CONTRIBUTIONS OF SOIL ORGANIC MATTER TO PRODUCTION POTENTIAL OF SOILS AND SOME PROPOSALS FOR PRACTICAL APPLICATION

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Annex II

to the report of W. FLAIG

9 Lectures about: Contributions of soil organic matter to production potential of soils and some proposals for practical application.

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Preface

The increase of yield/ha is important for the food production of Indian Population. For this purpose firstly high yielding varieties of rice, wheat and other crops were bred. These demand a higher quantity of mineral fertilizers. Fertilization alone does not allow the realization of the full genetical potential of cereals. The inorganic ions of the fertilizers enhance also microbial activity. The microorganisms use the carbon from soil organic matter as source for energy and reproduction. As a consequence of decrease of soil organic matter unfavourable processes in soil, the substrate for production occur, which cause a reduction in plant production. Therefore an increase of yield by fertilization alone is limited.

Another way to overwhelm the biological border for the use of the genetical potential of cereals is to utilize the effect of bioregulators on plant metabolism, such as CCC, "Cycocel" to increase the resistance against lodging of wheat. There are some other substances in soil organic matter, which have bioregulating effects under unfavourable growth conditions. This influence of soil organic matteron yield is well known by the farmers as "humate-effect".

Therefore, it is evident, that special procedures for maintaining a critical status of soil organic matter should be followed. More research about humus is necessary. An intensive cereal production is then economical only, when yields are always maintained at a relatively high level. One should try to minimize the yield depressions by appropriate procedures.

The lecutes are an introduction into the problems. The transfer of results of basic research into practice are mentioned and the experiences about the utilization of new findings are summarized.

I have to thank Mr. M.S. Sachdev (M.Sc.) for his indefatigable help, for the revision of the lectures and for compiling the references.

Wolfgang FLAIG

New Delhi, 29. September 1973

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Lecture 3

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1. Importance of "slow releasing nitrogen" fertilizer for plant production.

The fertilization with inorganic nutrients is an absolute preposition of high and economic plant production. The macronutrients must be available to the crops in sufficient amounts during the whole vegetation.

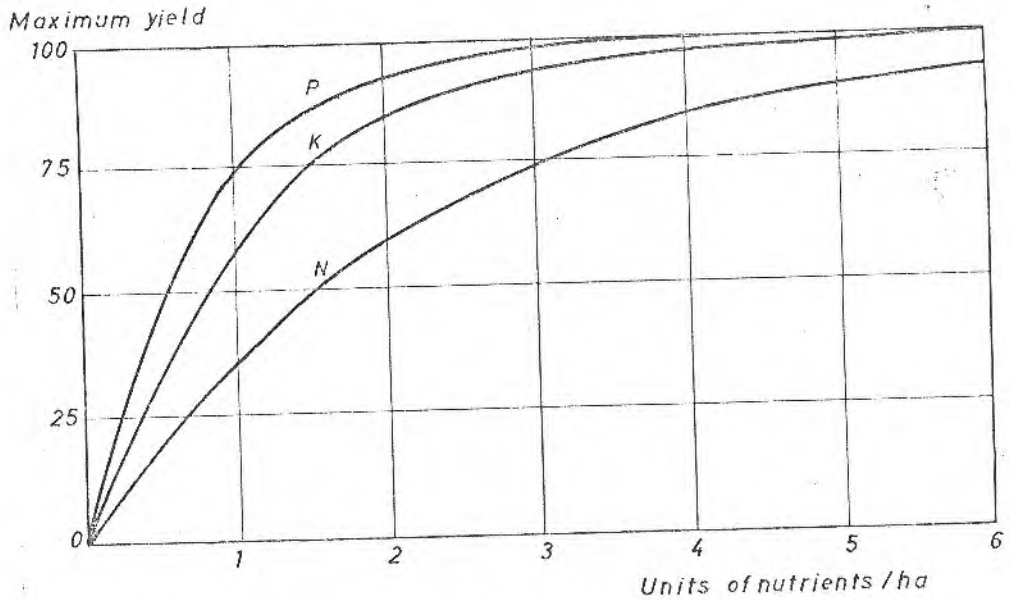


Fig. 1: Increase of yield by plant nutrients (according to Mitscherlich 1948).

From N, P, and K nitrogen has the smallest efficiency factor. To reach high yields - as high as possible - it is necessary to give corresponding high doses of nitrogen fertilizer. A single dosage of nitrogen for the total vegetation period is not possible, because high doses of salts such as ammonium or nitrate salts effect plasmolysis at the young plants. Nitrate ions or ammonium ions transformed in nitrate ions by nitrification can be leached by rainfall.

By these reasons nitrogen fertilizers must be given in two or three doses, which causes additional costs.

