7.8	53.2	10.1
Lime Mud	M4	7.9	53.4	10.6
	M7	7.7	52.9	11.5
Slaker Grit	M1	7.5	51.4	9.7
	M6[3]	8.0	43.3	10.0
Dregs	M1	7.5	49.7	10.9

Soil nutrient concentrations of greenhouse soils at the completion of the experiment are presented in Table 6. As would be expected, all of the treatments increased Ca concentrations above the control. Potassium concentrations were also increased by the two ash treatments. Magnesium concentrations were increased in most of the treatments; however, differences were only significant for the two ash treatments and for green liquor dregs. Sodium concentrations were not significantly elevated in any of the treatments.

Table 6. Nutrient concentrations of soils amended with inorganic mill residues averaged across target pH rates and soil types (*Experiment 1*).

Residue Type	Mill Code	N	P	K	Ca	Mg	Na
		ppm					
Control		346^{bc}[1]	403^{ab}	125^{c}	328^{e}	205^{d}	341^{abc}
Ag. Lime		402^{abc}	434^{a}	120^{c}	1552^{a}	299^{d}	274^{ab}
Ash	M2	350^{bc}	396^{abc}	198^{b}	1800^{a}	502^{a}	332^{abc}
	M5	415^{a}	443^{a}	384^{a}	1505^{abc}	376^{b}	343^{abc}
Lime Mud	M4	369^{abc}	370^{abc}	147^{c}	1552^{abc}	299^{bcd}	378^{ab}
	M7	322^{c}	313^{c}	122^{c}	1331^{abc}	220^{cd}	306^{abc}
Slaker Grit	M1	325^{c}	367^{abc}	128^{c}	1277^{dc}	269^{cd}	385^{a}
	M6[2]	330^{c}	335^{bc}	129^{c}	1673^{ab}	296^{bcd}	290^{bc}
Dregs	M1	326^{c}	338^{bc}	138^{c}	1056^{d}	318^{bc}	349^{abc}

[1] Treatments with dissimilar superscripts are significantly different (Duncan, a= 0.05)

Results from this greenhouse study do not indicate major beneficial effects of applying inorganic mill residues to seedlings. Since soil analyses show that several of the residues, particularly the ashes, increased soil K and Mg, it is possible that longer-term field trials will demonstrate benefits to forest land application of these materials. Total P contents of soils can be quite high and may not be significantly increased by residue additions even when P availability is increased. Changes in availability of P following ash addition are also being evaluated (see Special Projects).

Organic residue additions had much more impact on growth than inorganic residue additions (Table 7). Significant differences and interactions occurred among soils, residue types and residue rates. Several organic residues improved seedling growth for both soil types. Secondary and mixed biosolids (M7 and M8) were associated with exceptional seedling growth, particularly at the high application rate. Fresh knot rejects (M2 and M4) were exceptions to the generally increased growth associated with organic residue additions. Addition of these residues were associated with poorer growth.

Table 7. Loblolly pine seedling growth response to addition of mill biosolids at low (10 ton ac^{-1}) and high (60 ton ac^{-1}) application rates for two soil types.

Application Rate	Residue Type	Mill Code	Leon		Orangeburg	
			Hgt.	Dia.	Hgt	Dia.
			-cm-	-mm-	-cm-	-mm-
10 ton ac^{-1}	Control		40	9.3	50	11.7
	Peat		41	9.8	54	12.5
	Black Kow®		47	10.5	55	11.5
	Primary Biosolids	M2	41	8.9	46	9.6
		M4	49	10.7	56	9.6
	Secondary and Mixed	M3	49	9.9	48	10.1
	Biosolids	M8	**58**[1]	**14.2**	**62**	**14.9**
		M6	43	9.0	48	10.4
		M7	**52**	**13.8**	**65**	**15.2**
	Composted Sludge	M3	47	10.6	52	10.2
	ASB Sediments	M4	**51**	**11.4**	46	9.9
	Unwashed Knots	M3	**53**	**7.7**	45	9.6
	Washed Knots	M4	36	**7.7**	41	**9.2**
	Composted Knots	M3	40	8.1	50	10.7
60 ton ac^{-1}	Control		40	9.3	50	11.7
	Peat		47	11.2	50	11.3
	Black Kow®		51	11.2	54	11.3
	Primary Biosolids	M2	45	10.5	46	11.5
		M4	58	11.1	47	9.5
	Secondary and Mixed	M3	**65**	**13.6**	48	11.5
	Biosolids	M8	**64**	**14.0**	**83**	**18.6**
		M6	36	8.8	45	9.2
		M7	**64**	**14.4**	**82**	**19.9**
	Composted Sludge	M3	45	9.7	48	10.2
	ASB Sediments	M4	54	10.6	**62**	12.3
	Unwashed Knots	M3	36	10.4	44	9.1
	Washed Knots	M4	45	10.8	36	8.7
	Composted Knots	M3	43	9.3	40	9.6

[1] Values in bold differed significantly from overall average and Control treatment.

Some of the detrimental effect of knot rejects on seedling growth may be due to soil physical properties. Knot rejects are light in weight and the volume necessary to achieve the correct application rate displaced a large portion of the soil in each pot. Water holding characteristics of the resulting soil-residue mix were poor. These materials also have high carbon-to-nitrogen ratios and tend to immobilize N. Seedlings in pots amended with these residues had reduced foliar N concentrations (Table 8).

Table 8. Effects of organic residue additions on foliar nutrient concentrations of greenhouse-grown loblolly pine seedlings averaged for two soil types *(Experiment 1)*

Application Rate	Residue Type	Mill Code	N	P	K	Ca	Mg	Na
			--%--	-------------------		ppm	-------------------	
10 ton ac^{-1}	Control		0.63	1301	5434	3418	1421	1181
	Peat		0.96	1280	5510	3665	1510	1140
	Black Kow®		0.85	1280	6535	3040	1430	1115
	Primary	M2	0.73	978	5340	3553	1500	987
		M4	0.89	1131	5935	3990	1565	1045
	Secondary and	M3	0.84	1112	6300	3910	1505	1075
	Mixed Biosolids	M8	0.92	1280	5500	3260	1290	1000
		M6	0.88	1107	6185	3565	1690	1225
		M7	0.90	1183	5055	4415	1440	1180
	Composted Biosolids	M3	0.81	1145	6305	4060	1470	1005
	ASB Sediments	M4	0.97	1213	5930	4075	1435	1085
	Unwashed Knots	M3	0.63	680	4120	1946	827	2906
	Washed Knots	M4	0.63	704	3580	3005	1230	2095
	Composted Knots	M3	0.82	1002	5415	3105	1335	1100
60 ton ac^{-}1	Control		0.63	1301	5434	3418	1421	1181
	Peat		0.86	1100	5325	3885	1440	1210
	Black Kow®		0.84	1526	8660	3040	1330	1165
	Primary	M2	1.12	1094	7188	5028	2262	1320
		M4	0.83	928	6745	3665	1825	
	Secondary and	M3	0.96	1034	5220	4265	1830	1135
	Mixed Biosolids	M8	1.18	1164	6434	4365	1582	994
		M6	0.81	849	4322	3880	1817	1308
		M7	1.15	1121	4480	4806	1480	1080
	Composted Biosolids	M3	0.79	1020	5880	5280	1715	1075
	ASB Sediments	M4	0.81	1215	6920	4371	1182	1491
	Unwashed Knots	M3	-	-	-	-	-	-
	Washed Knots	M4	0.67	554	2720	2690	960	5260
	Composted Knots	M3	0.82	858	3615	3055	1415	1995

Experiment 2

This experiment evaluated loblolly pine seedling response to application of residues from mills 9-11 not included in Experiment 1. Application rates and procedures were similar to those utilized in Experiment 1 with the exception that one seedling was planted per pot. Treatments and rates used are provided in Table 9. As in Experiment 1, the experiment was replicated in four complete blocks. This study was harvested in January 1997 and results are not yet available.

Table 9. Residues utilized in the second tree seedling study (*Experiment 2*) to be harvested in January 1997.

