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ALKALI REQUIREMENTS FOR IMPROVING DIGESTIBILITY OF HARDWOODS BY RUMEN MICRO-ORGANISMS

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WOOD and wood residues provide a potential source of energy for ruminants because they contain 70 to 80% carbohydrate. In the United States alone, the quantities of low-quality wood and nonutilized residues can amount to more than 100 million tons per year (U. S. Forest Service, 1965). These wood residues have not been used successfully as ruminant feed supplements because of the poor digestibility of their carbohydrate components. Numerous attempts have been made to improve the digestibility. During World War II, Scandinavian countries used chemical wood pulps as a feed component for ruminants (Hvidtsen and Homb, 1951; Nordfeldt, 1951). After the War, the practice was discontinued because of high costs.

The effect of NaOH in improving the digestibility of straw has long been known (Beckmann, 1919). Other workers (Pew and Weyna, 1962; Stranks, 1959) have reported improved digestibility of hardwoods by cellulases and rumen-inhabiting bacteria after pretreatment with dilute NaOH. The increased rate of digestion of a dilute NaOH-treated hardwood by woodrotting fungi has been described (Tarkow and Feist, 1969). Wilson and Pigden (1964) have described the improvement in digestibility of poplar after treatment with dilute NaOH. Their work showed that up to 10% NaOH (based on the weight of the wood) is required for maximum digestibility.

The mechanism for this improvement in digestibility on treatment with dilute NaOH has been described (Tarkow and Feist, 1969). The work presented here, based on this mechanism, indicates the minimum NaOH requirement for maximum improvement in digestibility of two hardwoods, quaking aspen and red oak. The effect of lignin content on digestibility is also described for 12 hardwood species.

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Materials and Methods

The *in vitro* technique used to determine digestibilities of the treated and untreated hardwood samples with rumen fluid was described recently (Mellenberger *et al., in press*). Wood samples were ground through a 40-mesh screen in a Wiley mill. Samples of air-dry black ash, American basswood, yellow birch, paper birch, eastern cottonwood, American elm, silver maple, sugar maple, red oak, white oak, quaking aspen and bigtooth aspen were investigated.

The NaOH treatments consisted of slurrying the air-dry, ground wood at room temperature in various amounts of 0.5 or 1% NaOH solutions, with occasional stirring, for the times indicated (tables 1 and 2). The treating times were determined previously (Tarkow and Feist, 1969).

The NaOH-to-wood ratio was varied by using different quantities of NaOH solution for 5 g of wood sample. After the treatment, the samples were washed thoroughly with distilled water and air-dried; yields were determined and lignin contents measured (Saeman *et al.*, 1954).

Results and Discussion

Twelve species of hardwoods are listed in table 1 with their *in vitro* digestibilities and their lignin contents. The lignin contents shown were obtained after NaOH treatment. These lignin contents were only slightly lower than those found for untreated wood, indicating that little lignin was removed by the treatment. Yields after dilute NaOH treatment are given for all but bigtooth aspen. Values for digestibility are averages of duplicates. Standard deviation was 2%.

The *in vitro* digestibility of untreated hardwoods varied greatly (table 1). When the hardwoods were treated with 1% NaOH at room temperature, their digestibilities were markedly increased. There was, however, a difference in response to the treatment. For example, the digestibility of basswood increased from 5 to 56%, whereas the digesti-

TABLE 1. DIGESTIBILITY OF NaOH-TREATED HARDWOODS[a]

Sample	*In vitro* digestibility[b]		Lignin content[c]	Yield[d]
	Before treatment	After treatment		
	%	%	%	%
Black ash *Fraxinus nigra*	17	36	20	91
American basswood *Tilia americana*	5	56	20	89
Yellow birch *Betula alleghaniensis*	5	19	21	94
Paper birch *Betula papyrifera*	9	38	21	92
Eastern cottonwood *Populus deltoides*	4	12	24	93
American elm *Ulmus americana*	9	14	23	93
Silver maple *Acer saccharinum*	26	41	18	92
Sugar maple *Acer saccharum*	6	28	23	87
Red oak *Quercus rubra*	3	14	24	86
White oak *Quercus alba*	4	20	23	89
Quaking aspen *Populus tremuloides*	35	50	20	87
Bigtooth aspen *Populus grandidentata*	38	..	20	..

[a] 5 g wood treated with 100 ml of 1% NaOH; equivalent to 20 g NaOH per 100 g wood.
[b] Average of duplicate determinations. Standard deviation, 2%.
[c] Lignin content measured on treated wood.
[d] Residual weight as percent of original weight following alkali treatment, washing and drying.

bility of red oak increased from 3 to only 14%. This difference in response to NaOH treatment appears to be related to the lignin content of the wood. The results in figure 1 show that as lignin content of the treated wood increases, *in vitro* digestibility decreases. Saarinen *et al.* (1959) have shown that, in general, the digestibility of partially delignified wood was also inversely related to lignin content.

