PHYSICAL PROPERTIES OF HEMICELLULOSE EXTRACT AS A FEED INGREDIENT

by

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INTRODUCTION

Hemicellulose extract is a by-product of an industrial process for producing hardboard. It is the concentrated soluble product obtained from the steam treatment of wood at elevated temperatures and pressures without the use of acids, alkalis, or salts. It is produced at plant locations in Laurel, Mississippi, and Ukiah, California, by Masonite Corporation. Although the process is essentially the same in both places, the hemicellulose extract produced in California has a much lower viscosity.

Hemicellulose extract is sold as a liquid under the trade name of Masonex^{R}, and a spray dried product is also sold under the trade name of Dried Masonex^{R}. This research dealt only with the liquid product.

Hemicellulose extract is used as a source of energy in animal feeds in much the same manner as cane molasses.

The purpose of this research was to study the physical properties of liquid hemicellulose extract. There were indications that the viscosity of the product from Mississippi was too high at times for it to be handled easily with conventional equipment in a feed mill, and the problem of reducing or controlling the viscosity was of primary importance.

Enzymes and surfactants were used in an attempt to reduce the viscosity of hemicellulose extract from Mississippi. The ease with which a liquid feed ingredient is handled in the feed mill depends upon the temperature, because the viscosity of liquids increases with a decrease in temperature. For this reason the viscosity of hemicellulose extract as a function of temperature was determined, and a comparison was made with the characteristics of the viscosity of cane molasses.

Hemicellulose extract from Mississippi was blended with cane molasses and with hemicellulose extract from California. The effect of dilution with water on the viscosity and degrees Brix was examined using the material from Mississippi.

Hemicellulose extract is normally neutralized before it is placed on the market. It was therefore desirable to determine a relationship between the amount of neutralizer and the pH. A relationship between the pH and the viscosity was determined using several neutralizers. The pH and the viscosity were determined at various times after neutralization to detect any drift.

A ration containing hemicellulose extract was pelleted to investigate any differences in pellet durability and energy requirement compared with a ration containing cane molasses.

REVIEW OF LITERATURE

Cross (3) reported on the manufacture of hemicellulose extract. A schematic diagram of this process is shown in Figure 1. Wood chips are charged into a 26 cu. ft. steel digester, cooked by direct high-pressure steam up to 1,000 psi for one to two minutes, then suddenly released and blown to a collector cyclone. This hydrolysis takes place

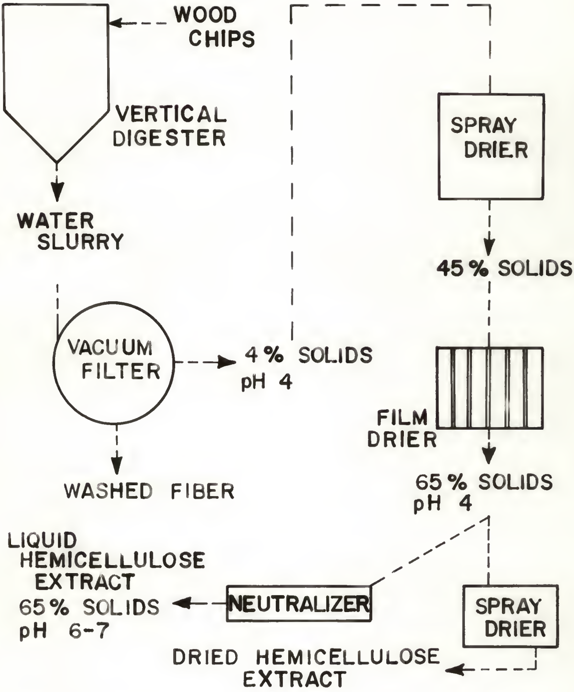

FIG. 1. PROCESS FLOW FOR PRODUCTION OF HEMICELLULOSE EXTRACT.

without the use of acids, alkalis, salts, or other accelerating agents. The coarse fiber in a water slurry is further refined with disk grinders. The milled pulp is washed in a triple-stage countercurrent vacuum-drum washer and is further processed to hardboard. The wash water (500,000 gal./day) from the first stage contains about 4% to 5% dissolved and colloidal solids, including simple sugars and polysaccharides. (This wash liquor will be referred to later as 4% hemicellulose extract.) The wash liquor from the first-stage washer is then taken to a battery of spray driers in which the concentration approaches 45% solids. (The output from the spray driers will be referred to as either 45% or 48% hemicellulose extract.) The spray drier is fed by the exhaust gases from a gas-turbine power generating plant. Some polymer formation and thermal degradation result from the hot gas temperature and lead to a darkening of the solution (3). A vertical-tube, falling-film evaporator, which is steam heated, concentrates the material from the spray drier to 65% solids. (This material will be referred to later as 65% hemicellulose extract.) The material coming from the evaporator (50,000 gal./day) is slightly acidic with a pH of 3 to 4. It is normally neutralized to a pH of 6 to 6.5 before it is ready for shipment to the customer.

Cross also reported that hemicellulose extract contains both pentose and hexose sugars and has a total carbohydrate content of 55% or more. It is a brown, free-flowing liquid with a density of 10.8 lb./gal. and resembles conventional cane molasses in total carbohydrate and feeding value.

The manufacturer (2) reports a typical chemical analysis of hemicellulose extract according to the location of manufacture as follows:

Item	Mississippi Liquid	Mississippi Dry	California Liquid
Protein (Min.), %	0.5	0.5	0.5
Fat (Min.), %	0.5	0.5	0.5
Fiber (Max.), %	0.5	1.0	0.5
Ash (Max.), %	5.0	3.0	5.0
Phosphorous (Min.), %	0.05	0.07	0.05
Calcium (Max.), %	2.0	0.5	0.5
Moisture (Max.), %	35	4.0	35
N.F.E. (Min.), %	55	80	55
Brix (Min.)	65°	--	65°
pH	5.5-6.5	--	5.5-6.5

The chemical nature of hemicellulose extract depends on the wood species used. On mild hydrolysis hardwoods tend to yield higher levels of five carbon sugars, and softwoods tend to yield higher levels of six carbon sugars. Typical product analysis shows nearly equal distribution of these plus soluble polysaccharide precursors of simple sugars. Concerning the liquid product, simple sugars will make up about 10% of the total weight, and pentosans and hexosans will make up about 45%. On the dried product these figures will be 18% and 62%, respectively. Bomb calorimeter values for hemicellulose extract correspond closely to the values for simple pure sugars (2).

The following has been accepted as a temporary official definition by the Louisiana Department of Agriculture:

> Hemicellulose extract is the concentrated soluble material obtained from the steam treatment of wood at elevated temperatures and pressures without the use of acids, alkalis, or salts. It contains both pentose and hexose sugars and has a total carbohydrate content of not less than 55%. Its Brix is determined by double dilution and is not less than 65 degrees.

Sherwood and Kelman (5) reported that a Brookfield Model LVF "Synchro-Lectric" spindle viscometer with an ultra-low adapter and a size 300 Fenske-Ostwald pipette viscometer gave similar results in estimating the activity of a cellulase and a polygalacturonase at various dilutions. They also reported that the spindle viscometer was equal or superior to the pipette in accuracy and reproducibility of results and that the simplicity of operation of the spindle viscometer resulted in a saving of time per sample.