Residue Type	Mill Code
Inorganic Residues	
Control	
Ag. Lime	
Ash	M9 M10 M11
Organic Residues	
Control	
Black Kow®	
Primary Biosolids	M9 M10 M11
Secondary and Mixed Biosolids	M9 M10-1 M10-2 M11
Woodyard Waste	M9
Woodyard Grit	M11

Crop Studies

Pulp and paper mill residues can be beneficial to agricultural production when applied as liming agents or as soil conditioners and slow-release sources of plant nutrients. Ash, lime mud, grits and dregs have the greatest potential for the former use whereas paper mill biosolids have shown to increase soil organic matter, enhance root development, improve germination rates of crop plants and increase water holding capacity of soils (Muse 1993, and Zibilske 1987). Two greenhouse studies were conducted to evaluate liming value of mill residues as well as crop response to soil conditioning effects of organic mill residues.

Experiment 1- Use of Mill Residues as a Lime Substitute

Approach and Methods: The first greenhouse pot study was initiated during September, 1995 to compare the value of representative inorganic and organic mill residues as lime substitutes under greenhouse conditions. For this study, 3000g of Tifton series surface soil collected from the UGA Agricultural Experiment Station in Tifton , GA (pH 4.6) was dried, sieved and weighed into 3 liter pots. Based on a laboratory incubation, test it was determined that 2.03 grams of $CaCO_3$ (equivalent to 0.5 tons/acre, $1.12 \cdot 10^3$ $kg \cdot ha^{-1}$) was required to raise the pH in the pots to a target pH of 5.6 (appropriate for alfalfa). Twenty-six residues were chosen for comparison (Table 10). These residues included both inorganic residues with lime equivalencies near 100% and organic residues with low value as lime substitutes. Each residue was added to soil at rates which would result in predicted changes in soil pH equivalent to agricultural lime based on the $CaCO_3$ equivalency (CCE) test of Horwitz (1980).

Table 10. Calcium carbonate equivalence (CCE) for mill residues used in greenhouse comparison with agricultural lime (*Experiment 1*).

Residue Type	Mill Code	pH	$CaCO_3$ Equiv.	Mass Added
		-s.u.-	--%--	-g/pot-
Control				
Ag. Lime		6.4	100	2.03
Lime Mud	M1	12.2	99.0	2.05
	M4	11.8	98.8	2.05
	M7	11.8	100.1	2.03
	M7-Mixed	12.3	95.8	2.12
	M10		99.0	2.05
Green Liquor Dregs	M1	13.2	96.4	2.10
	M5	12.7	82.3	2.46
Slaker Grit	M2	13.2	98.5	2.06
	M6	11.6	100.5	2.03
	M8		78.0	3.36
Ash	M1-Mixed	11.5	15.0	15.53
	M2-Bottom	13.4	23.6	8.59
	M2-Fly	12.0	25.2	8.05
	M6-Mixed	10.6	18.9	10.75
	M9-Mixed		60.4	3.36
	M10-Fly		23.1	8.76
Primary Sludge	M2	8.5	28.5	7.11
	M4	9.0	50.2	4.03
	M5	8.0	75.6	2.68
	M6	9.0	20.2	10.04
Secondary and Mixed Sludges	M3-Fresh	8.0	46.7	4.34
	M3-Compost	7.4	41.8	4.86
	M4	7.6	50.5	4.01
	M7	6.5	2.8	72.39
Knot Rejects	M3	8.9	1.0	208.97
	M4	9.2	2.1	95.61
Black Kow			1.0	202.70

Each residue was added to four replicate pots and thoroughly mixed, watered with distilled water and allowed to drain. Alfalfa was planted after pots had drained (about 24 hours). Seeds were planted (approximately 50 seeds/pot) by mixing 0.107 grams of seed with 30 grams of soil and spreading this mixture uniformly across the pot surface. To simulate typical field fertilization regimes, a fertilizer mixture was added to each pot to approximate application rates of 44 lb.ac^{-1} (50 kg·ha^{-1}) P, 178 lb·ac^{-1} (200 kg·ha^{-1}) K, 0.4 lb·ac-1 (0.5 kg·ha^{-1}) Mo and 18 lb·ac^{-1} (20 kg·ha^{1}) B.

Pots were watered with distilled water throughout the course of the 10-week experiment. Several pots were weighed before and after the initial watering to estimate the water holding capacity, and watering was then done by applying 80% of this capacity every other day.

Results: There appears to be some limitation in the ability of $CaCO_3$ equivalency tests (CCE) to predict short-term changes in soil pH following mill residue addition (Fig. 1). Agricultural lime increased soil reaction to pH 5.4, within 0.2 units of the target pH. This is considered acceptable performance for the lime requirement test. Final pH was under-predicted by the CCE test for all the residues we tested. As expected, pH response to inorganic residues was greater than to organic residues and. As a group, lime muds produced results most similar to agricultural lime. Soil pH response to additions of boiler ash was variable. Most ashes increased pH to near 5.0, about half of the pH increase achieved with equivalent amounts of agricultural lime. In contrast, addition of ash from M1 only increased soil pH from 4.6 to 4.7 which may indicate a slower reaction rate. Thus, these results suggest that the lime equivalency test used by many laboratories will tend to overestimate short-term soil pH response to applications of mill residues.

Although the purpose of this study was to compare pH response of mill residues with agricultural lime it should be noted that the alfalfa plants grew very slowly and exhibited marked chlorosis in several of the organic residues. As was indicated in our characterization analyses, primary sludges and knot rejects are low in N. The observed chlorosis was likely due to N deficiency.

Experiment 2- Wheat Response to Organic Mill Residues

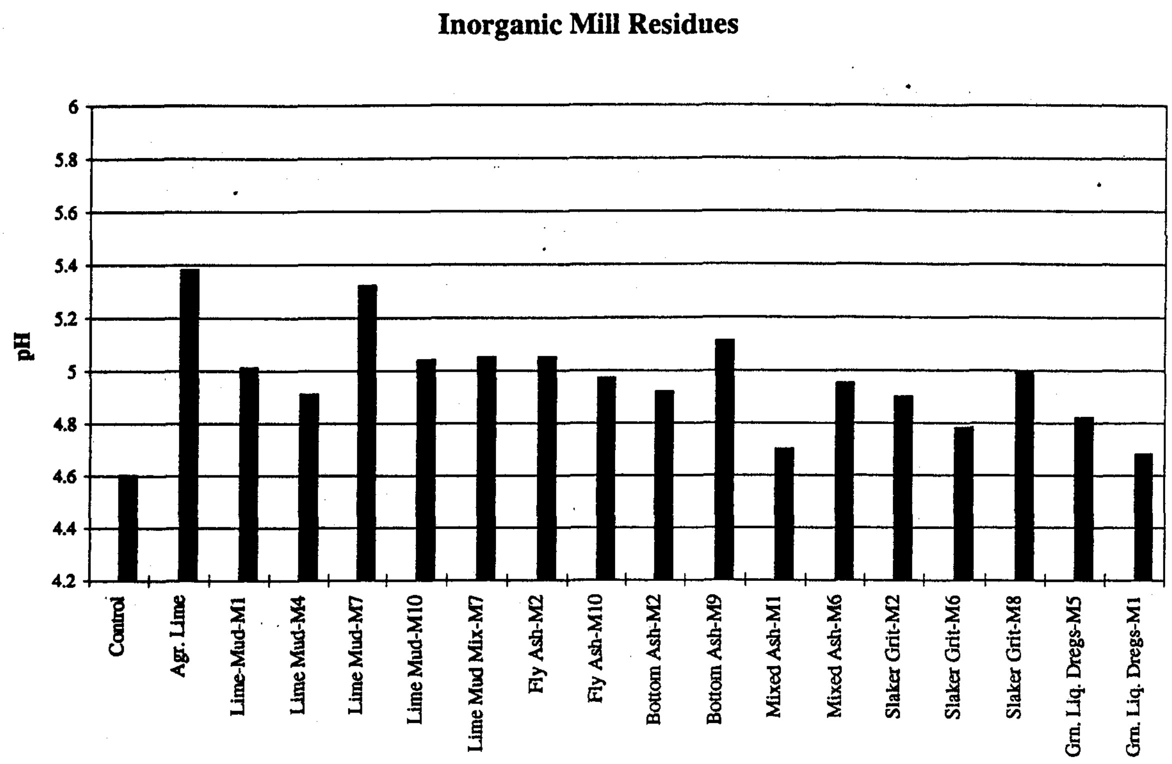
Approach and Methods: A second greenhouse study was initiated in January 1996 to evaluate wheat growth response to organic mill residue addition. Results from Experiment 1 indicated that nitrogen deficiency would be expected following application of either primary biosolids or knot rejects. To evaluate the impact on N availability, residues were added with and without a supplemental N source.