Twenty g NaOH per 100 g wood were used in the NaOH treatments just described. Two species, quaking aspen and northern red oak, were chosen for investigation of the NaOH requirement for maximum digestibility in this part of the study.

The *in vitro* digestibilities of quaking aspen and northern red oak samples treated with various amounts of dilute NaOH are shown in figure 2. The experiments with aspen are detailed in table 2. With quaking aspen the treatment time with 1% NaOH was 1 hr. and with 0.57. NaOH, 2 hours. It was shown previously (Tarkow and Feist, 1969) that treatment time is inversely proportional to NaOH concentration. Increasing treatment times beyond those indicated did not increase *in vitro* digestibility. It can be seen from figure 2 that the NaOH requirement for maximum digestibility of both species lies between 5 and 6 g of NaOH per 100 g of wood.

The value of 5 to 6 g of NaOH per 100 g of wood is most significant. It was suggested previously that the additional swelling of hardwoods in water following treatment with dilute NaOH is a result of the cleavage of ester bonds on xylan chains acting as cross-links that restrain swelling (Tarkow and Feist, 1968, 1969). The NaHO also saponifies acetates in the wood. Knowing the amount of esterified and non-esterified uronic acid groups and acetyl groups in the wood, it is possible to calculate the NaOH requirement for saponification and therefore for maximum digestibility. The results of these calculations are shown in table 3. The NaOH calculated as necessary to saponify acetate and uronic esters is 5.1 g of NaOH per 100 g of wood for the aspen and 4.5 g of NaOH per 100 g of wood for the oak. The observed values found for the NaOH requirement for maximum digestibility are 5 to 6 g of NaOH per 100 g of wood (figure 2). This is in good agreement with the calculated values and supports the hypothesis that the cleavage of uronic ester cross-links by NaOH results in additional subsequent swelling in water (accessibility).

TABLE 2. EFFECT OF NaOH TREATMENT ON *IN VITRO* DIGESTIBILITY: OF QUAKING ASPEN

NaOH concentration	Time	G NaOH per 100 g wood	Yield	*In vitro* digestibility
%	Hr.		%	%
0	..	0	100	35
1	1	2	98	41
1	1	4	96	48
1	1	6	95	51
1	1	8	94	50
1	1	10	93	54
1	1	20	87	50
0.5	2	2	98	47
0.5	2	4	95	50
0.5	2	6	95	55
0.5	2	8	93	53

* 5 g samples of wood were treated with various amounts of aqueous NaOH solutions for the times shown. G NaOH per 100 g wood was calculated from the amount of aqueous NaOH used.

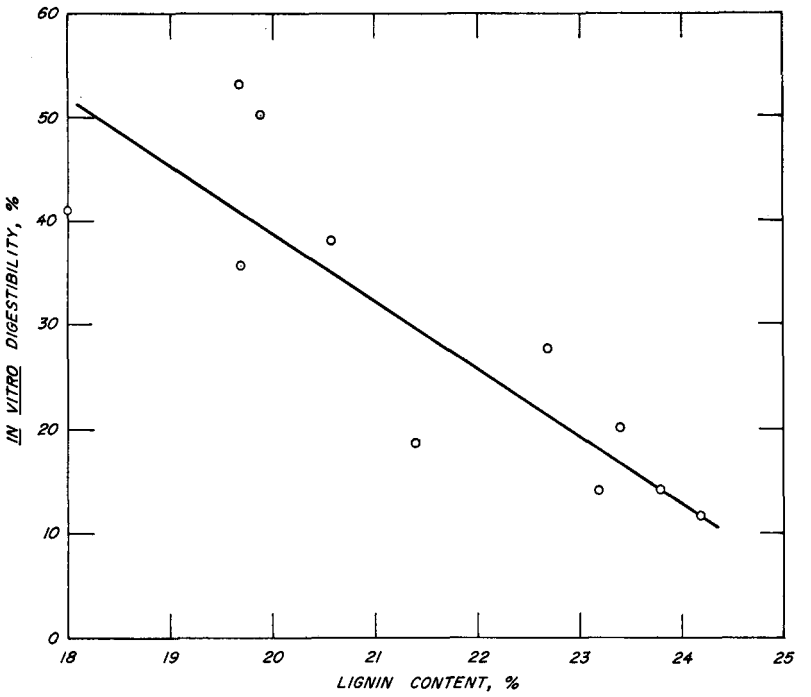

Figure 1. Relationship between in vitro digestibility and lignin content (of treated wood) for hardwoods listed in table 1 treated with 1% NaOH for 1 hr. at room temperature.