Therefore many attempts have been made for the synthesis of the so called "slow releasing" nitrogen fertilizers, which deliver organic bound nitrogen during the vegetation period by the action of microorganisms in form of ions, which can serve for plant nutrition. Several products have been proposed. Many of them would be qualified as "slow releasing" nitrogen fertilizers, but they are mostly too expensive for agriculture and only useful for special purposes. Some of them were cheaper but not appropriate. Others have been made by coating the granulates with plastics or sulphur. The rate of release of nitrogenous ions in soil solution is lowered by diffusion.

2. Sulfonated lignins as initial materials.

In this lecture it will be reported about the synthesis of a more or less "slow releasing" organic nitrogen fertilizer, which is produced by oxidative ammonisation of lignin waste products of cellulose industry, especially of ligninsulfonates.

The considerations for production of a slow releasing nitrogen fertilizer from lignin waste products can be summarised as follows:

(1) by different organic bindings of nitrogen in the initial material a release of nitrogen by microbial mineralization of the organic material should occur at different rate during vegetation period.

(2) Nitrogen from this fertilizer should be less leached than that from mineral fertilizers, because 50 per cent of N-lignin are of high molecular weight components.

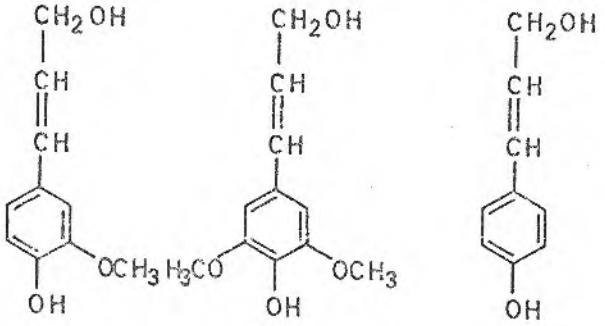
(3) Specific effect on plant growth may be due to the presence of low molecular weight degradation products from initial lignin material, which are similar to those.

The further reason to use ligninsulfonates for production, was that nearly the half of the wood in form of the lignin containing residues will be used for a further technical product and is not only burned as till now. The wast liquors can generally no more be discharged into rivers and lakes because of the pollution of the environment. To produce an organic nitrogen fertilizer from lignin residues by oxidative ammonisation is therefore also an important contribution to the hygiene of the environment.

At first the chemical reactions will be described which occur during the oxidative ammonisation of lignin sulfonates.

2.1 Differences of the chemical structure of spruce and beech lignin.

Mostly spruce and beech wood are used for cellulose production, in some cases also the straw of cereals.



Coniferous trees	+		
Deciduous trees	+	+	
Graminees	+	+	+

Fig. 2: Monomers of lignin.

The three monomers of the different lignins have a different content of methoxyl groups. This fact is important because the fixation of ammonia in ligninsulfonates is accompanied by a decrease of methoxyl content. Coniferous lignin consists mainly of coniferyl alcohol. Beech lignin is formed by a copolymerization of coniferous and sinapyl alcohol, whilst graminaceous lignin contains besides these monomers a larger amount of p-coumarylalcohol. If only the methoxyl content of the lignin would be responsible for the fixation of ammonia, then the nitrogen content of the N-Lignin would be very different. But there are some other reactions such as hydroxylation of p-coumaryl- and coniferylalcohol, which also contribute to the fixation of ammonia.

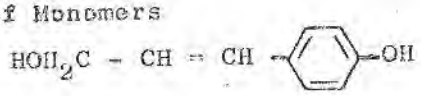
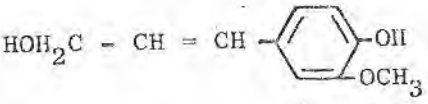
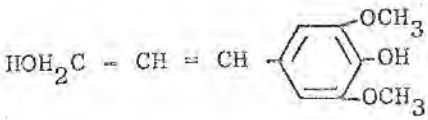
	Spruce	Beech
Content of lignin (Klason)	27 %	21 %
Methoxyl content of lignin	13 %	22 %
Composition in % of Monomers		
p-Coumarylalcohol 	14 %	5 %
Coniferylalcohol 	80 %	52 %
Sinapylalcohol 	6%	43 %

Fig. 3: Lignin and methoxyl content as well as the content of monomers in the lignin of spruce and beech (weight %).

The lignin content of the plants is different. Spruce contains 27% lignin and beech 21% lignin. The methoxyl content of beech lignin is nearly the double of this of spruce lignin. To build up a structure scheme of the lignins of spruce and beech different amounts of units are necessary as it can be seen from tab. 1.

Some two further results of lignin research must be mentioned in connection with a combined cellulose and organic nitrogen fertilizer production, because in this case lignin residues are no more a waste product.