Treatments consisted of a factorial combination of residue type, application rate and supplemental N. Three residue types were evaluated: primary biosolids from M6; secondary and mixed biosolids from M3, M8 and M10; and ASB sediments from M4. Three-liter pots were filled with dried and sieved surface soil from an Appling series soil (pH=6.3; double-acid extractable P, K, CA and Mg equaled 37, 124, 441 and 53 ppm, respectively) collected from the University of Georgia Plant Sciences farm in Athens. Six organic residues were added to these pots at rates equivalent to field application rates of 30 dry tons·ac^{-1} ($67 \cdot 10^3$ kg·ha^{-1}, 90 g·pot^{-1}) or 60 dry tons·ac^{-1} ($135 \cdot 10^3$ kg·ha^{-1}, 180 g·pot^{-1}). A seventh, no residue addition treatment, was included as a control. Nitrogen addition consisted of no added nitrogen (residue only addition) or addition of the equivalent of 50 lb·ac^{-1} available N (2.95 g·pot^{-1}) as poultry litter. These 28 treatments (7 residues x 2 rates x 2 N additions) were replicated as 4 complete blocks physically separated in the greenhouse.

Residues and poultry litter were mixed with soil in the pots, thoroughly wetted and allowed to drain overnight. The following day, 10 wheat seeds were planted in each pot, covered with a thin layer of soil and watered immediately to 80% of soil water holding capacity. Pots were observed daily and watered as needed until they were harvested during March, 1996.

Results: In the absence of supplemental N, wheat growth was generally increased by addition of ASB sediments (M4) or secondary and mixed biosolids (M7, M10) at the lower application rate (Fig. 2). As expected, wheat growth was reduced by addition of fresh primary sludge. Addition of N as poultry litter increased growth in almost all residue treatments. With the exception of plots amended with primary sludge, wheat growth was greater in all pots amended with mill biosolids and poultry litter than in the no treatment control.

Two residues, ASB sediments (M4) and secondary and mixed biosolids (M10), improved wheat growth at higher application rates (60 tons$\cdot$ac^{-1}) in the absence of supplemental N. As at the lower rate, addition of poultry litter generally increased wheat growth. In most cases, growth was greater in pots receiving 60 tons$\cdot$ac^{-1} sludge and poultry litter than in the unamended control.



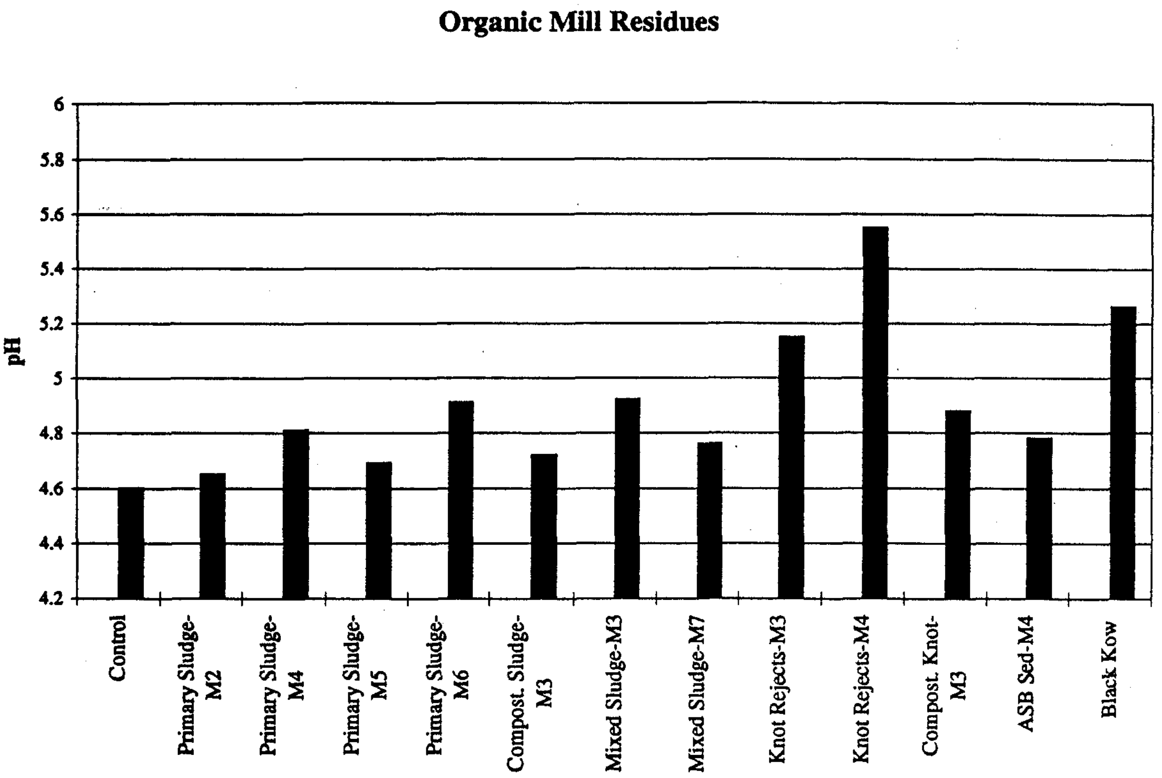
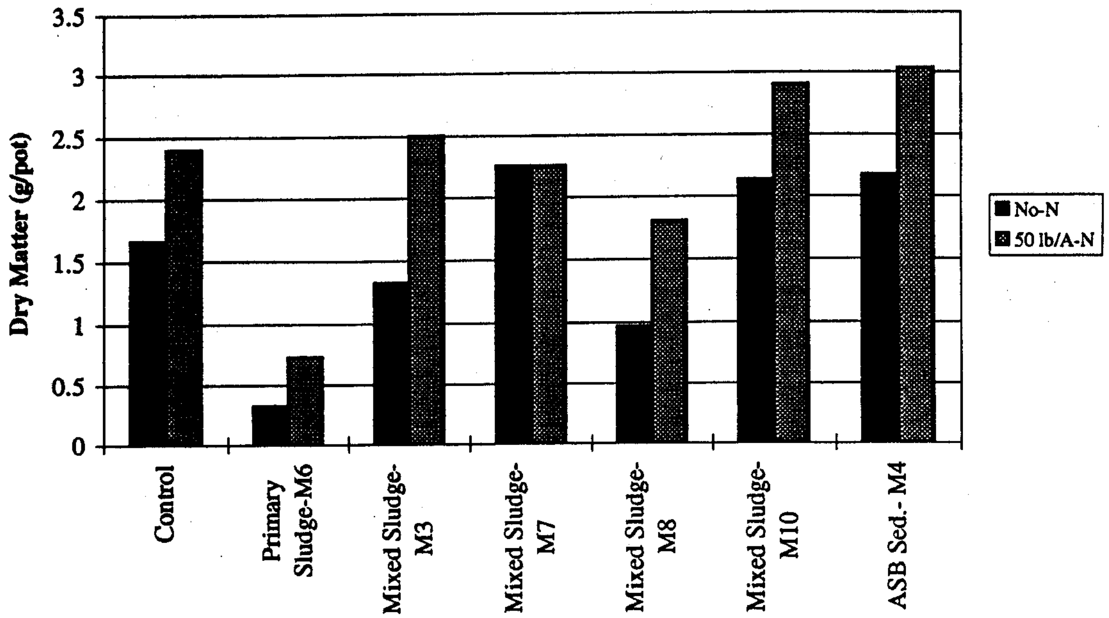


Fig. 1. Soil pH 10 weeks following addition of inorganic and organic mill residues to a Tifton soil at rates equivalent to 0.5 tons·ac^{-1}.

