Tobacco Stalks, Hemp Hurds, and Sorghum Bagass as Sources of Cellulose for Making High-Quality Paper

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By E. B. LEWIS, DANA G. CARD, and J. S. MCHARGUE'

Research on industrial uses of agricultural byproducts in America has had to do mostly with farm products in the channels of trade or manufacture, such as byproducts from the meat packing industry. Less attention has been given to industrial uses of crop residues on farms, such as tobacco stalks, sorghum refuse, and cull vegetables. The fiber industries, however, though they long regarded the forests as inexhaustible sources of fiber, have recently been forced to practice better methods of forest cutting and conservation, to look to the farms for fibrous byproducts, and to develop other economies of operation.

Consequently, American pulp and paper manufacturers have sponsored research into the use of farm by products as raw materials for the fiber industries. This research has been done chiefly at the Northern Regional Research Laboratory (Peoria, Illinois) on wheat, rye, oats, barley, and rice straw, though some work has been done on corn and cotton stalks, sugar-cane bagasse, flax shives, hemp hurds, and various grasses.

Few farm byproducts are assembled in large quantities at any one point because economy in handling has forced the leaving of all possible residues at or near the farm where crops are grown. Local markets, such as grain elevators, canneries and creameries, may assemble sufficient quantity of corncobs, pea vines, or buttermilk for commercial use, but for the most pat the byproducts from Kentucky farms are left at the farm where mey are grown. Tohacco stalks, wheat straw, and sorghum bagasse are examples.

Again, those byproducts left at the farm may be left in the field, or they may be gathered at some point—straw at a stack, for example, and tobacco stalks at the tobacco barn. For hyproducts left in the field, commercial outlets must be profitable enough to pay for gathering as well as for delivery to a local or central market. For materials already assembled on the farm, commercial outlets must compete with other possible uses on the farm.

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In the light of these conditions, burley tobacco stalks, sorghum bagasse (the refuse left after sweet sorghum is ground and pressed for sirup), and hemp hurds were selected as representative Kentucky farm byproducts for fiber research at the Kentucky Agricultural Experiment Station. The study had to do with the general availability of these products, their value to the farmer for use on the farm, the cost of assembling them, their possible uses for cellulose and pulp for high-grade papers, and the byproducts which might be obtained from them during processing for cellulose.

THE SAMPLES

Samples of the materials were obtained in batches of 30 to 40 pounds each. The burley tobacco stalks and hemp hurds were thoroughly air-dry, but the sorghum bagasse, being freshly crushed, was dried for about 30 hours at not over 85° C. The tobacco stalks were broken in a sack with a hammer. Portions of the tobacco stalks, hemp hurds, and dried bagasse were first ground in a large Wiley sampling mill and then in a laboratory-model Wiley mill to pass a 60-mesh screen. The ground samples were stored in glass jars, and each was kept thoroughly mixed so as to be uniform throughout.

The samples were analyzed by standard methods for moisture, ash, alcohol-benzene solubility, lignin, and pentosans, and for crude cellulose by a method¹ worked out in this laboratory. This method has certain advantages over the Norman-Jenkins procedure,² in economy of time, amount of yield, and accuracy of determination of alpha-cellulose and ash in crude cellulose. The crude cellulose was analyzed by standard methods for alpha- and gamma-cellulose, ash, lignin, and pentosans. The results of these analyses are given in Tables 1 and 2.

LABORATORY METHOD OF CELLULOSE ISOLATION REAGENTS

Sodium hypochlorite, 5.25 percent, grocery grade, was analyzed for available chlorine and used on a basis of 4.0 percent available chlorine.

¹ "Cellulose Determination in Agricultural Residues: A Modification of the Norman-Jenkins Procedure," by E. B. Lewis and J. S. McHargue. Accepted for publication by the Journal of Industrial and Engineering Chemistry, Analytical Edition.

^{*}A. G. Norman and S. H. Jenkins, Biochem. Jour. 27: 816-831, 1933.