Tab. 1: Methoxyl and nitrogen content of different lignin in per cent (BONDY and MEYER, 1948).

	% OCH_3	% N
Hartholz	20,0	0
Weichholz	15,0	0,2-0,3
Gramineen	10,0	1,2-1,6
Leguminosen	5,0	2,9-3,4

The methoxyl content of the lignins of grasses and leguminoses is lower than this of the trees, but the nitrogen content is higher. This means that the initial material contains already nitrogen. If lignin sulfonates are used, which have been produced by cooking the wood with ammonium bisulphite, the initial material contains up to 6% nitrogen. The efforts to increase the nitrogen content are then not so high.

The cooking process with ammonium bisulphite has some further advantages. The quality of cellulose is high and the free and fixed ammonia contained in the digesting liquor can be reduced as fertilizer nitrogen. The same is also the case for neutral sodium sulphite as pulping agent for production of corrugated board.

When under special circumstances the production of organic fertilizer is the main reaction and not that of cellulose, plants

with a high lignin content may become important.

Table 2: Lignin content of different plants in different climatic zones (MIGITA and KAWAMURA 1944)

Coniferus trees		
Picea abies (L.) Karst.	Pinaceae	27,5
Pinus densiflora Sieb. et Zucc.	Pinaceae	27,5
Cryptomeria japonica D. Don	Pinaceae	34,1
Chamaecyparis obtusa Sieb. et Zucc.	Pinaceae	31,0
Larix gmelinii Leder	Pinaceae	29,9
Decicuous trees		
Cold temperate zone		
Populus davidiana Dode	Salicaceae	20,1
Fagus crenata Blume	Fagaceae	21,0
Temperate zone		
Alnus firma Sieb. et Zucc. var. Sieboldiana Winkl.	Betulaceae	23,2
Quercus serrata Thunb.	Fagaceae	19,3
Euptelea polyandra Sieb. et Zucc.	Euptelaceae	25,7
Platanis orientalis L.	Platanacea	22,8
Cornus controversa Hemsl.	Cornaceae	24,0
Tropical zone		
Dalbergia cochonchinensis Pierre	Leguminosae	33,4
Pterocarpus santalinuns L.	Leguminosae	37,6
P. macrocarpus Kurz.; P. indicus Willd.	Leguminosae	40,9
Cassia siamea Lam.	Leguminosae	34,3
Diospyros Ebenum Koenig	Ebenacae	45,8
Macaranga hypoleuca Müll.Arg.	Euphorbiaceae	28,0
Phyllostachys reticulata C. Koch	Gramineae	25,4

Many plants in more tropical zones have a high content of lignin. This could be used for the production of organic N-fertilizers, when not the cellulose as such, but the carbohydrates formed by hydrolysis of cellulose with mineral acids, are important products for animal nutrition. According to a rough estimation both would

be needed approximately to the same amount as by-products either for fertilizers or for feedstuffs. The laboratory experiments demonstrate that in seminarid and tropical zones organic nitrogen fertilizers may be more effective for plant production than in humid climate. The plants with a higher lignin content grow in the mentioned zones and could be used as raw materials.

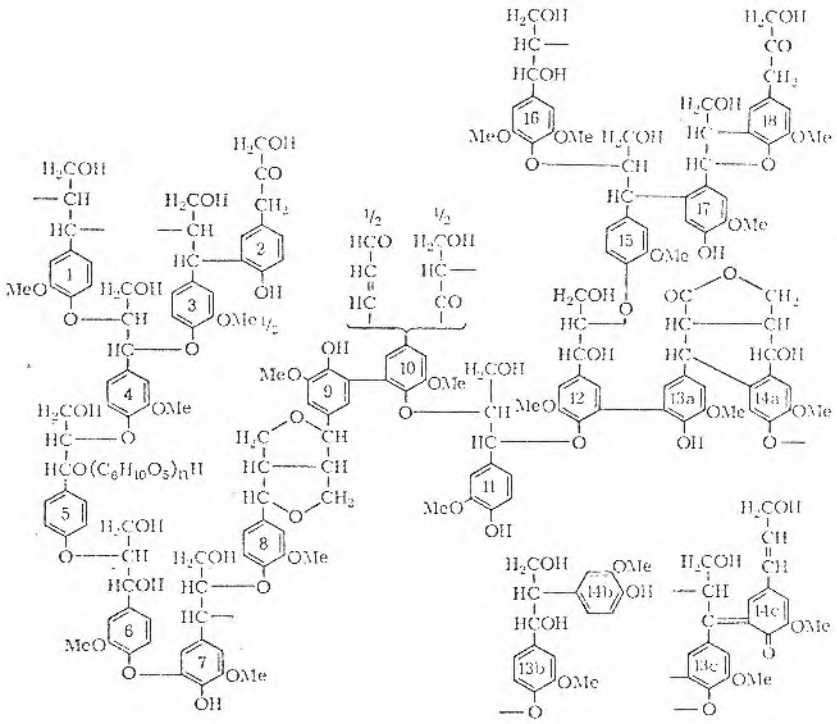


Fig. 4: Structure scheme of coniferous lignin (FREUDENBERG et al. 1968)