Wheat Yield 30 T/A Biosolids



Wheat Yield 60 T/A Biosolids

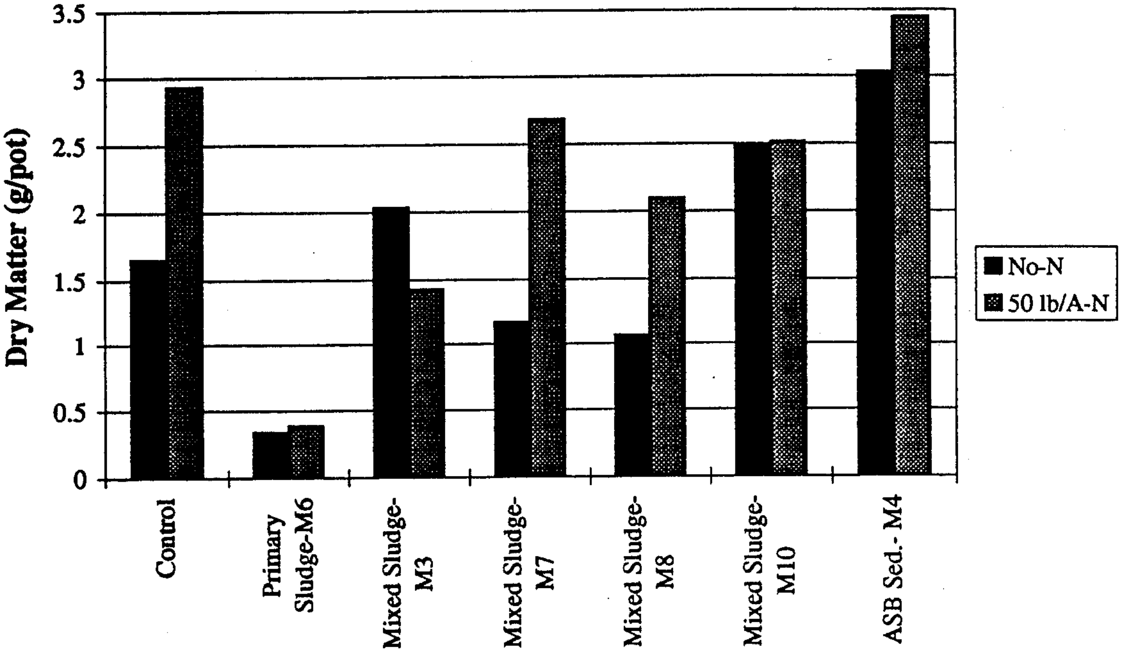


Fig. 2. Yield of wheat grown in Appling surface soil amended with pulp mill biosolids at two application rates (30 and 60 ton ac^{-1}) with and without N applied in poultry litter.

Field Experiments

Forest Land Application

In the spring of 1995, four experimental sites were chosen to study effects of paper mill residues applied to forest lands. Each site was divided into a regeneration area, where residues are applied and incorporated into the soil prior to planting, and an established stand where residues are broadcast on the soil surface in an intermediate-aged forest plantation (13 to 17 yrs.). These four sites bracket the range of soil textures and growing conditions commonly found in the Upper and Lower Coastal Plain regions of the Southeast. These sites are:

Site 1 Upper Coastal Plain -Weyerhaeuser Corp., Oglethorpe, GA

Site 2 Lower Coastal Plain -Union Camp Corp, Ogeechee Experimental Forest, Rincon, GA

Site 3 Sandhills- Savannah River Site, New Ellenton, SC.

Site 4 Lower Coastal Plain-Rayonier Corp., Satilla Forest, Brantley Cty., GA

Site 1 Upper Coastal Plain, Oglethorpe GA

Site Establishment: This site is located 3 miles west of Oglethorpe, GA in a heavily agricultural region of the Upper Coastal Plain in the west central part of the state. Topography is nearly level and soils are of the Orangeburg Series, which are deep sandy loams, commonly planted to cotton or peanuts in this region. The land is owned by Weyerhaeuser Corp. and is about 5 miles from Weyerhaeuser's Flint River Mill.

The Oglethorpe site was the first forestry site to be installed, and was ready for application of mill residues in January, 1996. It consists of two contiguous tracts; a regeneration area harvested in 1995, and an established stand in a 1981 loblolly pine plantation. One-fifth acre (0.08 ha) experimental treatment plots containing one-tenth acre (0.04 ha) measurement plots were established in both areas during the summer of 1995. The established stand was thinned from an average basal area of 115 $ft^2 \cdot ac^{-1}$ (8.1 $m^2 \cdot ha^{-1}$) to a residual basal area of 80 $ft^2 \cdot ac^{-1}$ (5.6 $m^2 \cdot ha^{-1}$). Both areas were spread with mill residues in late January, 1996. Each residue was applied to treatment plots at two levels. For residues used as liming agents, the low level corresponded to an amount needed to raise soil pH to 6.5 and a high rate was the amount required to raise pH to 7.5, based on the lime equivalence of the material. Biosolids were assigned a low and high level which bracketed the range commonly employed in operational mill sludge application. All residues were applied on a volumetric basis using a wet weight/dry weight to density conversion. The nominal rates for each residue spread at the Oglethorpe site are provided in Table 11. Immediately after the application of residues, the entire regeneration site was disked to incorporate the material into the upper soil surface. Each residue treatment and rate combination was applied to three replicate plots in a randomized complete-block design.

Within each interior measurement plot, a 2m x 2m subplot was covered with a plastic sheet prior to application of residues. When the spreading operation was finished, the plastic was removed and the subplot received the exact amount of residue assigned to the plot on a unit area basis. These subplots then received a "tag" dose of potassium bromide (KBr) solution to provide a check for predicted movement of dissolved ions in the soil solution. Ceramic cup soil lysimeters were installed at 1 meter depth adjacent to these tagged subplots.

The regeneration area was planted with loblolly pine seedlings in February 1996. To control vegetation re-growth, the regeneration area was treated with 2.5 oz·ac^{-1} of Oust® in late May 1996.

Table 11. Residues Applied at the Oglethorpe Forest Land Application Site, January, 1996.

Residue	Low Rate	High Rate
	--dry tons·ac^{-1}--	--dry tons·ac^{-1}--
Primary Biosolids	10.0	60.0
Secondary Biosolids	10.0	60.0
Ash	7.9	12.4
Green Liquor Dregs	1.6	2.4
Lime Mud	1.4	2.1

Monitoring: Post-application site monitoring consists of assessments of seedling and established stand growth and nutrition as well as soil and soil solution chemistry. Seedling and mature tree height and diameter are being measured annually during the dormant season. Foliage samples are being collected, digested and analyzed for macronutrient and selected metal concentrations. To assess soil change and provide data for future modeling efforts, 1m deep, intact soil core samples (5 cm dia.) are being periodically collected from the potassium bromide-tagged subplots. Soil solution samples are being collected quarterly.

Results: In the established stand area, the vigor and general appearance of the stand is very good. None of the trees in the treatment or control plots show chlorosis, short foliage or any other gross signs of physiological distress

Initial seedling survival in the regeneration stand, measured in April, was excellent, and exceeded 90%. From mid-April through the end of May, the Oglethorpe site received very little rain; however, weed re-growth was heavy. By late May, the regeneration site had a continuous cover of ragweed (*Ambrosia* spp. L.) and pigweed (*Chenopodium* spp. L.) approximately 1.5 m tall. The Oust application effected release of seedlings for about six weeks but by mid-July ragweed had closed over the seedlings and mortality began to increase. After a check of the regeneration site in October, it was clear that mortality of loblolly pine seedlings had reached a level where it would be difficult to distinguish treatment effects (residue type and level) from inter-species competitive effects from excessive weed growth. Thus, in December 1996, surviving pine seedlings were removed and the site was replanted in January, 1997.