1948] TOBACCO STALKS, HEMP HURDS, SORGHUM BAGASSE

TAELE 1.—AVERAGES OF ANALYSES OF HEMP HURDS, SORCHUM BAGASSE, AND STALKS OF BURLEY TOBACCO, AS PERCENT OF THE MOISTURE-FREE MATERIALS

Components	Hemp hurds	Sorghum bagasse	Tobacco stalks
Lignin	21.9	11.8	13.0
Pentosans	24.2	27.4	20.5
Crude cellulose	54.6	51.2	43.5
Ash	1.2	2.8	8.9
Sum	101.9	93.2	85.9
Soluble in alcohol-benzene	4.3	13.6	12.2
Alpha cellulose	39.0	35.0	31.2
Jamma cellulose	8.3	4.8	6.1
Moisture in the air-dry material	6.2	6.0	6.3

TABLE 2.—AVERAGE ANALYSES OF THE CRUDE CELLULOSE FROM HEMP HURDS, Sorghum Bacasse, and Stalks of Burley Tobacco, as Percent of the Moisture.-Free Materials

Components	Hemp	Sorghum	Tobacco
	hurds	bagasse	stalks
Lignin Pentosans Alpha cellulose Ash Sum Ash	0.5 24.4 71.4 15.2 0.2 111.7	$ \begin{array}{r} 0.4 \\ 33.8 \\ 68.3 \\ 9.4 \\ 0.2 \\ \hline 112.1 \end{array} $	$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$

- Acid hypochlorite, 30 ml of sodium hypochlorite (4.0 percent available chlorine) and 20 ml of 20 percent sulfuric acid to 1 liter of distilled water.
- Sulfite-bicarbonate solution, 31.0 gm anhydrous sodium sulfite and 10.3 gm U.S.P. sodium bicarbonate per liter.
- Alkaline salt solution, 20 gm sodium sulfite, 11 gm sodium sulfate, 9 gm sodium chloride, 9 gm sodium bicarbonate, 250 ml 0.5 N sodium hydroxide, diluted to 1040 ml with distilled water.
- 20 percent sulfuric acid: 124 ml concentrated sulfuric acid and 904 ml distilled water.
- 5 percent sulfuric acid: 29 ml concentrated sulfuric acid and 978 ml distilled water.

1:20 ammonium hydroxide: 50 ml concentrated ammonium hydroxide diluted to 1 liter with distilled water.

Ethyl alcohol, 95 percent.

Commercial ethyl ether.

METHODS

A portion of ground sample, 0.59 to 0.61 gm, was weighed into a filtering tall-form 30-ml medium-porosity Pyrex crucible, which was then attached to a filter flask. In all washes the crucible was filled almost to the top, and stirred with a glass rod equipped with a rubber "policeman." The material was then allowed to settle for 3 to 4 minutes before being filtered, using suction.

The first wash, with boiling alkaline salt solution, was repeated until the filtrate was clear and colorless. Then the sample was washed with the series boiling water, cool acid hypochlorite, and boiling sulfite-bicarbonate solution. At this point the residue was usually a dark purple. The washing series was repeated until a white residue was obtained after the final sulfite-bicarbonate wash, indicating complete removal of the lignin.

Then the sample was washed once with boiling water, twice with cool 5 percent sulfuric acid, once with $cool (approximately <math>27^{\circ}$ C) and four times with boiling 1:20 ammonium hydroxide, once with alcohol, and once with ether. Suction was continued until no odor of alcohol or ether was noticed in the residue.

The crucible with residue was then placed in a weighing bottle and dried in a vacuum oven (about 58° C and 28'' Hg) to constant weight. Usually 2 hours drying was enough. It was then cooled in the drier and weighed. The residue (crude cellulose) was then removed mechanically, quantitatively, and the weighing bottle was weighed with the crucible. The difference in weights is the weight of the crude cellulose. For accuracy, it is important that no weighings be made until this time because the crucible itself loses weight from the alkaline washes.

LABORATORY PULPING AND MAKING FIBER PRODUCTS

Batch pulping in the laboratory was done at first by the laboratory method of cellulose determination. This is an isolation procedure, except that 15-gram samples were used in No. 4 Büchner-Coors filter funnels with No. 10 preshrunk canvas filter circles. The funnels were filled on each extraction, giving a solid-liquid weight ratio of 1:42. Later it was found that pulping could be done by using 45 grams of sample and sodium hypochlorite with 5 percent available chlorine in the same proportion as sodium hypochlorite with 4.0 percent chlorine, or sodium hypochlorite of less available chlorine in sufficient quantity to give the equivalent chlorine concentration, maintaining the volume ratio of hypochlorite to acid at 3:2.