MATERIALS AND METHODS

Equipment and Samples

A Brookfield Model LVT "Synchro-Lectric" spindle viscometer (Brookfield Engineering Laboratories, Stoughton, Massachusetts) was used to determine the viscosity of 45% and 65% hemicellulose extract. A combination of eight spindle speeds and four spindles made possible the measurement of viscosity over a wide range. The Brookfield viscometer utilizes a beryllium copper spring as the connecting element between the motor and the spindle. When the spindle is suspended in the test fluid and rotated at a constant speed, the spring becomes wound in proportion to the torque necessary to overcome resistance to the spindle motion in the test fluid. The degree of winding of the spring is indicated by displacement of a pointer over a rotating calibrated dial. Dial readings are easily convertible to a unit of viscosity measurement, the centipoise (abbreviated cp.). The viscosity of water at 20.2°C. is 1.0 centipoise.

A size 100 capillary or pipette viscometer of the Ostwald type designed by Fenske and Cannon was used to compare the viscosity of 4% or diluted 48% hemicellulose extract after various enzyme treatments. A stop watch was used to measure the time for the test fluid to pass between two points on the tube. A corresponding time was determined using distilled water. A dimensionless time index was then calculated by dividing the time for the test fluid under study by the time for distilled water to pass between the two points on the capillary viscometer; this quotient is commonly called relative viscosity. Two or more enzyme treatments were then compared by examining the corresponding time indices. The sample with the lowest numerical time index would have the lowest viscosity.

All pH determinations were made on a Beckman Zeromatic pH meter. A special electrode with a ground glass sleeve was used on the 45% and 65% hemicellulose extract to help maintain good electrical contact between the electrode and the fluid under study.

The specific gravity determinations were made using a hydrometer with range 1.2 to 1.4. In some cases, the specific gravity was determined using a volumetric flask and a torsion balance. The degrees Brix was determined using a hydrometer which covered the range of values in question.

The hemicellulose extract used in the laboratory was shipped from the plant locations in five-gallon buckets. The material in these buckets was stirred thoroughly before samples were drawn for testing. All material was stored in sealed containers to prevent unnecessary loss of water.

Two surfactants were tested. Kem-Wet^R is a product of Chemical Industries, Des Moines, Iowa, and contains monoglycerides, diglycerides, and polyoxyethylene sorbitan. The manufacturer recommends that it be used at a level of 4 to 6 ounces per ton. Sirlene^R is a product of Dow Chemical Corporation and contains propylene glycol as its major ingredient.

All of the enzymes used were in a dry form, except for liquid Pectinol^R 59-L. A hemicellulase and a pectinase of unknown origin were used in preliminary studies. Gumase HP-150, Pectinol 59-L, Pectinol^R 41-P Concentrate, Cellulase 36, Rhozyme^R CL, Lipase A, and Lipase B enzymes were obtained for further study from Rohm and Haas Company, Philadelphia, Pennsylvania. Lipase B is an enzyme product which replaced Lipase A.

Lipase B and Rhozyme CL are standardized on the basis of their lipolytic activity, although they possess some proteolytic activity. The manúfacturer reports that both are stable under highly acidic conditions. Lipase B and Rhozyme CL contain the same enzyme systems, but Lipase B is a food grade enzyme and Rhozyme CL is its crude counterpart.

Pectinol 59-L and Pectinol 41-P Concentrate are food grade pectic enzymes used for hydrolyzing and solubilizing pectinous materials. They also possess some diastatic activity under the proper conditions. Pectinol 59-L exhibits a somewhat greater heat stability and a greater tolerance to lower pH values.

Cellulase 36 is a food grade enzyme preparation. Although it is standardized on the basis of its cellulolytic activity, it also possesses some diastatic and pectinolytic activity. Gumase HP-150 is a food grade enzyme which is standardized on the basis of its ability to reduce the viscosity of a hexosan (locust bean gum) solution. However, it is characterized by its ability to hydrolyze a class of polysaccharides commonly called vegetable gums or mucilages. Generally, these substances are hexose and/or pentose polymers.

Temperature and Blending Studies

Effect of Temperature on Viscosity of 65% Hemicellulose Extract and Cane Molasses. Samples of 65% hemicellulose extract from Mississippi and California were used in these studies. The sample of hemicellulose extract from California was not neutralized, but the sample from Mississippi had been neutralized in the plant with hydrated lime. A sample of cane molasses from a commercial source was also used, and its viscosity was higher than that of many samples of cane molasses. This sample is referred to as Sample IV in Appendix B.

The three samples referred to in the previous paragraph were warmed to approximately 60°C. in a forced air oven. The viscosity was measured with the Brookfield viscometer (spindle no. 4) as the samples were cooled. To suppress changes in viscosity caused by evaporation of water from the samples, the number of viscosity determinations was limited to no more than eight for each sample and the samples were sealed between tests.

Effect of Water Dilution on the Viscosity of 65% Hemicellulose Extract from Mississippi. A 350 gm. sample of 65% hemicellulose extract from Mississippi which had been neutralized in the plant with hydrated lime was utilized in this experiment. Distilled water was mixed with the sample in increments of 5 ml. or 10 ml. at room temperature until a total dilution of 60 ml. (17.1%) of water in the 350 gm. sample had been achieved. The viscosity was measured at 30°C. with the Brookfield viscometer (spindle no. 4) after each addition of water. The specific gravity was determined volumetrically before the addition of water and after the total of 60 ml. of water had been added.

Effect of Temperature on Viscosity of 65% Hemicellulose Extract from Mississippi Diluted with Water. Samples of 65%

hemicellulose extract from Mississippi and cane molasses from a commercial source were utilized. The samples of hemicellulose extract had been neutralized in the plant with hydrated lime.

Four 400 gm. samples of hemicellulose extract were prepared and identified as samples A, B, C, and D. Samples A and C were diluted with 30 ml. (7.5%) of distilled water at room temperature. Samples B and D were not diluted with water. A sample of the cane molasses was prepared for a reference and identified as sample E. The specific gravity of all samples was determined volumetrically.

To examine the effect of the temperature on the viscosity, samples A, B, and E were warmed to approximately 60°C., and the viscosity was measured with the Brookfield viscometer (spindle no. 4) as the samples were cooled. Samples C and D were cooled to approximately 0°C., and the viscosity was measured as the samples were warmed to approximately 60°C.

Viscosity of Blends of 65% Hemicellulose Extract from Mississippi and California. Eleven samples were prepared at room temperature with the hemicellulose extract from California in the blend varying from 0% to 100% by increments of 10%. The hemicellulose extract from California was neutralized in the plant with sodium hydroxide. The hemicellulose extract from Mississippi was neutralized in the laboratory with pelleted laboratory grade sodium hydroxide. The viscosity of the samples was measured with the Brookfield viscometer at temperatures of 2-3°C. (spindle no. 4) and 56-58°C. (spindle no. 2).

Effect of Temperature on Viscosity of Blends of 65% Hemicellulose Extract from Mississippi and Cane Molasses. Samples of 65% hemicellulose extract from Mississippi and cane molasses were blended according to percentage by weight at room temperature. Samples of cane molasses and hemicellulose extract from the same source as in the first experiment were utilized. The effect of temperature on viscosity was investigated following the same procedure as in the first experiment.

Surfactants

Kem-Wet. The manufacturer recommends that 6 ounces of Kem-Wet be pre-mixed with 1 quart of warm water and added to 1 ton of molasses.

A solution was made of 5.3 parts warm water plus 1.0 part liquid Kem-Wet by volume. The manufacturer's recommendations corresponded to 0.5 ml. of this solution in a 400 gm. sample of hemicellulose extract. A solution of Sirlene was made in the same manner as the Kem-Wet solution in order that dilution effects would be equalized.