Site 2 - Lower Coastal Plain, Rincon, GA

Site Establishment: This research site is located on Union Camp Corporation's Ogeechee Experimental Forest near Rincon, GA. Two adjacent tracts were selected for the regeneration and established stand study areas. The established stand is a 19-year-old loblolly pine plantation and the regeneration site is a recently harvested and site-prepared area. Topography is flat, with sandy soils and very poor drainage. Both the regeneration and established stand study areas have Pelham Series soils (Arenic Paleaquult), with the exception of a small inclusion of Mascotte Series (Ultic Haplaquod) in the northwest corner of the established stand.

To prepare the established stand for residue application, the stand was marked and thinned in the summer of 1996. All trees were removed from every fifth row; trees were selectively removed from intervening rows. The basal area of the stand was reduced from an average of 140 $ft^2 \cdot ac^{-1}$ to 100 $ft^2 \cdot ac^{-1}$. In early October the regeneration area was sprayed with a mix of Accord®, Arsenal® and Garlon®.

Plots were established in both regeneration and established stand areas in a configuration similar to the Oglethorpe site. Treatment plots were approximately one-fifth acre and inner measurement plots were one-tenth acre. Each measurement plot contained a small (6 x 9 ft., 2 x3 m) subplot which was covered with plastic for later tagging with a potassium bromide solution.

The entire site, established stand and regeneration area, was spread with mill residues in late October 1996. Four mill residues were used, and each residue was applied at a low and high level (Table 12). As on the Oglethorpe site, when the spreading was complete, plastic covers were removed from the subplots, and each subplot was hand spread with the target amount of the residue assigned to the plot on a unit area basis.

Table 12. Residues Applied at the Rincon Forest Land Application Site, October 1996.

Residue	Low Rate	High Rate
	--dry tons $\cdot ac^{-1}$--	--dry tons $\cdot ac^{-1}$--
Primary Biosolids	10.0	60.0
Ash	8.9	13.4
Dregs-Grit Mix[1]	1.2	1.8
Lime Mud	1.2	1.7

[1] This material was a 50-50 mix by volume of green liquor dregs and washed slaker grit.

Following spreading, the regeneration area was double-bedded to incorporate residues and provide raised planting rows for the seedlings. No bedding or incorporation of residues was done in the established stand. The regeneration area was machine planted with loblolly pine seedlings at a 12 x 6 ft. (4 x 2 m) spacing in late-November.

Monitoring: As on the Oglethorpe site, post-application site monitoring of this site will consist of assessment of seedling and established stand growth and nutrition as well as soil and soil solution chemistry.

Site 3 - Sandhills, Savannah River Site New Ellenton, SC

This land application site is located within the Savannah River Site which is jointly administered by the US Department of Energy and the US Forest Service. The tract chosen for land application of residues is typical of the Sandhills region, with rolling hills and deep, excessively drained coarse-textured soils in the Troup (Arenic Kandiudult) and Lucy Series (Grossarenic Kandiudult). Development of this site was completed in late 1996 and the area is now ready for application of residues.

In the established stand area, a 1981 loblolly pine plantation, research plots were installed and monumented. The stand was marked for a complete removal of every fifth row of trees with selective thinning in the intervening four rows. Selective thinning was done as a crop tree release, with retention of the largest and best formed trees. The stand was thinned in the summer of 1996 with minimal damage to the residual stand. Basal area was reduced from an average of 115 $ft^2 \cdot ac^{-1}$ (8.1 $m^2 \cdot ha^{-1}$) to 80 $ft^2 \cdot ac^{-1}$ (5.6 $m^2 \cdot ha^{-1}$).

The regeneration area was located in a harvested oak-pine stand which had only minimal site preparation prior to selection for the study. In October, the area was sprayed with an Accord®-Arsenal® mix to control hardwood sprouts, and in December it was burned for slash reduction.

Land application of residues on the established stand and regeneration areas was scheduled for early February, 1997. Establishment of this site required an experimental use permit from the State of South Carolina, Department of Health and Environmental Control (DHEC). The appropriate application was submitted in June; however, as of this date, we have not received this permit. It appears likely that we will need to reschedule residue application for later in 1997.

Site 4 Slash Pine Flatwoods, Brantley Co. GA

This site is located on Satilla Forest, a tract owned by Rayonier Corp. in the northern portion of Brantley County. In 1995 an established stand study area was selected in a 1979 slash pine plantation. Brantley County, in southeast Georgia, is the only site of the four developed to date where slash pine is the tree species. The soil at this site is a Leon Series (Aeric Alaquod), which is coarse-textured and classed as somewhat poorly drained. This soil has a spodic (leached, siliceous) horizon, which, along with better drainage, distinguishes it from the study site at Rincon, GA.

Stand density in this plantation averages approximately 100 $ft^2 \cdot ac^{-1}$. Light penetrates through the canopy resulting in a considerable amount of underbrush.

In June and July of 1996, research plots were located in the established stand. A regeneration area will be selected this spring. It will not be possible to locate the regeneration area contiguous to the established stand on the Brantley County site. However, the regeneration area will be established on a Leon Series soil as close as possible to the established stand area.

Crop Land Application

Three sites, which represent soil conditions common to agricultural areas in Georgia, were selected for crop field studies: a well-drained Upper Coastal Plain site in Tifton, GA on Tifton series soil (Typic Kandiudult); a well-drained Piedmont site near Athens on an Appling soil (Typic Kanhapludult); and a poorly drained Middle Coastal Plain site in Midville, GA on a Grady (Paleaquult) and Rains (Typic Paleaquult) soil complex. Two field experiments, which paralleled crop greenhouse studies, were conducted during 1996 on each site: an experiment to study crop response to mill residues as liming materials, and an experiment to evaluate crop response to application of organic mill residues.

Experiment 1- Use of Residues as a lime substitute

Approach - Inorganic residues from selected mills were applied to small plots on each site during February 1996. Plot sizes were approximately 18 x 25 feet (5.5 x 7.6 m), but varied slightly from site to site depending on the planting equipment available. Application rates of each material were determined based on standard assessments of lime requirement for the crop and previously determined CCE of residue materials (Table 13). At each site, ash treatments consisted of one low CCE ash (CCE <15%) and one high equivalence ash (CCE >15%). Fertilizer P and K were applied on each plot during the first part of March of 1996.

Table 13. Residue treatments, site conditions and baseline fertilization rates used in crop field studies of mill residue applied as an agricultural lime substitute.

Location	Initial Soil pH	Baseline Fertilization Rate N	P	K	Residue Type	Mill Code	$CaCO_3$ Equiv.	Application Rate (Dry)	
	-s.u.-	-----$lbs \cdot ac^{-1}$----					-- % --	$-lb \cdot ac^{-1}-$	$lb \cdot plot^{-1}$
Athens	4.7	200	66	108	Control	-	0	0	0
					Ag. Lime	-	100.0	1800	18.6
					Ash	M9	60.4	3000	30.9
					Ash	M1	13.1	13846	143.0
					Slaker Grit	M1	98.8	1822	18.8
					Lime Mud	M1	99.0	1818	18.7
Tifton	5.0	200	40	75	Control	-	0	0	0
					Ag. Lime	-	100	1500	15.5
					Ash	M10	23.1	6522	67.4
					Ash	M1	13.1	11539	119.2
					Slaker Grit	M7	99.9	1502	15.5
					Lime Mud	M7	100	1500	15.5
Midville	4.9	200	66	75	Control	-	0	0	0
					Ag. Lime	-	100	1300	13.4
					Ash	M1	13.1	10000	103.3
					Ash	M9	60.4	2167	22.4
					Slaker Grit	M7	99.9	1301	13.4
					Lime Mud	M7	100	1300	13.4

Pioneer 3245 hybrid corn seed was planted at a spacing of 30,000 plants ac^{-1} in March, 1996 as the primary crop for the first year of this two-year experiment. Wheat is being rotated with corn during second year of the study (1997). Both liming plots in Athens and Tifton were irrigated when needed. Plots were not irrigated in Midville.