Some reactions during sulfonation and oxidative ammonisation of lignin-sulfonates will be explained with the structure scheme

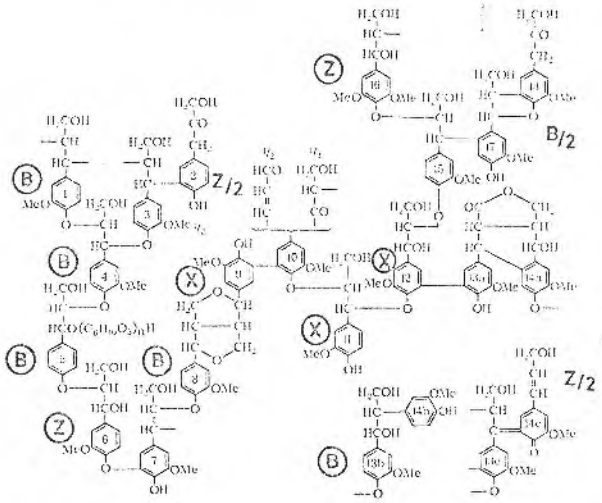
of FREUDENBERG et al., 1968). The constitution model consists of 18 units, 14.5 (80%) of coniferyl-, 2.5 (14%) of p-coumaryl- and 1 (6%) of sinapylalcohol molecules. During polymerization a loss of 1,96 atoms of hydrogen and an addition of 0,4 molecules of water per unit of 9 carbon atoms occurs. According to a proposal of HOLMBERG (1942) the basic formulae of the different lignins are calculated for 9 carbon atoms, because all lignins are composed by phenylpropane - C_6-C_3- units. The differences between the lignins of different plants and alterations in the high molecular weight structure can be better overlooked in this way. One does not give only the elemental analysis as it is generally done in the case of other organic compounds.

It shall be mentioned that the connection between units 2 and 3 as well as units 13 and 14 can partly also occur by an o-quinonid structure (FREUDENBERG and HARKIN, 1964). Such types of compounds add relatively easily ammonia.

Furthermore methoxyl groups in semiquinonoid structures have in alkaline solution more a character of ester than of other groups and can be hydrolyzed in alkaline solution. The o-diphenols formed in this way are very reactive compounds for transformation of ammonia in organic bound nitrogen or for further condensation reactions even for heterocyclic nitrogen compounds.

2.2 Reactions of lignin with salts of bisulphite.

Not all units of the molecule react with the acid bisulphite salts, such as calcium, magnesium or ammonium. The sulfonation depends upon the substitution of the carbon atom 1 - the next to the phenyl-ring - of the side chain. One differentiates between the units X, Z and B. Without going further into details, it was found that sulfonisation occurs exclusively at the secondary alcohol group of the carbon atom 1 of the side chain. Thereby not only the unsubstituted secondary alcohol groups in the units Z - were sulfonated. After the cleavage of the dialkylether linkage in the units X and the cleavage of the alkyl-arylethers in the units B by the acidic sulphite solution the additionally formed secondary alcohol groups are also suffonated.



Sulfonated		*Not sulfonated*
Units	*1,4,5,6,8,9, 11,12,13b,16, 10/2,14c/2, 17/2*	*2,3,7,15,18, 10/2, 14c/2, 17/2*
total	*ca. 11,5 units or 64 %*	*total ca. 7,5 units or 36 %*

Distribution of sulfogroups in the units of lignin on the single groups (FREUDENBERG and NEISH, 1968):

	Found or estimated	*Deducted from model*
Group X	*0,15*	*0,17*
Group Z	*0,15*	*0,20*
Group B	*0,30*	*0,28*
Sum	*0,60*	*0,65*

Fig. 5: Sulfonated units in the model of the constitution of spruce lignin.

In the graph the numbers of the units are mentioned, which are sulfonated and which are not. 11.5 units or 64% are sulfonated and 7.5 units or 36% remain unsulfonated.

By sulfonation the molecule of lignin is degraded to smaller parts. For the following oxidative ammonisation some reactions during sulfonation are very important.

First of all the lignin becomes water soluble and the oxidative ammonisation can be carried out in a homogenous system. This has many advantages for the technical process.

Furthermore, the content of phenolic hydroxyl groups may be increased by the cleavage of alkyl-arylether groups during sulfonation. Thereby the possibilities of reaction with ammonia become increased.