Unneutralized samples of 65% hemicellulose extract from Mississippi were treated with 0.5 ml. of Kem-Wet solution, Sirlene solution, or water per 400 gm. of sample. Cane molasses from a commercial source was treated in the same manner. Four samples were used in each treatment.

Sirlene. Four 350 gm. samples of unneutralized 65% hemicellulose extract from Mississippi were prepared and identified as samples A, B, C, and D. Samples B and D had been previously treated with a lipase enzyme at a level of a cost equivalent of $1/ton. Samples A and B were treated with Sirlene to cumulative levels of 1%, 2%, and 4% by weight of the original sample weight; samples C and D were treated in the same manner with water. The viscosity at 30°C. was measured with the Brookfield viscometer (spindle no. 3) after each treatment.

Enzymatic Treatment of 4% and 48% Hemicellulose Extract

Before attempting to reduce the viscosity of 65% hemicellulose extract with enzymes, work was conducted using unneutralized 4% hemicellulose extract coming from the vacuum filters in the plant operation in Mississippi. Tests were also conducted on 48% hemicellulose extract from the spray drier. The 48% samples were diluted with distilled water to six times the original volume for testing in the capillary viscometer. A test was also conducted on 45% hemicellulose extract to examine the effect of neutralization with lime on enzyme activity. These viscosity determinations were made with the Brookfield viscometer (spindle no. 2). No attempt was made to enzymatically treat hemicellulose extract from California.

All viscosity measurements were conducted in a water bath held at 29.5°C.

The enzymes used in these tests were extracted in distilled water at room temperature for 24 hours at a level of 1 gm. of enzyme in 25 ml. of water. The solution was filtered to remove the insoluble material. The filtrate was then placed in a refrigerator until it was used in the tests. This procedure was altered in tests examining the effect of the concentration of the enzyme.

This preliminary study was conducted to help select several enzymes which might be effective in reducing the viscosity of 65% hemicellulose extract from Mississippi. For this reason, duplication was not attempted in most tests.

Enzymes reported here are those which indicated more than marginal activity. Preliminary work by the author and his associates showed that alpha- and beta-amylase were not effective in reducing the viscosity of 4% hemicellulose extract. Heating this material to a temperature of about 95°C. produced a substrate which would allow good growth with *Saccharomyces cerevisiae*.

Effect of Enzymes on Viscosity. A series of tests were conducted on both 4% and 48% hemicellulose extract. The enzymes which were tested included a hemicellulase, a pectinase, Gumase HP-150, Pectinol 59-L, Pectinol 41-P Concentrate, Lipase A, and Cellulase 36. The procedure and the treatment of the samples are included with the results of the tests in Tables 3, 4, 5, and 6.

Effect of Temperature on Enzyme Activity. Pectinase was used in this test. The samples used in this test were 65% hemicellulose extract which had been diluted to approximately 10% solids before treatment with the enzyme solution. The procedure and the treatment of the samples are included with the results of the test in Table 8.

Effect of Time on Enzyme Activity. A hemicellulase enzyme was used in this test. A 25 ml. sample of 4% hemicellulose extract was treated with 1 ml. of hemicellulase enzyme solution. Another 25 ml. sample was treated with 1 ml. of distilled water. The time index was determined on these samples at various time intervals after treatment. When the viscosity was not being determined, the samples were held at 43°C. The procedure and the treatment of the samples are included with the results in Table 9.

Effect of Concentration of the Enzyme. The effect of the concentration of the enzyme solution was examined using Lipase A, Lipase B, and Rhozyme CL on 4% hemicellulose extract. The concentration of Lipase A and Cellulase 36 was also examined using 48% hemicellulose extract. The procedure and the treatment of the samples are included with the results in Tables 10, 11, 12, and 13.

Effect of Neutralization with Lime on Lipase B Activity. Six 400 gm. samples of unneutralized 45% hemicellulose extract from Mississippi were prepared. Three samples were neutralized to different pH levels with industrial grade lime, and the pH and viscosity were determined on all six samples. Viscosity measurements were made at 30°C. with the Brookfield viscometer (spindle no. 2).

All samples were treated with 0.015 gm. of Lipase B. The samples were held at 44°C., and the pH and viscosity at 30°C. were determined at 3 and 66 hours after enzyme treatment.

Enzymatic Treatment of 65% Hemicellulose Extract

The enzymatic treatment of 4% hemicellulose extract from Mississippi resulted in a reduction of viscosity. Utilizing the information gained in these tests, Lipase B, Rhozyme CL, Gumase HP-150, and Cellulase 36 were evaluated on unneutralized 65% hemicellulose extract from Mississippi. (These enzymes were evaluated at treatment levels corresponding to a cost equivalent of \$1, \$2, and \$4 per ton.)

Comparative Effect of Enzymes. Four 300 gm. samples of unneutralized 65% hemicellulose extract were prepared. The enzymes were added in the dry form at room temperature at levels indicated in Table 15. The viscosity at 29.5°C. was measured with the Brookfield viscometer (spindle no. 3) after the samples had been held at 49°C. for 11 hours and at 29.5°C. for 2 hours.

In an additional test, five 400 gm. samples of unneutralized 65% hemicellulose extract were prepared. One sample was not treated with an enzyme, and the enzymes were added to the other four samples at room temperature at levels shown in Table 16. The viscosity at 29.5°C. was measured with the Brookfield viscometer (spindle no. 4) after the samples had been held at 55°C. for 13 hours and at 29.5°C. for 1½ hours.

Effect of Concentration of Enzyme. The effect of Lipase B, Rhozyme CL, Gumase HP-150, and Cellulase 36 in reducing the viscosity of unneutralized 65% hemicellulose extract was examined at three levels.

Lipase B. Eight 400 gm. samples of unneutralized 65% hemicellulose extract were heated to 50°C. and treated with Lipase B at three levels as shown in Table 17 with a duplicate sample at each level. Two samples were not treated. The samples were held at 52°C. for 11 hours and at 29.5°C. for 1½ hours. Then the viscosity at 29.5°C. was measured with the Brookfield viscometer (spindle no. 4).

In a second test, four 350 gm. samples were treated at room temperature with Lipase B at three levels as shown in Table 17. One sample was not treated with an enzyme. These samples were held at 51°C. for 11 hours and at 29.5°C. for 8 hours. The viscosity at 29.5°C. was measured with the Brookfield viscometer (spindle no. 4). Then the samples were cooled to 1°C., and the viscosity determined again.

A third test was conducted in a manner similar to the previous test except that the treated samples were held at 51°C. for 11 hours and at 29.5°C. for 3 hours before the viscosity measurements were made at 29.5°C.

Rhozyme CL. Four 350 gm. samples of unneutralized 65% hemicellulose extract were treated at room temperature with Rhozyme CL at three levels. One sample was not treated and used as a control. The samples were held at 51°C. for 11 hours and at 29.5°C. for 5 hours before the viscosity measurements were made at 29.5°C. with the Brookfield viscometer (spindle no. 4). Then the samples were cooled to 1°C., and the viscosity determined again.

A second test was conducted in a manner similar to the first test except that the treated samples were held at 51°C. for 11 hours and at 29.5°C. for 3 hours before the viscosity measurements were made at 29.5°C.

Gumase HP-150. Four 350 gm. samples of unneutralized 65% hemicellulose extract were treated at room temperature with Gumase HP-150 at three levels. One sample was not treated and used as a control. The samples were held at 51°C. for 11 hours and at 29.5°C. for 2 hours before the viscosity measurements were made at 29.5°C. with the Brookfield viscometer (spindle no. 4).