In preliminary experiments, pulp sheets of various thicknesses and areas from cardboards to tissues were made up from hemp hurd, sorghum bagasse, and tobacco-stalk pulps prepared as above. The sheets were formed first by beating definite weights of the pulps in the Waring Blender with 600 ml of water for about 2 minutes and making up to definite volume, shaking thoroughly, pouring out definite portions, diluting each portion to about 4 liters, and filtering through Büchner-Coors filter funnels using No. 10 canvas tilter circles prepared as before. For the sorghum bagasse pulp, half of the batch was beaten about 2 minutes and the other half about 5 minutes in the Waring Blender. The sheets were dried overnight at about 80° C between drying screens, steamed to bring up the moisture content, and pressed out between metal plates using both a laboratory hand press and a hydraulic press.

Later experiments in making pulp sheets were carried out the same as before except that after the pulp suspensions of definite volumes and pulp weights were made up, the suspensions were beaten for 10 minutes in portions of approximately 600 ml each in the Waring Blender. In these experiments the sheets were not only made from the individual pulps but from pulp mixtures of 50 percent tobacco stalks and 50 percent sorghum bagasse, and from 50 percent hemp hurds and 50 percent sorghum bagasse. Also, sheets were made from hemp hurd pulp as above using 3 percent casein filler.

QUALITY OF THE PAPERS MADE

As experiments progressed, steady improvement was made in evenness of formation, smoothness of surface, tensile strength, and whiteness of the pulp sheets. A summary of results obtained in making paper from each of the three raw materials, under different treatment in beating and in combinations of materials, is given in *Table 3*. Three sheets were made from each material when beaten 2 minutes and 10 minutes. Weight, thickness, and tensile strength of

	Size of test strip		Breaking
	Weight	Thickness	strength
	gm	in.	kg
Hemp hurds	-	1 1	_
Beaten 2 minutes	0.2413	0.0067	6.00
Beaten 10 minutes	0.2759	0.0065	12.95
Sorghum bagasse			
Half beaten 2 minutes and half		1 1	
beaten 5 minutes	0.2081	0.0073	4.03
Beaten 16 minutes	0.2397	0.0069	9.72
Tobacco stajks			
Beaten 2 minutes	0.2124	0.0076	0.60
Beaten 10 minutes	0.2954	0.0064	12.38
½ hemp, ½ sorghum			
Beaten 10 minutes	0.2300	0.0070	8.47
½ tobacco, ½ sorghum			
Beaten 10 minutes)	0.2479	0.0068	9.33
Hemp with 3 percent casein			
Beaten 10 minutes	0.2103	0.0033	10.27
Bond note paper	0.2270	0.0040	5.70
Typewriter bond	0.2456	0.0044	5.45

TABLE 3.—TENSILE STRENGTH OF TISSUES MADE FROM HEMP HURDS, SORCHUM BACASSE, AND TOBACCO STALKS (Average of 3 Samples)

the three sheets were averaged for comparisons between materials and methods,

Samples left in the blender for 10 minutes made much heavier, stronger, but thinner paper than those beaten for only 2 minutes. From each of the farm byproducts used, the longer beating made distinctly better paper because of better pulp hydration. Prolonged beating, beyond 10 minutes, was of no advantage, however, because it produced too high a rate of hydration and seemingly also too much cutting of the fibers.

Among the samples beaten for 10 minutes the hemp hurds and tobacco stalks gave somewhat stronger paper than the sorghum bagasse. Mixing materials gave no improvement over either material used alone.

Paper made from sorghum bagasse seemed superior to that made from the other two products. It was more flexible, had a softer, more velvety texture, and appeared to withstand folding better than the other papers. These different characteristics of sorghum paper probably are due to the longer fibers of the sorghum bagasse. No accurate data on lengths of fibers were taken, but the sorghum fibers generally were $1\frac{1}{2}$ to 2 times the length of those of tobacco stalks or hemp hurds.

Nearly all of the paper stood up well under handwriting and typewriting tests. All of those made from pulp beaten 10 minutes were stronger, but most of them also were thicker and slightly heavier, than the high-grade commercial typewriter bond papers with which they were compared.

In general, the experiments in cellulose analysis, pulping, and pulp sheet making indicate that hemp hurds, sorghum bagasse, and burley tobacco stalks are good sources of raw material for the fiber industries. From the high-strength pulps which can be developed there is the possibility on the one hand of making heavy wrapping papers, cardboards, and boxboards which, in general, are the lower priced fiber products, and on the other hand of making high-grade drawing paper, stationery, and typewriting bonds, which are in the high-price brackets. Because of economic considerations discussed in the following section, the research dealt mainly with the higher priced products.