Monitoring: Corn was harvested at maturity, dry weight determined, and a subsample of dried sample was ground and analyzed for nutrient concentrations. Following harvest, surface soil samples were collected from each plot using an Oakfield tube, composited and analyzed for pH, P, K, Ca and Mg. To assess potential metal movement, undisturbed soil cores will be collected using a Giddings core sampler during the winter of 1997. To provide additional chemical monitoring of the soil, a series of lysimeters were installed in plots both in Athens and Tifton during the month of August 1996 (after the corn plots were established). Lysimeters were installed at two depths. For shallow monitoring of the plow layer, lysimeters were installed 12 inches below the soil surface. For deeper monitoring (to include the B horizon in the monitoring process), lysimeters were installed 24 inches below the surface.

***Results*:** Soil samples from the liming plots were collected during September 1996, soon after the crop was harvested (Table 14). Soil pH was significantly greater than the untreated control in all residue treatments. Generally, mill residues performed as well as agricultural lime. Soil pH did not differ among residue and agricultural lime treatments and was near the target pH. Plots receiving low CCE ash from M1 were the exception. Ash with low CCE increased the pH significantly more than either agricultural lime or the other residues. This suggests that the laboratory test underestimated the liming effect of this material and, therefore, the rate of application calculated to produce a target pH was higher than necessary.

Table 14. Soil pH measured at the end of one growing season and corn yields from field plots amended with agricultural lime and selected mill residues.

	Athens		Tifton		Midville	
Treatment	pH	Yield	pH	Yield	pH	Yield
	--s.u.--	---lb ac^{-1}--	--s.u.--	---lb ac^{-1}---	--s.u.--	---lb ac^{-1}---
Untreated Control	4.8	6766^{a}[1]	5.7	6567^{a}	5.4	1079^{a}
Agricultural Lime	5.5	12686^{b}	6.1	7363^{b}	5.7	1059^{a}
Low Equiv. Ash	6.8	15731^{c}	7.4	6965^{b}	6.5	1861^{b}
High Equiv. Ash	5.4	11094^{b}	6.3	8259^{b}	5.6	972^{a}
Slaker Grit	5.7	11035^{b}	6.1	7960^{b}	5.7	972^{a}
Lime Mud	5.6	10647^{b}	6.3	9851^{b}	5.7	1009^{a}

[1] Treatments with dissimilar superscripts are significantly different (Duncan, a= 0.05)

Corn yields were significantly improved by addition of either agricultural lime or mill residues at both the Athens and Tifton sites (Table 14). Yields were extremely low at Midville due to severe drought and lack of irrigation and are unreliable as indicators of treatment impacts. As suggested by soil test results, mill residues did provide a suitable lime substitute. In no case was corn yield lower in plots treated with mill residue than in plots treated with agricultural lime. Significantly higher yields occurred on plots receiving low CCE ash from M1. A general relationship existed between corn yield and soil pH on these experimental plots (Fig. 3). Small differences in yield that occurred among treatments, and

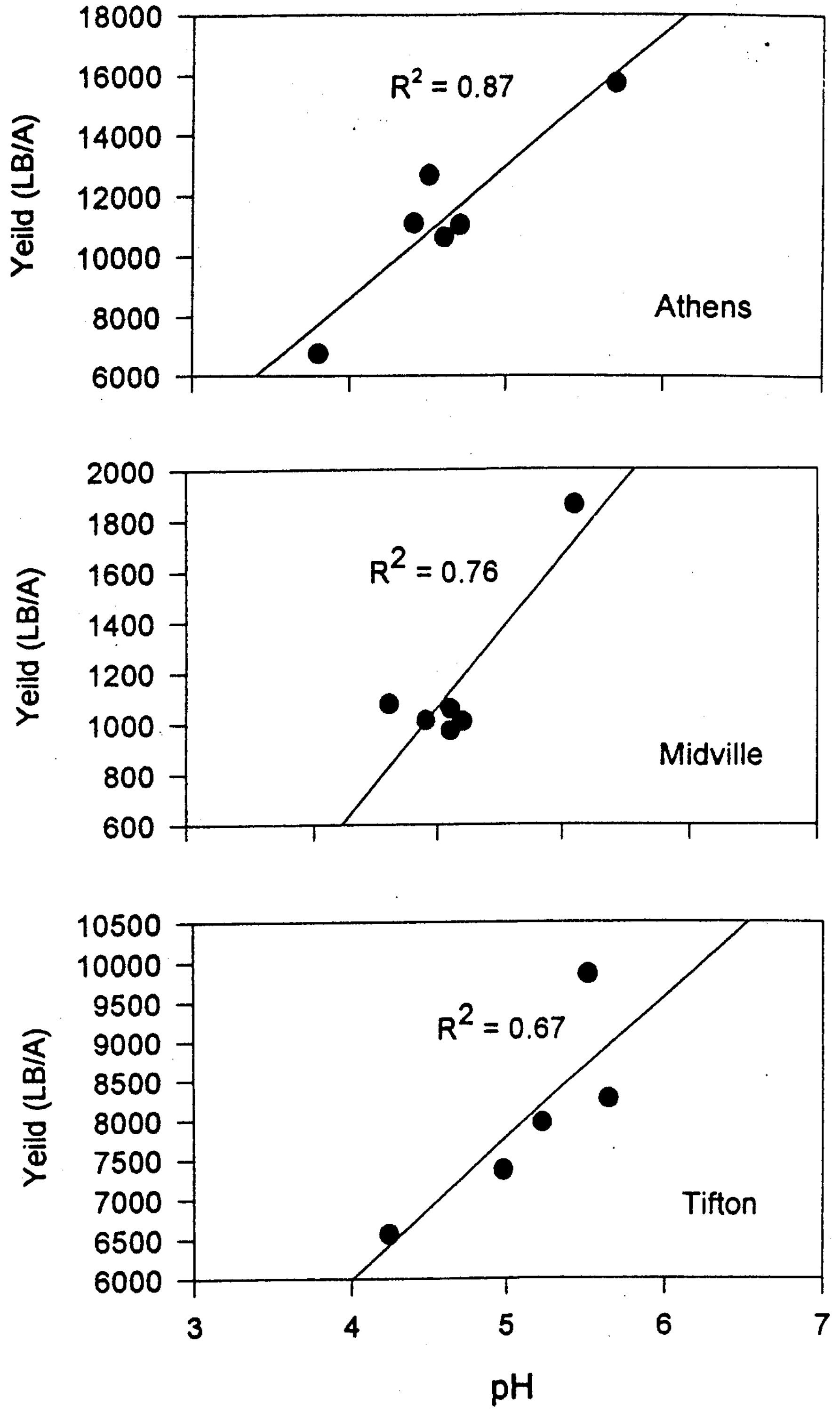


Fig. 3. Relationship between corn yield and soil pH (measured at the end of the growing season) at three field sites.

increased yields for the M1 ash, were likely due to differences in soil pH rather than due to contribution from other factors.

Results from this field experiment contrast with those of earlier greenhouse experiments, where mill residues did not increase pH as much as agricultural lime. In this experiment, mill residues were as effective in raising soil pH as agricultural lime. Moreover, with the exception of one low CCE ash from M1, lime equivalence tests adequately expressed the liming value of these materials when evaluated after a full growing season.

Experiment 2-Organic Residues as a Crop Soil Amendment

Approach: The value of organic mill residues as soil amendments was evaluated in field studies at the Athens and Tifton sites. At each site, representative organic residues were applied to small experimental plots (approximately 18 x 25 feet) at three rates: 0 (control), 30, and 60 tons·ac^{-1}. Approximately 50% of the total sludge application was applied to plot surfaces in April 1996, and the materials were disk-harrowed into the surface. The remaining 50% of the material was applied to the plot surface without incorporation. As most primary and some secondary biosolids are low in available N, a N supplement in the form of poultry litter was added to plots receiving these sludges. The amount of poultry litter applied was equivalent to 100 lb·ac^{-1} N. Pioneer 3243 hybrid corn seeds were planted at both sites in early May. These plots were irrigated as needed in a manner representative of typical irrigation practices in the area. The Tifton site was irrigated five times with supplemental watering equivalent to 3.8 inches of rainfall. At the Athens site, limed plots were watered six times (supplemental water totaled 4.5 inches) and biosolid-amended plots were watered 14 times (supplemental water totaled 10.5 inches). Weed growth was controlled through application of Roundup® (2 lb·ac^{-1} active ingredient) and Atrazine® (2 lb·ac^{-1} active ingredient) at both sites.