During the process of oxidative ammonisation of the sulfonates the methoxyl content decreases and concurrently the content of organic bound nitrogen increases. The phenols which are formed by the methylether cleavage react with ammonia to different compounds, which contain nitrogen in different organic linkage.

The fixation of nitrogen in organic linkages occurs only when oxygen and ammonia are present contemporarily. For the case of at first oxidation and then ammonisation or vice versa nearly no nitrogen fixation can be observed.

Lignin treated with mineral acids such as hydrochloric or sulphuric acid is changed partly by condentation and partly by

cleavage reactions. Primary results of oxidative ammonisation of such pretreated lignins show however that the production of organic nitrogen fertilizers is also possible. Only for large scale production the special conditions must be worked out.

In countries in which humus problems in connection with physical properties of the soil - for instance erosion and others - exist, in the type of organic nitrogen fertilizer on lignin basis- also an important material for humus formation - not only the nitrogen component but also the organic part is of great interest. It was found that an increase of soil carbon content of about 10% and of nitrogen content of about 60% occured in pot experiments with pseudogley-chernozem after ten years under our humid climatic conditions.

2.3 Degradation of ligninsulfonates in alkaline solution.

Only by the use of labelled compounds a part of the mechanism of degradation of lignisulfonates could be elucidated.

2.31 in presence of oxygen

In the presence of oxygen with labelled lignisulfonates it could be shown that their degradation is faster than that of lignin itself. When the sulfonic acid rest is split off mainly vanillin and acetaldehyde is formed to a higher amount. Acetaldehyde is able to condense with phenolic compounds. This reaction may

lead to alterations of the final products such as organic nitrogen fertilizer (KRATZL et al. 1964, 1966).

2.32 in absence of oxygen

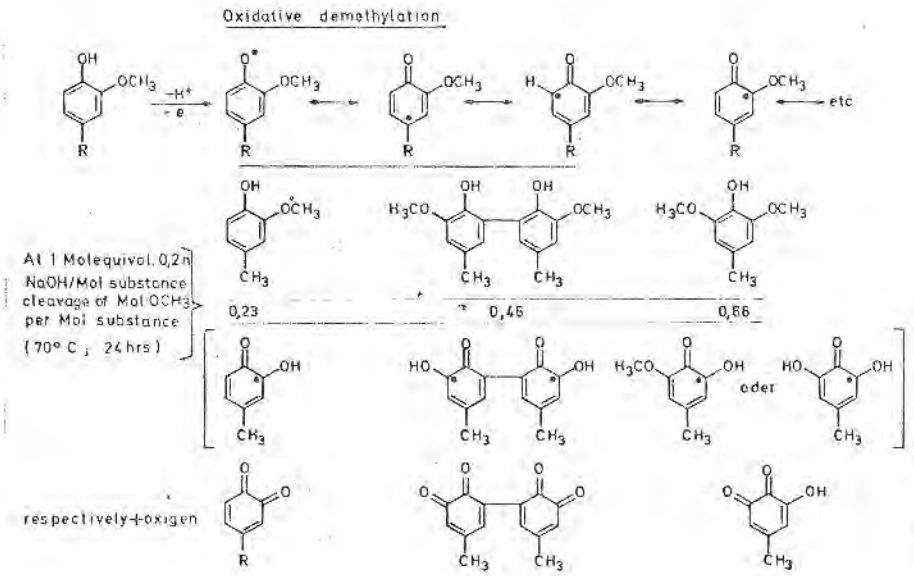


Fig. 6: Oxidative demethylation

In 0.2 N NaOH solution methoxylated phenols split off different amounts of methylalcohol. The amount increases with the tendency of these compounds to form semiquinonoid intermediates.

Dimerisationof the monomers and formation of o-diphenols has been established. By further oxidation corresponding quinones are formed (KRATZL et al. 1967).

In connection with the use of ligninsulfonates from beach for the production of organic nitrogen fertilizers it is interesting that the syringyl components are remerkably faster deme-thylated

than these of guaiacyl type. This may be the reason, that lignin of beech wood is faster transformed by oxidative ammonisation than this of conferous trees.