AVAILABILITY OF THE BYPRODUCT MATERIALS

Industrial uses of farm byproducts depend upon (1) discovery of useful products into which they can be made; (2) development of economical processes by which such products can be made commercially; and (3) assembling sufficient quantities of the raw materials at costs such that competition of other products can be met. With respect to sorghum bagasse, tobacco stalks, and hemp hurds, the first and second of these considerations have been answered, in part, in the foregoing pages. Feasibility of commercial production must of course await trials on a commercial scale. The third problem involves costs of transporting and handling the material, and consideration of its alternate value for use on the farm as fertilizer, fuel, feed, bedding material for livestock, and so on.

TOBACCO STALKS

The value of tobacco stalks for fertilizer has long been stressed, and many farmers save this valuable crop residue for the nitrogen, **BULLETIN NO. 515**

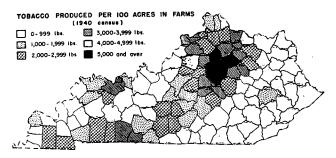


Fig. 1.—Heaviest tobacco production in Kentucky is concentrated in the Bluegrass area and in certain counties in western Kentucky. One-fourth of the counties grow half of the tobacco produced.

potassium, and phosphorus compounds it contains. When kept under shelter until spread in pastures or on land where some crop is growing, the stalks can be valued at \$10 to \$12 a ton.¹ It is estimated, however, that in commercial pulping of tobacco stalks for paper and various other fiber products, up to 40 percent of the pentosans as furfural, 90 percent of the lignin, and 85 percent or more of the total fertilizer materials could be recovered. If it is assumed that Kentucky will produce annually about 300 million pounds of burley tobacco and about 75 million pounds of dark tobacco, it may be anticipated that 100,000 tons of air-dry tobacco stalks will be produced each year.²

Tobacco is produced in all parts of Kentucky, but its production is heaviest in the Bluegrass area (Fig. 1) and fairly heavy in some of the western counties. One-fourth of the counties of the state produce half of the tobacco, and individual land owners in several instances produce 100 or more acres of tobacco under their own supervision. This high concentration of production is favorable to assembling tobacco stalks for industrial use.

Value and Use of Tobacco Stalks in Farm Practice, Ky. Agr. Exp. Sta. Bul. 486, by M. E. Weeks and P. E. Karraker, page 5.

³ This assumes somewhat more than prewar production of burley, but about prewar average production of dark tobacco. The yield of stalks is estimated at the rate of 600 pounds of stalks to each 1,000 pounds of burley leaf and 300 pounds of stalks to each 1,000 pounds of dark tobacco leaf.

Loose tobacco stalks are bulky and hard to handle, but a fairly inexpensive press could be used on farms for tying the stalks into bundles or bales.

Expanded production of burley tobacco in recent years and the consequent shortage of barn space has resulted in the use of warehouses and other buildings at market centers for curing. When stripping is done at these points, stalks of course are assembled there. If this practice continues it will offer a source of tobacco stalks for commercial use. Currently there is talk of a stripping and grading service to which farmers might bring their tobacco on the stalk. At this central point it would be stripped, graded, and tied into hands. leaving the stalks. If such a service develops it also would offer a source of tobacco stalks.

SORGHUM BAGASSE

Sorghum bagasse is wet and heavy as it leaves the sorghum mill, and it decomposes slowly when spread in the field, causing considerable trouble by clogging tillage implements. Consequently, as it has little value as fertilizer, it is usually dried and burned for disposal. In pulping sorghum bagasse about 37 percent of the pentosans and 90 to 95 percent of the lignin could probably be recovered as byproducts.

While much less sorghum bagasse than tobacco stalks is produced in Kentucky, the bagasse is strictly a byproduct of little or no value at present and is a nuisance to dispose of. Most of the bagasse now produced in Kentucky is in areas where tobacco production is light (*Fig. 2*) and farm incomes are relatively low. Even small income from its sale might therefore be especially appreciated.

The average production of sweet sorghum in Kentucky during recent years has been 12,000 to 13,000 acres but the latest figures for each county are those given by the Census of Agriculture for the year 1939, which total 11,253 acres in the state.

Because sorghum bagasse is distinctly a byproduct, no accurate information is available as to the total amount produced. As a result of field tests made by the Agronomy Department of the Kentucky Agricultural Experiment Station, and other observations, however, it is thought that roughly 3 tons of dry bagasse per acre is a fair esti-

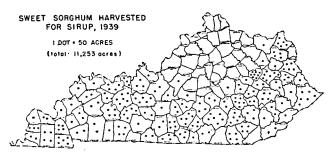


Fig. 2.—Much of the sorghum in Kentucky is grown in the areas of rougher topography where farm production for home use is given special emphasis.

mate for the state as a whole.¹ This would suggest an annual production of around 35,000 tons. As only $\frac{1}{2}$ to 1 acre commonly is grown per farm, assembling of any appreciable amount of sorghum bagasse would involve gathering it from a large number of farms.