Results: First year corn yields for the two study sites are presented in Table 15. Excellent yields were achieved on the Athens site. Although yields were generally higher in residue-amended plots, these increases were not statistically significant. Plots treated with the highest rate of primary biosolid had significantly lower yields than other plots because of increased feeding on newly germinated plants by birds.

Corn yields were generally poor at the Tifton site, presumably because of reduced germination and insufficient irrigation. Somewhat surprisingly, plots treated with primary sludge had greater yields than any other treatment or the control. We attribute these improved yields to the mulching effect of unincorporated primary sludge. This suggests a potential agronomic use for these materials which will be investigated in future research.

Table 15. Yield of irrigated corn grown on soils amended with organic paper mill residues and poultry manure as a source of supplemental nitrogen.

Residue	Rate	Corn Yield Athens	Corn Yield Tifton
	tons·ac^{-1}	lbs·ac^{-1}	lbs·ac^{-1}
Control		7351^{b1}	941^{a}
Primary Biosolids	30	8619^{b}	2768^{b}
	60	6009^{a}	4157^{c}
Secondary Biosolids	30	8700^{b}	359^{a}
	60	8545^{b}	407^{a}
Composted Sludge	30	8244^{b}	1180^{a}
	60	7608^{b}	1477^{a}

[1] Treatments with dissimilar superscripts are significantly different (Duncan, a= 0.05

Special Projects

Phosphorus Availability in Pulp and Paper Mill Ash

Catherine Merz

Boiler ash may provide a practical means of recycling Ca, K, P and other nutrients removed from sites during harvesting of crops or forests. Phosphorus availability in ash is of particular interest, as over 160,000 acres of forest land in the southeastern US are fertilized with phosphorus each year. Many of these forest lands are in close proximity to mills that could supply ash as a substitute for commercial P fertilizer. In turn, the mills would benefit from reduced landfill costs. Two questions are often raised regarding the use of ash as a P source: (1) do standard fertilizer tests of available P adequately express P availability in ash?, and (2) is soil availability of P reduced at the elevated pH expected to occur following addition of ash as a P source?

Several experiments are being conducted to address these questions. To address the first question, ash samples from all participating mills were tested for available P using the standard acid-oxalate (AOAC) fertilizer test method (Kane 1995), and results were compared with a resin extraction procedure (Schumann 1996). Resin extraction procedures are gaining favor among agronomists because resins function in a similar manner to charged soil colloids, require little soil disturbance, and produce a better correlation to plant uptake than other methods of extraction. Results of our comparisons between AOAC and resin-extraction procedures are presented in Fig 4. The correlation between the two procedures is excellent (r^2=0.98) indicating that standard AOAC fertilizer tests should give a reliable index of P availability in mill ash.

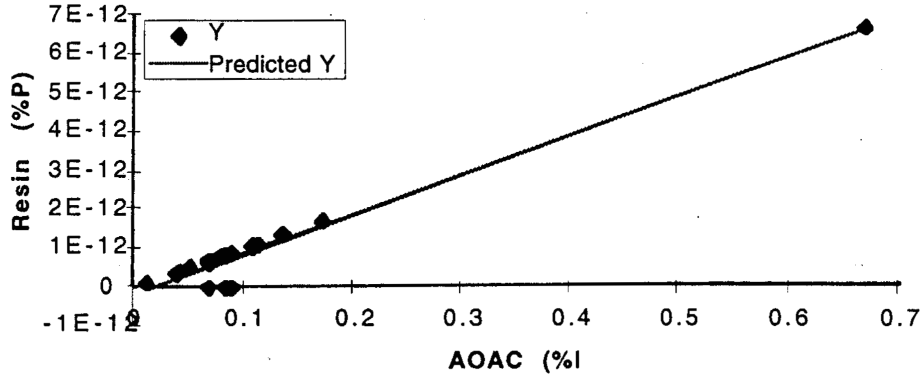


Fig. 4. Correlation between resin-extractable P and acid-oxalate fertilizer tests for available P in 13 ash samples from 11 participating mills.

To further evaluate the value of ash as a P source, an incubation study is being conducted using a P-deficient Lower Coastal Plain soil (Leaf series, Typic Albaquult). In this study, available soil P is being determined at one month intervals following addition of three ash sources and compared with available soil P in soils amended with conventional triple super phosphate fertilizer (TSP) and with a non fertilized control. All treatments (except the control) were amended with AOAC

P equivalent to 40 lb.ac^{-1}. Soil pH was adjusted by addition of acid or base to produce a range of final soil pH for each amendment type. Samples were incubated near field capacity and room temperature, and P extracted by double acid at monthly intervals.

Double acid extractable soil P concentrations have been determined through the fourth month of incubation and these preliminary results are presented in Table 16. The mean double-acid extractable P concentrations of all three ash-amended soils are significantly higher than extractable P concentrations of both the TSP-amended soil and the untreated control (TSP and the control are also significantly different). There is no clear trend in available P over time for any P source. Although some differences in extractable P concentrations exist among levels of soil pH, these differences are not consistent.

Table 16. Double-acid extractable P concentrations in unamended soil and soil amended and incubated for four months with ash from pulp mill boilers.

Treatment	Target pH	Actual pH	Double Acid Extractable P Months Since Start of Incubation 1	2	3	4
			--------------------ppm	------------------		
Ash 1[1]	4.0	4.2	15.0	1.8	11.0	12.7
Ash 1	5.0	5.3	15.3	0.5	9.3	13.8
Ash 1	6.0	6.4	16.5	12.6	6.3	10.3
Ash 1	7.0	7.1	15.2	0	6.0	9.0
Ash 1	8.0	7.2	13.2	13.9	6.0	9.1
	Average		15.0	3.4	7.7	11.0
Ash 2	4.0	4.4	16.7	2.7	10.8	16.3
Ash 2	5.0	5.3	18.9	3.0	13.5	17.4
Ash 2	6.0	5.7	29.1	4.7	10.7	13.6
Ash 2	7.0	6.5	39.0	1.5	11.2	13.7
Ash 2	8.0	7.2	15.7	3.9	10.0	12.3
	Average		23.9	3.2	11.2	14.7
Ash 3	4.0	4.4	24.8	3.0	12.5	8.0
Ash 3	5.0	5.1	26.0	3.0	10.7	14.1
Ash 3	6.0	6.2	11.7	6.4	10.9	14.4
Ash 3	7.0	6.8	17.9	3.5	8.3	14.5
Ash 3	8.0	7.0	23.2	3.4	8.1	10.7
	Average		20.9	3.8	10.1	12.3
TSP	4.0	4.0	14.6	4.4	4.8	5.3
TSP	5.0	4.3	4.8	2.7	4.1	6.9
TSP	6.0	5.0	5.4	2.3	4.1	9.6
TSP	7.0	5.3	5.0	3.1	3.6	10.7
TSP	8.0	5.6	2.3	2.4	-1.0	6.1
	Average		6.4	3.0	3.1	7.7
Control	4.0	4.1	0	-0.3	-0.5	0.5
Control	5.0	4.4	0.1	-0.8	-0.7	0.4
Control	6.0	4.8	-0.1	-0.9	-0.3	0.4
Control	7.0	5.4	0.3	2.5	3.7	0.4
Control	8.0	5.7	0.3	1.2	-0.4	3.5
	Average		0.1	0.3	0.4	1.0

[1] AOAC P were: Ash 1 =0.068%, Ash 2=0.082% and Ash 3=0.014%.

These preliminary results indicate that boiler ash can be applied as a P source in young pine plantations and that standard tests of available P may be adequate for determining ash application rates. Elevated soil pH does not appear to limit use of ash as a P source. Greenhouse and field tests are currently being conducted in an effort to verify these preliminary conclusions.