3. Oxidative ammonisation of Ligninsulfonates.

3.1 Nitrogen fixation in dependence of oxygen consumption.

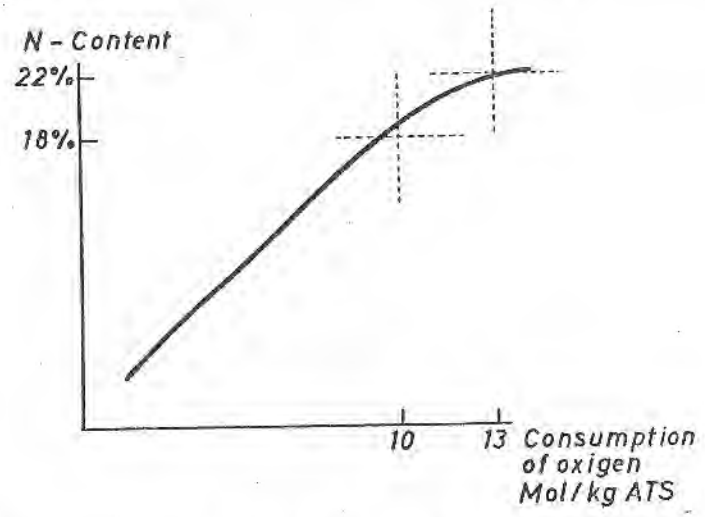


Fig. 7: Scheme of the formation of "N-Lignin "

With increasing consumption of oxygen the nitrogen content of the final product increases. After a turnover of 10 to 13 Mol oxygen per kg dry matter of lignin containing waste liquor the N-Lignin contains 18 to 22% of total nitrogen. By a good mixture of gaseous and liquid phase the reaction is carried out at 10 to 30 atmospheres and 110-130°C. The distribution of nitrogen in the organic nitrogen fertilizer N-Lignin is :

Total N 18-20% N
from this (total N content - 100%)
30-40% NH_4 - N
Ca 10% "Amide - N"
50% bound in other organic form.

"Amide-N" is nitrogen, which can be hydrolized with 30% sodium hydroxide solution after the destillation of the ammonium ions with magnesium oxide. With urease about 11% of the total nitrogen of N-Lignin can be determined as urea nitrogen. The total amount of urea in the product is about 5%. N-Lignin is soluble respectively dispersable in water. 50% of the product consists of high molecular weight substances, which do not dialize. In the high molecular weight fraction are about 40-45% of the organic bound nitrogen. This is important in connection with its properties as nitrogen fertilizer because this part of N-Lignin is not leached by heavy rainfall.

Reductive cleavage with sodium amalgam according to Burges et al. (1964) in N-Lignin from spruce the main product is vanillic acid. This and other physiologically active substances cause special properties of this organic nitrogen fertilizer.

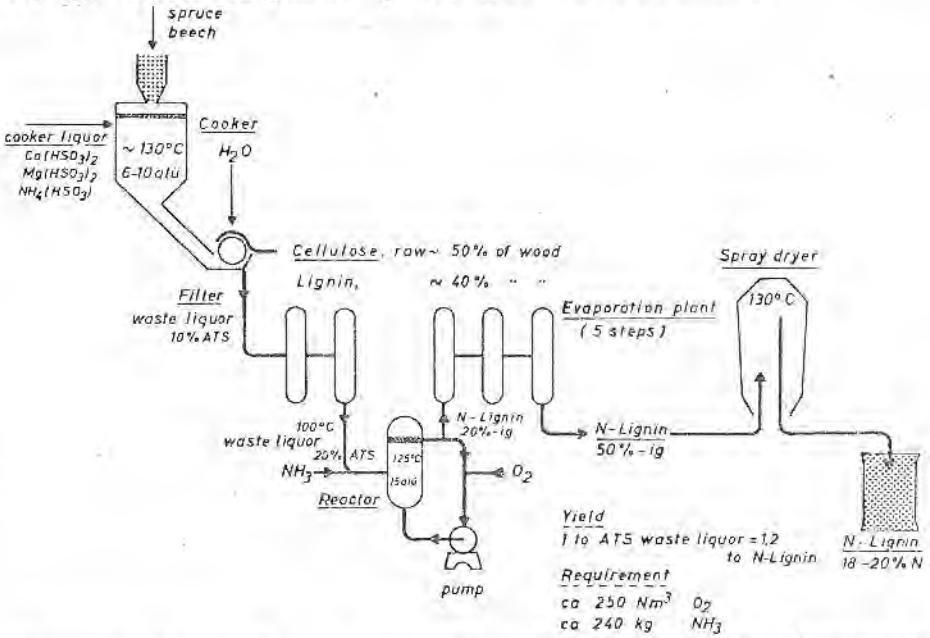


Fig.8: Flow scheme for production of N-Lignin (organic nitrogen fertilizer) from waste liquors of cellulose industry.

After cooking the wood with sulphite solution the waste liquor is filtrated from cellulose and preconcentrated to a dry matter content of 20%.

In the reactor the waste liquor is oxidized and ammonised at a temperature of 125°C and a pressure between 12 and 15 atmospheres by circulating with a pump. After one or two hours the reaction mixture is concentrated by an evaporation system to a dry matter content of about 50% and then dried in a spray dryer to a powder. The powder is granulated and bagged for shipment.

At the moment a pilot plant is running with 500 tons per year. It was built by Lurgi in Frankfurt/Main and Chemische Fabrik-Kalk in Köln in cooperation.