As freshly crushed bagasse, if closely packed, is subject to deterioration by fermentation, it would need to be dried before baling for storage or shipping. Experiments made at this Station indicate that sorghum bagasse spread about a foot thick over any area under shelter will dry out satisfactorily in a week or so if turned every two or three days. It could probably be dried as effectively in favorable weather by spreading it outside over well-drained ground.

For baling dried bagasse an inexpensive hand press of some sort could be developed to bale the few tons available per farm. The material could be taken in conveniently sized bales by wagon or truck to local assembly points for shipment to manufacturing centers. Moisture tests would have to be made in order to determine the market value of the material, and perhaps quality grades could be established.

¹ Sorghum grown for fodder produced about 2.5 tons per acre of airdry fodder in 1939. Sorghum for silage produced about 8.5 tons, green weight. Green sorghum cane contains about 60 percent water and 40 percent oven-dry matter. If air-dry material contains 20 percent moisture, the silage cane would have produced about 4.2 tons per acre of air-dry material. Production of sirup averages about 60 gallons, of 12 pounds each, per acre. Tests suggest that about 1/20 of the original green weight of cane is retained in the sirup. On this basis, green sweet sorghum yields about 7 tons, green weight, per acre or nearly 3.5 tons of air-dry bagasse.

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HEMP HURDS

A byproduct of a crop which, in the past, was important on Kentucky farms is the hurds or woody part remaining after the hemp fiber has been removed. When hemp growing was in its heyday in Kentucky, retting and breaking was done on the farms, and hurds were commonly burned. As burley tobacco came to compete with hemp for the use of land and labor in Central Kentucky, and sisal and other fibers came upon the market, hemp growing almost disappeared from Kentucky farms.

Shortage of fiber during World War II, however, stimulated hemp production and one or two large mills were set up to break hemp and prepare the fiber for market. At such plants hurds can be used as fuel for power. In Kentucky, however, small portable hempbreaking machines were preferred, and these left the hurds on the farms. With the immediate emergency over and good prices for burley tobacco in effect, growing hemp is again relatively unimportant. The development of harvesting and breaking machinery, more adapted to corn-belt conditions than to Kentucky, suggests that hemp will not again be grown extensively in Kentucky.

The use of hemp hurds, however, for manufacturing processes would offer an outlet to the few Kentucky farmers who continue to grow hemp and to those in Wisconsin and the corn-belt who now find hemp production profitable.

In Kentucky, hurds would need to be gathered at the farm, after breaking, for shipment to points of manufacture. Their lack of adaptability for baling complicates this problem. In areas where large breaking mills are used, such as in Wisconsin, the hurds could be diverted from use as fuel to other uses. If the hurds were pulped it is estimated that about 45 percent of the pentosans could be recovered as furfural and 90 to 95 percent of the lignin could be recovered.

Because of the woody stem and branching growth of seed hemp, making the plants bulky and awkward to handle, it would not seem feasible to use these plants as a source of cellulose for commercial use.

Scarcity of material and difficulty of assembly suggest little promise of an important use of hemp hurds in Kentucky unless they are needed to supplement other materials in order to make a marketable product.

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CONCLUSIONS

Paper of good quality can be made from tobacco stalks, hemp hurds, or sorghum bagasse. This shows promise for successful industrial use of these products.

Tobacco stalks are available in large quantities in Kentucky, but they have a high fertilizer value on farms, and commercial use of them must offer even higher values to be attractive to farmers. As much of their fertilizer value could be recovered in making paper, their use for this purpose would seem feasible. Tobacco stalks are regarded as having the best promise of the three materials, for manufacture of paper and byproducts of processing.

Hurds from hemp are largely a waste product, which technically offers a good source of cellulose for paper manufacture, with promise of byproduct furfural. Their use in Kentucky, however, is unpromising because of the small quantity available and the difficulty of handling them.

Sorghum bagasse is produced in small quantities in widely scattered areas of Kentucky, totaling only about a third of the total tonnage of tobacco stalks produced in the state. It is strictly a farm waste material which must be given immediate care in drying if it is to have commercial value. Technically it is a good source of cellulose and shows promise for commercial use in the manufacture of highgrade paper.

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