Cellulase 36. Four 350 gm. samples of unneutralized 65% hemicellulose extract were treated at room temperature with Cellulase 36 at three levels. One sample was not treated. The samples were held at 51°C. for 11 hours and at 29.5°C. for 5 hours before the viscosity measurements were made at 29.5°C. with the Brookfield viscometer (spindle no. 4).

Neutralization Studies

45% Hemicellulose Extract. Fourteen 250 gm. samples of unneutralized 45% hemicellulose extract from Mississippi were prepared, and the pH of each sample was determined. The viscosities were determined with the Brookfield viscometer (spindle no. 2). Seven samples were neutralized to different pH levels with known amounts of lime (CaO) which had been obtained from the plant in Mississippi. The other seven samples were neutralized to different pH levels with known amounts of pelleted sodium hydroxide which had been crushed with a mortar and pestle. The pH and viscosity were determined again about 24 hours after neutralization. To detect any drift, the pH of the samples was again determined about one month and three months after neutralization.

65% Hemicellulose Extract. Sixteen 400 gm. samples of unneutralized 65% hemicellulose extract from Mississippi were prepared. The viscosities were determined with the Brookfield viscometer (spindle no. 4). The specific gravity was determined on four additional samples with a hydrometer. A sodium hydroxide solution with the same specific gravity was prepared using the same hydrometer, and the percent sodium hydroxide by weight was found from tables (4). Hydrated lime was prepared by adding water to lime (CaO) until the mixture contained approximately 45% solids after evaporation in a forced air oven at 105°C. for 24 hours.

Fourteen of the sixteen samples were neutralized at room temperature with four levels of sodium hydroxide solution and three levels of the hydrated lime with duplicate samples at each treatment level. The remaining two samples were not neutralized. The pH and the viscosity were determined the following day and at one-week intervals for four weeks.

Eighteen 400 gm. samples of 65% hemicellulose extract from California were prepared and neutralized using the procedure described for material from Mississippi. The viscosities were determined using the Brookfield viscometer (spindle no. 3). Sixteen of the eighteen samples were neutralized with four levels of sodium hydroxide solution and four levels of hydrated lime with duplicate samples at each treatment level. The remaining two samples were not neutralized. The pH and viscosity were determined the following day and at one-week intervals for four weeks.

Effect of Neutralization on Degrees Brix. The specific gravity was determined with a hydrometer on three samples of unneutralized 65% hemicellulose extract from California. A sodium hydroxide solution with the same specific gravity was prepared using the same hydrometer, and the percent sodium hydroxide by weight in the solution was found from standard tables (4).

Eighteen 400 gm. samples of unneutralized 65% hemicellulose extract from California were prepared. Sixteen of these samples were neutralized with the sodium hydroxide solution at eight different levels with duplicate samples at each treatment level. The two remaining samples were not neutralized. The pH and viscosity (Brookfield viscometer, spindle no. 3) were determined two days later. The specific gravity and degrees Brix were also determined on the combined samples from each treatment level.

Twenty-two 400 gm. samples of unneutralized 65% hemicellulose extract from Mississippi were prepared and neutralized using the procedure described for material from California. The samples were neutralized at ten different levels with duplicate samples at each treatment. Two samples were not neutralized. The pH and viscosity (Brookfield viscometer, spindle no. 4) were determined two days later. The specific gravity and degrees Brix were also determined with hydrometers on the combined samples from each treatment level. The degrees Brix by double dilution was also determined by diluting portions of the combined samples with an equal weight of water. A Brix hydrometer was then used to determine the degrees Brix of the diluted sample. The reading obtained should be one-half the degrees Brix of the original sample.

Pelleting Studies

The purpose of this experiment was to compare the energy required to produce pellets from a beef cattle ration containing 10% cane molasses (formula AH-65-C) or 10% liquid 65% hemicellulose extract (formula AH-65-D) and to examine the pellet durability of the pellets produced from both rations. Five commercial ureas were also examined with regard to energy requirements and pellet durability in feed containing cane molasses and hemicellulose extract. The formulation of the two basic rations is given in Table 38. The two rations differed only in whether cane molasses or hemicellulose extract was used and in the type of urea used. The modulus of uniformity (1), fineness modulus, and percent nitrogen of each type of urea are given in Table 39.

All of the dry ingredients except the rolled milo were mixed in a 500-lb. horizontal batch mixer and transferred to the continuous mixing system where the rolled milo was blended with the original mix of dry ingredients. The cane molasses or hemicellulose extract was then added in a horizontal, high-speed molasses blender. The feed was then pelleted.

The pelleting was done with a 25 h.p. California Master model pellet mill using a 3/16-in. by 1 3/4-in. die, and the pellets were cooled in a California vertical pellet cooler, size 2-B. Energy requirement was measured with a recording watt-hour meter. In each pelleting test, the pellet mill was started with the pellet cooler on continuous discharge, and the pellets were sacked off immediately. As soon as stable operating conditions were obtained, the cooler was placed on automatic discharge and the recording watt-hour meter started. When the cooler discharge is set on automatic, it discharges a quantity of pellets only when the cooler becomes full. The pellets discharged from the cooler were passed over a 4-mesh scalping screen. The whole pellets were sacked off and weighed. The fines from the scalper were returned to the pellet mill. Samples of cold pellets were collected to determine the pellet durability index (6). At the end of a test the watt-hour meter reading was recorded and divided by the amount of pellets in tons produced under stable operating conditions to give the energy requirement in watt-hours per ton. At least a ton of pellets was produced in each test, and in some tests, as many as five tons of pellets were produced.

RESULTS AND DISCUSSION

Temperature and Blending Studies

Effect of Temperature on Viscosity of Hemicellulose Extract and Cane Molasses. Figure 2 shows the effect of temperature on the viscosity of cane molasses and on hemicellulose extract from Mississippi and California. The values plotted in Figure 2 are tabulated in Table 25 in Appendix A. The pH, specific gravity, and degrees Brix of the samples are tabulated in Table 26 in Appendix A.

Figure 2 shows the difference in viscosity of the hemicellulose extract from Mississippi and California. Although all three samples were free flowing at higher temperatures, the viscosity of the hemicellulose extract from Mississippi increased much more rapidly than that of the cane molasses as the temperature was decreased. These results represent single samples; however, the trends illustrated in Figure 2 were found to hold true in general. Note that the sample of hemicellulose extract from California was unneutralized, because other work reported in this thesis shows that neutralization may cause some increase in viscosity. (A sample of neutralized material was not available at the time this experiment was performed.)

Effect of Water Dilution on the Viscosity of 65% Hemicellulose Extract from Mississippi. Figure 3 shows the effect of dilution on the viscosity of hemicellulose extract at 30°C. The values plotted in Figure 3 are tabulated in Table 27 together with values of specific gravity and the cumulative percent reduction in viscosity after each dilution.

Figure 3 shows that the viscosity of hemicellulose extract from Mississippi can be reduced significantly by dilution with water. The reduction in viscosity was greater for a given amount of water for the beginning increments of dilution; when less than 6% water was added, the viscosity had been reduced to less than 45% of the original viscosity.

Effect of Temperature on the Viscosity of Hemicellulose Extract from Mississippi Diluted with Water. Figure 4 shows the effect of the temperature on viscosity. The values plotted in Figure 4 are tabulated in Table 28 of Appendix A.