This may account for the increase in digestibility of hardwoods.

It is also significant that at a treating level of 5 to 6 g of NaOH per 100 g of wood and using a 1% NaOH solution the yield for aspen was 94%. About two-thirds of the 6% loss in mass is due to the unavoidable loss of acetyl groups following treatment with alkali and washing with water. This means that with certain hardwoods increased diges-

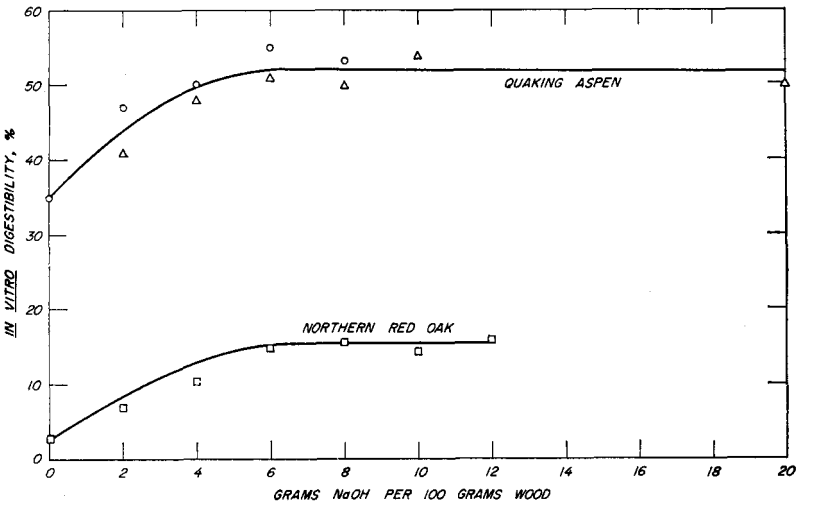

Figure 2. Relationship between digestibility and degree of treatment (grams of NaOH per 100 g of wood). Quaking aspen treated with required volume of 0.5% NaOH, room temperature, 2 hr. (o); 1% NaOH, 1 hr. (A); and northern red oak. 1% NaOH, 4 hr. (□).

TABLE 3. CALCULATED NaOH REQUIREMENT FOR MAXIMUM SUBSEQUENT SWELLING IN WATER AND DIGESTIBILITY OF QUAKING ASPEN AND RED OAK

Species	Uronic acid	Total carboxyl content[a]	Free carboxyl content[b]	Uronic ester content[c]	Acetyl content		Calculated NaOH requirement	
	%	Meq. per 100 g wood	Meq. per 100 g wood	Meq. per 100 g wood	%	Meq. per 100 g wood[d]	Meq. per 100 g wood[e]	G per 100 g wood
Quaking aspen	4.2	23.9	7.2	16.7	4.5	104.7	128.6	5.1
Red oak	4.4	25.0	9.1	15.9	3.8	88.4	113.4	4.5

[a] Calculated using uronic acid molecular weight of 176.
[b] From calcium ion-exchange value (Millett, Schultz and Saeman, 1958).
[c] Total carboxyl content less free carboxyl content.
[d] Acetyl molecular weight equals 43.
[e] Uronic ester content plus acetyl content plus free carboxyl content.

tibility can be obtained by a simple treatment at room temperature without a serious loss of material.

Summary

The *in vitro* rumen digestibility of hardwoods increases following treatment with dilute NaOH for 1 to 4 hr. followed by washing. The response, however, varies inversely with lignin content of the treated materials. Digestibility rises to a maximum value with increasing ratio of alkali-to-wood. The minimum ratio at the maximum digestibility appears to be independent of species and is 5 to 6 g NaOH per 100 g of wood or about 100 lb/ton. The maximum digestibility with aspen is about 50%; with red oak, about 15%. This optimum ratio of NaOH to wood is in good agreement with the value calculated on the assumption that the only function of the alkali is to saponify ester linkages, thereby permitting additional swelling in water.

Sodium hydroxide is inexpensive, and treating conditions are mild (room temperature, 1% NaOH solution). It is possible that washing of the treated wood is unnecessary or, at most, slight neutralization by weak acid may be required. These facts indicate that dilute NaOH treatment of certain hardwoods could be a potential method to provide energy feeds for ruminants. A variety of hardwoods could be utilized—thmaximum digestibility possible being dependent on lignin content. Feeding tests with ruminants using alkali-treated hardwoods are being conducted and will be described in a future report.

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