For 1 ton N-Lignin 0,85 ton of dry matter from sulphite waste liquor is required. This corresponds to 8,5 ton sulphite waste liquor taken at an average 100% dry matter content of 10%. The requirement of ammonia is about 0,25 ton and the requirement of oxygen about 28 Nm^3. The yield of N-Lignin is about 110% of the theory.

4 Oxygen uptake during ammonisation and effect of the formed products on plant growth (pot experiments).

We began with pot experiments in the year 1960. In the first years mainly influence of production conditions on suitability as fertilizer had been investigated.

Table 3: Yield of wheat grain of some pot experiment with N-lignins of different composition due to the conditions of production (SOCHTIG, 1972)

Fertilizer oxygen consumption in Mol O_2/kg		total N %	g N/ pot	rel.yield fertilization			N-uptake % fertilization		
				inorg.	1:1	Org.	inorg.	1 : 1	org
NH_4NO_3		35,0	1,2	100	-	-	79	-	-
N-Lignin 1*)	8,5	16,55	1,2	"	99	92	-	62	53
" 2*)	9,0	18,22	1,2	-	104	94	-	68	54
" 3*)	13,9	20,90	1,2		110	105	-	71	60
" 4*)	15,3	21,38	1,2		112	95	-	73	60
" 5**)	13,0	18,05	1,2		104	91	-	67	60
NH_4NO_3	-	35,0	2,4		-	-	66	-	-
N-Lignin 2*)	9,0	18,22	2,4		89	107	-	61	80

*) produced from NH_4 - bisulfit - liquor (spruce)

**) produced form Ca-bisulfit-liquor (spruce)

The initial material of the production of N-lignin have been Ammoniumbisulphite and Calciumbisulphite. The amount of the cation which was in the waste liquor influences the properties of N-lignin. With oxidation of N-lignin the yields are little less, but with product 3 they are higher than other of Ammonium nitrate. In the case of highest oxidation a decrease of yield is again observed. Mixtures of pure mineral- fertilizers and N-lignin in a ratio 1: 1 show with increasing oxidation not only an increasing yield which is nearly in all cases higher than this with mineral, fertilizers, but also an increase of nitrogen uptake. Differences in yield are less, than it can be explained by a diminished uptake of nitrogen.

The effect of N-lignin from Calciumbisulphite waste liquor at the same degree of oxidation are less effective than those form Ammoniumbisulphite liquors. Distinct increase of yield by N-lignin can be observed in the case of higher doses of Nitrogen, which a results a depression of the yield in the case of mineral fertilizers and their mixtures with N-lignin. This effect can not be explained alone by total uptaken nitrogen.

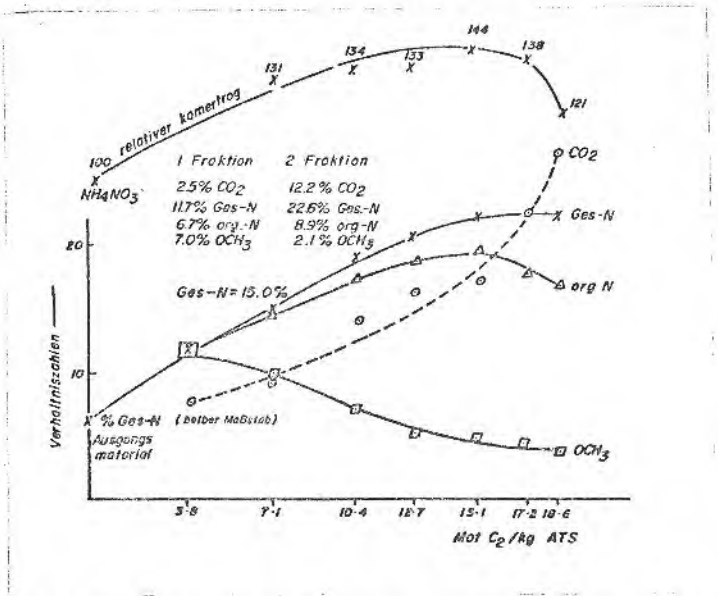


Fig. 9: Composition of differently produced N-lignin and the effect on plant yield at temporary water saturation of soil.

Further experiments about the influence of the production conditions on the properties of N-lignin has been made also under abnormal conditions for instance under temporary water saturation of soil.

Relative yield of grain increases up to a middle oxidation degree. By higher oxidation the yield decreases, although the nitrogen content in N-lignin had increased further. The content of organic bound nitrogen is higher in the case of the middle oxidised products. By higher oxidation degree a strong increase of released CO_2 occurs. It can be assumed that at higher oxidation different degradation processes are occuring which form mostly low molecular weight compounds. These may lead to compounds which have a negative influence on plant growth in higher concentrations.