The plot in Figure 4 is semi-logarithmic. While all four hemicellulose samples exhibited only a slight curve, the cane molasses plotted a sharper curve and of a different shape. These curves again emphasize that the rate of viscosity increase at lower temperatures is less for cane molasses than for hemicellulose extract from Mississippi. Figure 4 also indicates that dilution decreased the viscosity of hemicellulose extract

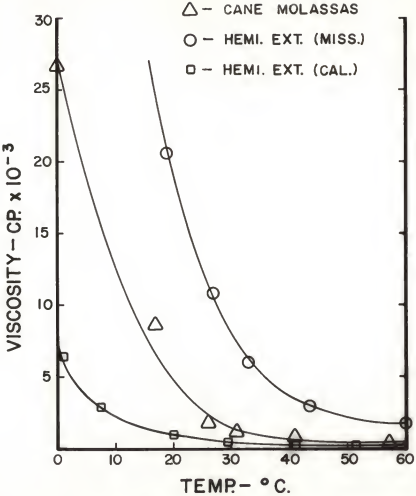

FIG. 2. EFFECT OF TEMPERATURE ON VISCOSITY OF HEMICELLULOSE EXTRACT AND CANE MOLASSAS.

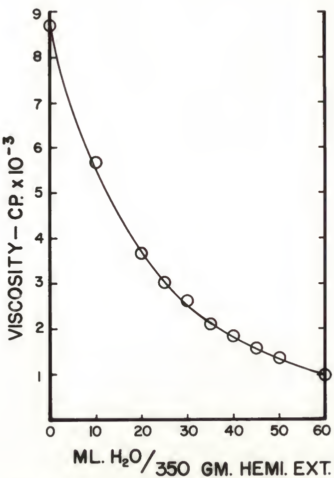

FIG. 3. EFFECT OF WATER DILUTION ON THE VISCOSITY OF HEMICELLULOSE EXTRACT (MISS.) AT 30 °C.

from Mississippi much more at lower temperatures than at higher temperatures.

Viscosity of Blends of Hemicellulose Extract from Mississippi and California. Figure 5 shows the effect on the viscosity of hemicellulose extract from Mississippi by blending with hemicellulose extract from California. The plot is semi-logarithmic, and it shows the effect at both 2-3°C. and 56-58°C. The data plotted a straight line at the lower temperature, but the curve at the upper temperature is slightly concave downward. This indicates a greater reduction in the viscosity at lower temperatures. The values plotted in Figure 5 are tabulated in Table 30 in Appendix A.

The dotted lines in Figure 5 represent the limits on the viscosity of cane molasses, 200 to 800 centipoises at 57.2°C., imposed by a feed manufacturer.* With these particular samples a blend consisting of about 20% hemicellulose extract from California would fall below this upper limit of 800 centipoises.

Effect of Temperature on Viscosity of Blends of Hemicellulose Extract from Mississippi and Cane Molasses. Figure 6 shows the effect of temperature on the viscosity of blends of cane molasses and hemicellulose extract from Mississippi. The values plotted in Figure 6 and the results of 5% and 10% blends are tabulated in Tables 31 and 32 of Appendix A.

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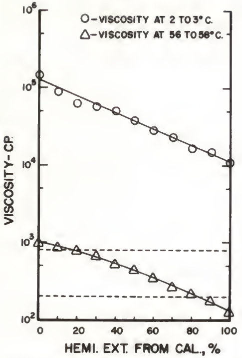

FIG. 5. VISCOSITY OF BLENDS OF HEMICELLULOSE EXTRACT FROM MISS. AND CAL.

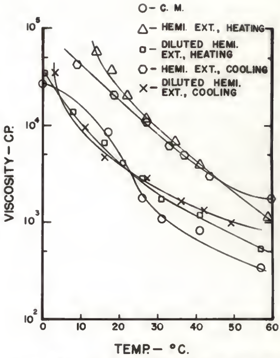

FIG. 4. EFFECT OF TEMPERATURE ON VISCOSITY OF HEMICELLULOSE EXTRACT (MISS.) DILUTED WITH 7.5 % WATER.

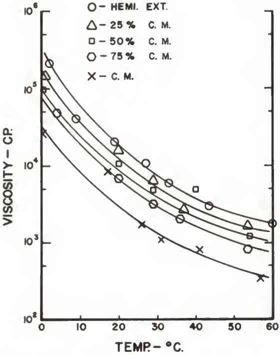

FIG. 6. EFFECT OF TEMPERATURE ON VISCOSITY OF BLENDS OF HEMICELLULOSE EXTRACT (MISS.) AND CANE MOLASSAS.

The results indicate that the viscosity of the blends decreased as the percent cane molasses in the blend was increased. However, the viscosity of the hemicellulose extract decreased more for a given percent cane molasses in the lower range of temperature than in the upper range of temperature.

Surfactants

Kem-Wet. The effects of Kem-Wet, Sirlene, and water on the viscosity at 30°C. of hemicellulose extract from Mississippi are shown in Table 1. Each result is the average of four tests.

The results shown in Table 1 indicate that Kem-Wet and Sirlene were not effective enough in reducing the viscosity to be generally useful, at least under the existing laboratory conditions and the treatment levels which were used.

Sirlene. The cumulative percent reduction in viscosity effected with water and with Sirlene on 65% hemicellulose extract from Mississippi is tabulated in Table 2. The values used in preparing Table 2 are given in Table 33 in Appendix A.

Sirlene was more effective, on the average, than water in reducing the viscosity at the 1% and 2% levels, but at the 4% level dilution with water reduced the viscosity more than the Sirlene. This trend was evident for both the enzymatically and non-enzymatically treated samples.

In all cases, water and Sirlene produced a greater reduction in the viscosity of the enzymatically treated samples.

Table 1. Effect of Kem-Wet, Sirlene, and water on the viscosity of 65% hemicellulose extract from Mississippi and cane molasses.

Treatment	Viscosity (centipoises) at 30 °C. Hemicellulose Extract: Average	Range	Cane Molasses: Average	Range
Kem-Wet	990	980-1,010	657	628-722
Sirlene	1,033	1,020-1,060	641	634-650
Water	1,028	1,020-1,040	653	630-682

Table 2. Percent reduction in viscosity of 65% hemicellulose extract treated with Sirlene and water.

Sample	Cumulative Percent Reduction in Viscosity					
	1% Sirlene	1% Water	2% Sirlene	2% Water	4% Sirlene	4% Water
A	6.9	--	14.4	--	19.9	--
B*	21.2	--	29.4	--	34.8	--
C	--	8.8	--	16.7	--	42.0
D*	--	13.8	--	16.8	--	46.5

*Enzymatically treated with a lipase enzyme prior to utilization in this experiment.

Enzymatic Treatment of 4% and 48% Hemicellulose Extract

Effect of Enzymes on Viscosity. The results of the tests comparing the effect of various enzymes on the viscosity of 4% hemicellulose extract are shown in Tables 3, 4, 5, and 6. The results in Table 3 indicate that the hemicellulase produced a

Table 3. Effect of a hemicellulase and a pectinase in reducing the viscosity of 4% hemicellulose extract.

Sample and Treatment	Relative Viscosity*
25 ml. of 4% hemicellulose extract plus 1 ml. of enzyme solution or distilled water held at 50°C. for 8 hours.	
Test A	
Hemicellulase Solution	1.164
Pectinase Solution	1.212
Distilled Water	1.368
Test B	
Hemicellulase Solution	1.190
Pectinase Solution	1.258
Distilled Water	1.347
25 ml. of 4% hemicellulose extract plus 1 ml. of enzyme solution or distilled water held at 43°C. for 23 hours.	
Hemicellulase Solution	1.164
Pectinase Solution	1.225
Distilled Water	1.356

*See page 6.

greater reduction in the viscosity than pectinase. All enzyme treatments were effective in reducing the viscosity when they were compared with the distilled water treatment.