Impact of Pulp and Paper Mill Residues on Forest Soils: Fate and Mobility of Selected Residue Constituents

Herwig Goldemund

Due to the difficulty and expense of completing field studies of each material which is a candidate for land application, regulatory agencies rely on computer simulations to develop policy and to evaluate the potential impacts of residue application. As part of our efforts to extend conclusions from our four forestry field sites, we are evaluating LEACHM (Leaching Estimation and Chemistry Model) as a tool for predicting the fate and mobility of potential contaminants from pulp and paper mill residues. LEACHM (Hutson and Wagenet 1992) was developed at Cornell University and is a well established model of water and solute movement. It is a finite difference model that numerically solves Richards' equation for water flow and the convection-dispersion equation (CDE) for solute movement. A summary of the input data required for LEACHM is presented in Table 17.

Table 17. Data input requirements for modeling element and contaminant movement following residue application to forest land.

Soil Physical Properties	Crop and Weather Data	Chemical Properties
Particle size distribution	Relative root distribution	Initial soil profile concentration
Soil temperature (optional)	Wilting point	Solubility
Initial water content	Min. root water potential	Vapor density
Particle density	Crop cover fraction	Adsorption isotherms
Bulk density	Precipitation	Molecular diffusion coefficient
Surface flux density	Potential evapotranspiration	Possible transformation and degradation rate constants
AEV/BCAM (fitting parameters)		
Saturated hydraulic conductivity		
Dispersivity		

Undisturbed soil cores were collected from control and treated plots at the Oglethorpe site to determine saturated hydraulic conductivities and bulk densities, and for developing moisture release curves necessary for calculating unsaturated hydraulic conductivity. Parameters are fitted using a mathematical software packet (Mathcad Plus® 6.0). Small weather stations have been set up at the Oglethorpe and Rincon sites to collect rainfall, temperature and wind data necessary to calculate evapotranspiration. To provide an assessment of our modeling efforts, measured movement of a bromide tracer applied to small (2m x 2m) plots will be compared with model predictions. If we can successfully describe bromide movement, model results will also be compared to distribution of metals measured in intact core samples. In addition to these field comparisons, more intensive research on the effects of residue addition on contaminant transport under controlled laboratory settings is being planned.

Effects of Pulp and Paper Mill Biosolids on Physical Properties of Agricultural Soils

Rachel Cochran

Research related to the effects of pulp mill biosolids on soil physical properties was initiated in November 1996. The goals of the research are to determine decomposition rates of organic sludges (primary and secondary) and to evaluate changes in soil physical properties resulting from land application of these materials. Previous studies have generally concluded that additions of pulp mill biosolids can have beneficial effects on soil properties including decreased bulk density, increased water-holding capacity, and improved structure and stability (Macyk 1996, Zhang et al. 1993). Soil structure and water-holding capacity are important attributes for crop growth. Increased soil stability is associated with improved water infiltration and reduced runoff from crop and forest land.

Decomposition of primary and secondary biosolids will be studied under controlled laboratory conditions. Decomposition rates and changes in soil physical properties (bulk density, water-holding capacity, hydraulic conductivity and aggregate stability) resulting from biosolid amendments will be assessed and compared with results from field studies. A preliminary incubation study using primary sludge to establish methodologies and measurement frequencies to be used for a future larger-scale study was completed in December 1996. In this study, a sandy loam soil (Appling series, surface soil) was amended with primary sludge from Mill 6 at rates of 67 tons$\cdot$ha^{-1} and 135 tons$\cdot$ha^{-1}. Supplemental N was added at three rates and the soil incubated for 18 days at 25 °C. Soil water contents were maintained at approximately field capacity during the incubation.

Following the 18-day incubation, soil aggregate stability was determined by wet sieving. Aggregates of uniform size (6 mm) were initially wetted by capillary action and placed on a nest of four sieves: 0.25 mm, 0.5 mm, 1 mm and 2 mm. Sieves were mechanically agitated for 30 minutes and the oven-dry mass of soil remaining on each sieve determined (Table 18). Aggregates of sludge-amended soils were significantly more stable than the unamended control. Mean-weight diameter (MWD) of all treatments exceeded 1.9 mm compared to 0.83 mm for the control; however, differences among sludge and N treatments do not appear statistically significant. Greater than 90% of the aggregates from sludge-amended treatments remained in the 2 mm sieve, indicating minimal slaking as compared to the unamended control in which only 12% of the initial soil remained. Sludge-amended treatments also had lower percentages of soil dispersed (1.9 - 4.2%) than the untreated control (15.5%).

These preliminary data suggest that organic sludges have a positive effect on aggregate formation and stabilization. Much of this effect is due to increased soil carbon contents (Table 19). As the N addition rate was increased, soil carbon decreased indicating increased rates of microbial decomposition.

Table 18. Aggregate stability of sandy loam surface soil amended with pulp mill biosolids after 18-day laboratory incubation.

Biosolid Addition Rate	N Addition Rate	Mean-weight Diameter	Soil Fraction Retained				Soil Dispersed
			2 mm	1 mm	0.5 mm	0.25 mm	
tons·ha^{-1}	kg·ha^{-1}	--mm--	% dry mass				
Control	0	0.83	11.5	28.3	30.0	14.3	15.5
67	0	1.94	91.8	2.2	1.2	0.6	4.2
67	240	1.96	94.3	2.4	0.6	0.3	2.4
67	720	1.99	97.1	0.6	0.3	0.1	1.9
135	0	1.97	95.2	1.3	0.6	0.2	2.7
135	240	1.99	96.8	0.7	0.2	0.1	2.2
135	720	1.98	96.3	1.3	0.3	0.2	2.0

Table 19. Carbon content of soil amended with primary biosolids after 18-day incubation.

Biosolid Addition Rate	N Addition Rate	Carbon
tons·ha^{-1}	kg·ha^{-1}	- % -
Control	0	1.3
67	0	2.1
67	240	2.1
67	720	1.9
135	0	3.4
135	240	3.1
135	720	2.6

A larger-scale decomposition and aggregate stability experiment will be completed during 1997. In addition, runoff quantity and quality, and soil erosion from sludge-amended soils will be studied using a rainfall simulator.

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Presentations and Project-related Activities

"Pinus taeda seedling growth following application of ash and sludge residues from Kraft pulp mills: glasshouse and first-year field trials. L. A. Morris, and W. L. Nutter. Presentation, Land Application of Wastes in Australia and New Zealand: Research and Practice. Proc. 14th Tech. Session of the New Zealand Land Treatment Collective, Sep. 29-Oct. 4, 1995, Canberra, Australia, (p. 56-66).

"Agronomic value of pulp and paper byproducts as soil amendment and liming agents" M. H. Golabi, L. A. Morris, W. P Miller, L. M. Shuman, M. E. Sumner and G. Weeks. Soil Science Society of America Annual Meeting, Nov. 3, 1996, Indianapolis.

"Characteristics and potential for using pulp and paper mill wastes in forest production" L. A. Morris. Presentation, Ann. Meeting of the So. Sec., Air and Waste Management Assoc., Aug. 14-16, 1966, Bil

"Long-term environmental impacts of municipal wastewater irrigation to forests at Clayton County, Georgia, USA" W. L. Nutter, L, Phillpott and L. A. Morris. Presentation, Land Application of Wastes Australia and New Zealand: Research and Practice. Proc. 14th Tech. Session of the New Zealand Land Treatment Collective, Sep. 29-Oct. 4, 1995, Canberra, Australia, (p. 56-66).

Personnel

One new student, Rachel Cochran, was added to the project during 1996. Rachel is working on her MS with Dr. Miller and is evaluating the effects of mill residue additions on soil physical properties (see Special Project Reports). Several major changes are expected in 1997. Dr. Wade Nutter has announced his retirement, effective on June 30. Dr. Nutter has been a driving force in forest land application research for 30 years and the project will miss his expertise. Also, Dr. Mohammad Golabi is expected complete his crop studies during 1997 and will leave the project. We appreciate all of Dr. Golabi's efforts on the project and wish him the best in his future endeavors.