Investigation with Ultra-centrifuge demonstrate that the average particle weight increases with oxidation to a certain amount and decreases again with higher oxidation. These results have caused that the further N-lignin production was made under middle conditions of oxidation.

5. Principal differences of fertilizer effect between mineral nitrogenous fertilizers and N-lignin.

In the following some typical examples are selected from a large number of experiments. It was mentioned before, that the nitrogen is differently organic bound. Only a third of the total nitrogen is ammonium nitrate, two third must be mineralised by

microorganisms, before they can be uptaken by the plant. Further more in the low molecular weight part of N-lignin organic compounds are present which can have a specific influence on plants. They are also transformed by the activity of microorganisms. For this reason, the effect of N-lignin has been proved in comparison to ammonium nitrate on fertile soil on the one hand and sterile on the other.

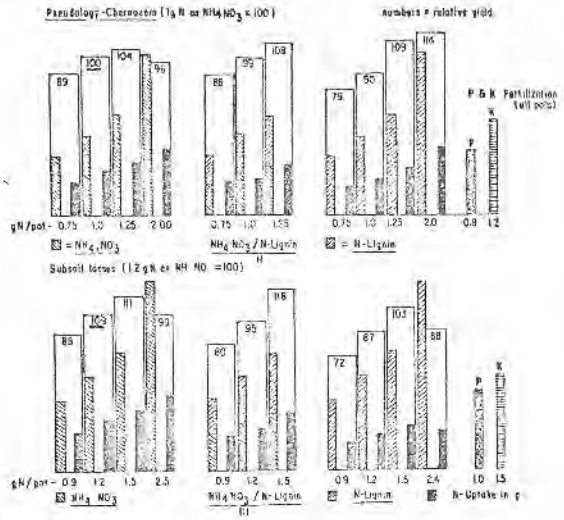


Fig. 10: Yield of grain of summer wheat on pseudogley chernozem and its sub soil "Loess" at different levels of N-lignin (Pot experiment).

One of the tests has been done with pseudogley-chernozem from Hildesheim, one of the best German agricultural soil and its sterile sub-soil loess. The mineral composition of this two are the same. Both the soils differ in their content of humus and in their microbiol activity. In both the experiments the nitrogen fertilization was increased from 0.75 g N up to 2 g N per pot. The doses in the case of loess have been a little higher according to the difference of nutrients in comparison to the surface soil.

The yields with ammonium nitrate (left part of the figure), which was added to the soil totally at the beginning of the experiment, shows the typical curve with a maximum at 1.25 or 1.5 g N respectively per pot. Both curves are very similar. Another information is received by fertilization with N-lignin alone. While in the case of chernozem a certain loss of yield occurs at lower doses, the curve of yield goes up to the highest dose. The yield is higher than in the case of highest possible yield with mineral fertilizers.

The uptake of nitrogen (black columns) show not so large differences that the differences of yield could be explained by uptake of nitrogen. Two reasons may be possible for the differences in the last part of the curve of yield:

(1) By a more adequate supply of plant with nitrogen in the case of N-lignin;
(2) by the effect of organic constituents of N-lignin on plant growth and yield.

In the case of use of fertilizerz on the sterile sub-soil, the yields with N-lignin are not so high as in the case of mineral fertilizers. The curves of uptake of nitrogen demonstrate clearly that the efficiency of nitrogen is due to less mineralisation of N-lignin. The highest dose of N-lignin effects a inhibition, which is no more explicable by effect of nitrogen. For this

inhibition effect low molecular weight parts of N-lignin are responsible. These are not degraded or transformed in the sterile soil and apparently not sorbed, because this type of soil has no orgahic substance.

It was observed that on such sterile soils specially favourable effect occur when commensurate amounts of fertilizers the both fertilizers, N-lignin and Ammonium Nitrate have been mixed in ratio 1:2 (group of columns in the middle of the figure). Also, here it can be observed that partly higher yields can be obtained by lower uptake of nitrogen.

By the reason that a very favourable effect can be obtained by mixtures of N-lignin with mineral fertilizers different mixtures have been scrutinised. It could be shown that mixtures with Ammonium Nitrate or Calcium Ammonium Nitrate are more favourable than mixtures with Ammoniumsulphate, urea and some other nitrogenous organic materials under our humid conditions.

In the last time, also mixed fertilizers with phosphate and potassium has been produced in larger quantities.

Field experiments during 3 years which have not been conducted in Germany only but also in different other European countries proved the positive effect of this mixed organic fertilizers. The results will be evaluated in a Symposium in autumn this year.

For evaluation of experiments with N-lignin or its mixtures with inorganic fertilizers several more factors must be born in mind than with inorganic fertilizers alone as we will see in the next lecture.

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