The results in Table 4 indicate that the Gumase HP-150 was more effective in reducing the viscosity than the Pectinol 59-L and the Pectinol 41-P Concentrate. Pectinol 41-P Concentrate was the least effective.

Table 4. Effect of Gumase HP-150, Pectinol 59-L, and Pectinol 41-P Concentrate in reducing the viscosity of 4% hemicellulose extract.

Sample and Treatment	Relative Viscosity
25 ml. of 4% hemicellulose extract plus 1 ml. of enzyme solution or distilled water held at 43°C. for 3 hours.	
Gumase HP-150 Solution	1.231
Pectinol 59-L	1.260
Pectinol 41-P Concentrate Solution	1.330
Distilled Water	1.347

The results in Table 5 indicate that Gumase HP-150 is superior to the pectinase, the hemicellulase, and the Pectinol 59-L. The results of the second test indicate that Pectinol 41-P Concentrate is better than Pectinol 59-L. These results do not agree with those in Table 4.

The results in Table 5 indicate that Pectinol 59-L may be more effective than the pectinase and the hemicellulase in a

Table 5. Effect of a hemicellulase, a pectinase, Gumase HP-150, Pectinol 59-L, and Pectinol 41-P Concentrate in reducing the viscosity of 48% hemicellulose extract.

Sample and Treatment	Relative Viscosity
25 ml. of 48% hemicellulose extract plus 1 ml. of enzyme solution or distilled water held at 50°C. for 5 hours. Diluted to 150 ml. for viscosity determination.	
Hemicellulase Solution	1.957
Pectinase Solution	1.972
Gumase HP-150 Solution	1.950
Pectinol 59-L	2.029
Distilled Water	2.303
25 ml. of 48% hemicellulose extract plus 1 ml. of enzyme solution or distilled water held at 50°C. for 4 hours. Diluted to 150 ml. for viscosity determination.	
Hemicellulase Solution	1.824
Pectinase Solution	1.857
Gumase HP-150 Solution	1.813
Pectinol 59-L	1.922
Pectinol 41-P Concentrate Solution	1.790
Distilled Water	2.024
25 ml. of 48% hemicellulose extract plus 1 ml. of enzyme solution or distilled water held at 50°C. for 21½ hours. Diluted to 150 ml. for viscosity determination.	
Hemicellulase Solution	1.536
Pectinase Solution	1.554
Gumase HP-150 Solution	1.482
Pectinol 59-L	1.519
Distilled Water	1.886

long reaction period. However, Gumase HP-150 was superior to both of these enzymes.

The results in Tables 6 and 7 show that the Lipase A was considerably better than the hemicellulase, Gumase HP-150, and Pectinol 59-L. There is also an indication that Cellulase 36 produces a greater reduction in viscosity than the Gumase HP-150.

The data in Table 8 indicate that a pectinase enzyme produced a greater reduction in viscosity at 38°C. than at 30°C. or 22°C.

Effect of Time on Enzyme Activity. The results in Table 9 indicate that a hemicellulase enzyme produces a marked reduction in the viscosity of 4% hemicellulose extract within 4 hours after treatment. The rate of reduction in viscosity decreases after 4 hours.

Effect of Concentration of the Enzyme. The data in Table 10 show that a concentration of 0.250 gm. Lipase B per 25 ml. of enzyme solution produced almost the same reduction in viscosity as 1.000 gm. Lipase B per 25 ml. of enzyme solution.

The results in Table 11 show that the reduction in viscosity increased as the concentration of Rhozyme CL increased to 1.000 gm. per 25 ml. of enzyme solution. This was quite different from the effect of the concentration of Lipase B.

The results in Table 12 indicate that Lipase A reacted in much the same manner as Lipase B on both 4% and 48% hemicellulose extract.

Table 6. Effect of Lipase A, Gumase HP-150, Pectinol 59-L, and a hemicellulase in reducing the viscosity of 48% hemicellulose extract.

Sample and Treatment	Relative Viscosity
25 ml. of 48% hemicellulose extract plus 2 ml. of enzyme solution or distilled water held at 49°C. for 24 hours. Diluted to 150 ml. for viscosity determination.	
Hemicellulase Solution	1.642
Gumase HP-150 Solution	1.606
Lipase A Solution	1.527
Pectinol 59-L	1.703
Distilled Water	1.936

Table 7. Effect of Lipase A, Gumase HP-150, and Cellulase 36 in reducing the viscosity of 48% hemicellulose extract.

Sample and Treatment	Relative Viscosity
25 ml. of 48% hemicellulose extract plus 2 ml. of enzyme solution or distilled water held at 49°C. for 24 hours. Diluted to 150 ml. for viscosity determination.	
Gumase HP-150 Solution	1.600
Lipase A Solution	1.455
Cellulase 36 Solution	1.575
Distilled Water	1.850

Table 8. Effect of temperature on the activity of a pectinase enzyme in reducing the viscosity of 10% hemicellulose extract.

Sample and Treatment	Temperature (^{o}C.)	Relative Viscosity
25 ml. of 10% hemicellulose extract plus 1 ml. of a pectinase enzyme solution held at constant temperature for 19 hours.	22	1.706
	30	1.726
	38	1.598
25 ml. of 10% hemicellulose extract plus 1 ml. of distilled water held at constant temperature for 19 hours.	22	1.904
	30	1.910
	38	1.935

Table 9. Effect of time on the activity of a hemicellulase enzyme in reducing the viscosity of 4% hemicellulose extract.

Sample and Treatment	Time (Hours)	Relative Viscosity
25 ml. of 4% hemicellulose extract plus 1 ml. of hemicellulase enzyme solution held at 43^{o}C.	2.5	1.227
	4	1.217
	21	1.175
	46	1.149
25 ml. of 4% hemicellulose extract plus 1 ml. of distilled water held at 43^{o}C.	2.5	1.360
	4	1.362
	21	1.364
	46	1.369

Table 10. Effect of concentration of Lipase B enzyme solution in reducing the viscosity of 4% hemicellulose extract.

Sample and Treatment	Relative Viscosity
Test A	
25 ml. of 4% hemicellulose extract plus 1 ml. of Lipase B enzyme solution held at 53°C. for 29 hours.	
1.000 gm. Lipase B per 25 ml. of enzyme solution	1.144
0.500 gm. Lipase B per 25 ml. of enzyme solution	1.140
0.250 gm. Lipase B per 25 ml. of enzyme solution	1.161
0.125 gm. Lipase B per 25 ml. of enzyme solution	1.210
0.000 gm. Lipase B per 25 ml. of enzyme solution	1.343
Test B	
25 ml. of 4% hemicellulose extract plus 1 ml. of Lipase B enzyme solution held at 49°C. for 21 hours.	
1.000 gm. Lipase B per 25 ml. of enzyme solution	1.128
0.500 gm. Lipase B per 25 ml. of enzyme solution	1.226
0.250 gm. Lipase B per 25 ml. of enzyme solution	1.167
0.125 gm. Lipase B per 25 ml. of enzyme solution	1.203
0.000 gm. Lipase B per 25 ml. of enzyme solution	1.347

Table 11. Effect of concentration of Rhozyme CL enzyme solution in reducing the viscosity of 4% hemicellulose extract.

Sample and Treatment	Relative Viscosity
25 ml. of 4% hemicellulose extract plus 1 ml. of Rhozyme CL enzyme solution held at 53°C. for 25 hours.	
1.000 gm. Rhozyme CL per 25 ml. of enzyme solution	1.209
0.500 gm. Rhozyme CL per 25 ml. of enzyme solution	1.242
0.250 gm. Rhozyme CL per 25 ml. of enzyme solution	1.283
0.125 gm. Rhozyme CL per 25 ml. of enzyme solution	1.299
0.000 gm. Rhozyme CL per 25 ml. of enzyme solution	1.344

Table 12. Effect of concentration of Lipase A enzyme solution in reducing the viscosity of 4% and 48% hemicellulose extract.

Sample and Treatment	Relative Viscosity
25 ml. of 4% hemicellulose extract plus 1 ml. of Lipase A enzyme solution held at 49°C. for 21 hours.	
1.000 gm. Lipase A per 25 ml. of enzyme solution	1.140
0.500 gm. Lipase A per 25 ml. of enzyme solution	1.158
0.250 gm. Lipase A per 25 ml. of enzyme solution	1.198
0.000 gm. Lipase A per 25 ml. of enzyme solution	1.346
25 ml. of 48% hemicellulose extract plus 1 ml. of Lipase A enzyme solution held at 49°C. for 21 hours. Diluted to 150 ml. for viscosity determination.	
1.000 gm. Lipase A per 25 ml. of enzyme solution	1.619
0.500 gm. Lipase A per 25 ml. of enzyme solution	1.675
0.250 gm. Lipase A per 25 ml. of enzyme solution	1.774
0.000 gm. Lipase A per 25 ml. of enzyme solution	1.992

The results in Table 13 indicate that Cellulase 36 produced a greater reduction in the viscosity as the concentration increased.

Table 13. Effect of concentration of Cellulase 36 enzyme solution in reducing the viscosity of 48% hemicellulose extract.

Sample and Treatment	:Relative :Viscosity
25 ml. of 48% hemicellulose extract plus 1 ml. of Cellulase 36 enzyme solution held at 49°C. for 21½ hours. Diluted to 150 ml. for viscosity determination.	
1.000 gm. Cellulase 36 per 25 ml. of enzyme solution	1.670
0.500 gm. Cellulase 36 per 25 ml. of enzyme solution	1.778
0.250 gm. Cellulase 36 per 25 ml. of enzyme solution	1.886
0.125 gm. Cellulase 36 per 25 ml. of enzyme solution	1.953
0.000 gm. Cellulase 36 per 25 ml. of enzyme solution	2.014

Effect of Neutralization with Lime on Lipase B Activity. Table 14 shows the effect of enzyme treatment on neutralized and unneutralized 45% hemicellulose extract from Mississippi. The reduction in viscosity was much greater on the samples which were not neutralized. However, the neutralized samples suffered a greater reduction in viscosity as the pH value increased. All enzymes have a pH at which their activity is the highest, depending upon such factors as the substrate, temperature, moisture, and salts. The increase in the percent reduction of the viscosity of the neutralized samples was apparently due to a nearer optimum pH value.

Table 14. Effect of Lipase B on the viscosity of 45% hemicellulose extract neutralized with lime.

Neutralizer	pH	Viscosity (Centipoises) at 30°C. After 3 Hours at 44°C. Before Enzyme	After Enzyme	Percent Reduction	After 66 Hours at 44°C. Before Enzyme	After Enzyme	Percent Reduction
CaO	5.7	77.0	76.0	1.3	77.0	71.5	7.1
CaO	6.2	81.5	81.0	0.6	81.5	74.5	8.6
CaO	6.5	94.0	92.0	2.1	94.0	83.0	11.7
None	4.1	60.5	55.5	8.3	60.5	49.5	18.2
None	4.1	61.0	56.5	7.4	61.0	51.0	16.4
None	4.1	60.0	55.5	7.5	60.0	51.5	14.2

Table 14 also shows the effect of time on the reduction of viscosity after treatment with an enzyme. Less than one-half of the reduction in viscosity occurred within the first three hours.

Enzymatic Treatment of 65% Hemicellulose Extract

Comparative Effect of Enzymes. The results in Table 15 indicate that Lipase B was more effective than Rhozyme CL in reducing the viscosity of 65% hemicellulose extract. Furthermore, a combination of the two enzymes produced a 30.9% reduction in the viscosity compared to 32.9% with Lipase B and 25.5% with Rhozyme CL.

The results in Table 16 show that Lipase B and Rhozyme CL were much more effective than Gumase HP-150 and Cellulase 36 in reducing the viscosity of 65% hemicellulose extract when they were used at higher levels. The negative values given for the

Table 15. Effect of Lipase B, Rhozyme CL, and a combination of the two enzymes in reducing the viscosity of 65% hemicellulose extract.

Enzyme	Treatment: Dollars per Ton	Treatment: Gm. per 300 gm.	Viscosity (cp.)	Viscosity Reduction (%)
None	--	---	7,730	0.0
Lipase B	1.00	0.012	5,190	32.9
Rhozyme CL	1.00	0.150	5,760	25.5
Lipase B and Rhozyme CL	.50 .50	.006 .075	5,340	30.9

Table 16. Comparison of effect of enzymes in reducing the viscosity of 65% hemicellulose extract.

Enzyme	Treatment: Dollars per Ton	Treatment: Gm. per 400 gm.	Viscosity (cp.)	Viscosity Reduction (%)
None	--	--	10,540	0.0
Lipase B	2.00	.028	9,120	13.5
Rhozyme CL	2.00	.350	9,530	9.6
Gumase HP-150	2.00	.044	10,960	-4.0
Cellulase 36	2.00	.044	10,860	-3.0

percent reduction in viscosity with Gumase HP-150 and Cellulase 36 can probably be attributed to the evaporation of water from the samples when the enzymes were mixed with the hemicellulose extract. The percent reduction in viscosity achieved with Lipase B and Rhozyme CL was considerably less than that achieved in the preceding test. The treatment temperature in this test was 55°C., whereas in the preceding test it was 49°C. Although the difference in temperature is small, the higher temperature may have been high enough to partially inactivate the enzymes.

Effect of Concentration of Enzyme. Lipase B. The results of three tests examining the effect of the concentration of Lipase B on the viscosity of 65% hemicellulose extract are shown in Table 17. Each value shown for Test I is the average from two samples. The enzyme was added to the samples at 50°C. in Test I, and this may account for the lower percent reduction in viscosity compared to Test II and Test III. The enzyme was added to the samples at room temperature in Test II and Test III, and a 70.6% and 36.7% reduction in viscosity at the $4 per ton level was achieved.

The total treatment time was 19 hours for Test II and 14 hours for Test III. However, the samples in Test II were held at 29.5°C. for 5 hours longer than in Test III, and this may account for the difference in the percent reduction in viscosity. The initial viscosity of the samples used in Test II was higher than the viscosity of the samples used in Test III,

Table 17. Effect of the concentration of Lipase B in reducing the viscosity of 65% hemicellulose extract.

Enzyme	Treatment: Dollars per Ton	Treatment: Gm. per Sample	Viscosity (cp.)	Viscosity Reduction (%)
Test I.* 400 gm. samples				
None	--	--	11,560	0.0
Lipase B	1.00	.014	11,800	-2.1
Lipase B	2.00	.028	10,440	9.7
Lipase B	4.00	.056	9,760	16.3
Test II. 350 gm. samples				
None	--	--	10,740	0.0
Lipase B	1.00	.014	3,780	64.6
Lipase B	2.00	.028	3,580	66.7
Lipase B	4.00	.056	3,160	70.6
Test III. 350 gm. samples				
None	--	--	6,180	0.0
Lipase B	1.00	.014	4,830	21.8
Lipase B	2.00	.028	4,170	32.5
Lipase B	4.00	.056	3,910	36.7

*Each value shown for Test I is the average from two samples.

and there may have been more substrate available for the enzyme to act upon in Test II. The variation in effectiveness may also have been due to the source of the hemicellulose extract used in each test, because all tests were not made on samples from the same shipment from Mississippi.

In Test II, a $1 per ton level of Lipase B achieved almost the same effect as the $4 per ton level. However, in Test I and Test III the percent reduction in viscosity increased in a closer proportion to the amount of enzyme used.

Rhozyme CL. The results of two tests examining the effect of the concentration of Rhozyme CL on the viscosity of 65% hemicellulose extract are shown in Table 18. The percent reduction in the viscosity was nearly the same in both tests at the $1 per ton level. At the $2 and $4 per ton levels the percent reduction in the viscosity was considerably greater in Test I than it was in Test II. The reaction time was 4 hours longer at 29.5°C. in Test I, but the difference in percent reduction of viscosity was probably due to variation in the sources of the samples used in the two tests.

Gumase HP-150. The results in Table 19 show that Gumase HP-150 had very little effect in reducing the viscosity of 65% hemicellulose extract. The negative values for the percent reduction of viscosity were probably due to evaporation of water from the samples when they were mixed with the enzyme.

Cellulase 36. The results in Table 19 indicate that Cellulase 36 was certainly more effective than the Gumase HP-150,

Table 18. Effect of the concentration of Rhozyme CL in reducing the viscosity of 65% hemicellulose extract.

Enzyme	Treatment: Dollars per Ton	Treatment: Gm. per 350 gm.	Viscosity (cp.)	Viscosity Reduction (%)
Test I.				
None	--	--	6,190	0.0
Rhozyme CL	1.00	.175	4,920	20.5
Rhozyme CL	2.00	.350	3,820	38.3
Rhozyme CL	4.00	.700	3,030	51.1
Test II.				
None	--	--	6,010	0.0
Rhozyme CL	1.00	.175	4,940	17.8
Rhozyme CL	2.00	.350	4,680	22.1
Rhozyme CL	4.00	.700	3,950	34.3

Table 19. Effect of the concentration of Gumase HP-150 and Cellulase 36 in reducing the viscosity of 65% hemicellulose extract.

Enzyme	Treatment: Dollars per Ton	Treatment: Gm. per 350 gm.	Viscosity (cp.)	Viscosity Reduction (%)
None	--	--	5,440	0.0
Gumase HP-150	1.00	.022	5,700	-4.8
Gumase HP-150	2.00	.044	5,680	-4.4
Gumase HP-150	4.00	.088	5,130	5.7
None	--	--	5,660	0.0
Cellulase 36	1.00	.022	5,020	11.3
Cellulase 36	2.00	.044	4,730	16.4
Cellulase 36	4.00	.088	4,870	14.0

and it was as effective as Lipase B in Test I of Table 17. There was little variation in the percent reduction of viscosity.

Neutralization Studies

45% Hemicellulose Extract. Figure 7 shows the relationship between the pH and the amount of lime or sodium hydroxide added during neutralization. The average pH of the samples before neutralization was 4.2. Figure 7 shows that the relationship is not linear with either lime or sodium hydroxide. Above the 1% level of addition the sodium hydroxide produced a greater increase in pH when equal quantities of lime and sodium hydroxide were used.

Figure 8 shows the effect of pH on the viscosity of 45% hemicellulose extract using both lime and sodium hydroxide as a neutralizer. The viscosity of the samples neutralized with lime was greater at all pH values represented. This indicates an advantage in using sodium hydroxide as a neutralizer. The viscosity of the sample neutralized to pH 7.5 with sodium hydroxide was first thought to be an error; in later experiments, using 65% hemicellulose extract, a very similar result was obtained. This will be discussed later in more detail.

Figures 9 and 10 show the pH as a function of time for the samples neutralized with sodium hydroxide and lime, respectively. In both cases the samples with higher original pH values exhibited the greatest decrease in pH. It also appears

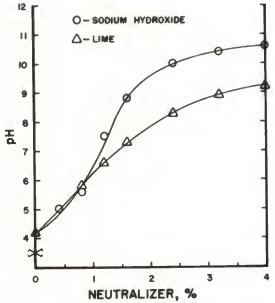

FIG. 7. RELATIONSHIP BETWEEN pH AND NEUTRALIZER ADDED TO 45% HEMICELLULOSE EXTRACT (MISS.).

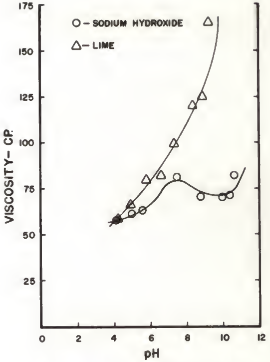

FIG. 8. EFFECT OF pH ON VISCOSITY OF 45% HEMICELLULOSE EXTRACT (MISS.).

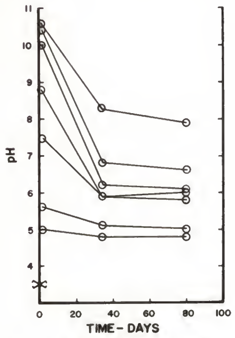

FIG. 9. RELATIONSHIP BETWEEN pH AND TIME SINCE NEUTRALIZATION OF 45% HEMICELLULOSE EXTRACT (MISS) WITH SODIUM HYDROXIDE.

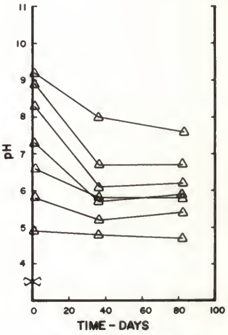

FIG. 10. RELATIONSHIP BETWEEN pH AND TIME SINCE NEUTRALIZATION OF 45% HEMICELLULOSE EXTRACT (MISS.) WITH LIME.

that the significant part of the decrease in pH had taken place within the first month. It is interesting to note that the average value of all samples neutralized with sodium hydroxide decreased from pH 8.3 to 6.0 while the average value of those neutralized with lime only decreased from pH 7.3 to 6.1. Hence, the pH values of the samples neutralized with sodium hydroxide appear to drift more than those neutralized with lime.

The values plotted in Figures 7, 8, 9, and 10 are tabulated in Tables 34 and 35 of Appendix A.

65% Hemicellulose Extract. The material from Mississippi had a specific gravity of 1.298, and the sodium hydroxide solution of equal specific gravity contained 27.2% sodium hydroxide by weight. The material from California had a specific gravity of 1.315, and the sodium hydroxide solution of equal specific gravity contained 29.6% sodium hydroxide by weight.

Figures 11 and 12 show the relationship between pH and viscosity at 30°C. of 65% hemicellulose extract from Mississippi and California, respectively, after neutralization with sodium hydroxide solution and hydrated lime. The values plotted in Figures 11 and 12 are tabulated in Tables 36 and 37, respectively, of Appendix A. Each value is the average of two samples. The viscosity increased as the pH increased when both types of material were neutralized with lime. When sodium hydroxide was used as a neutralizer, the factor of dilution definitely had an effect. Note particularly the increase in viscosity when the sodium hydroxide was added at the 1.0% level, corresponding to a pH of about 6.0. The viscosity at the 1.0% sodium hydroxide level of neutralization was greater than the original viscosity for the material from California, whereas it was not greater for the material from Mississippi. The effect of dilution will not be as great in the plant operation because industrial grade sodium hydroxide is about 50% sodium hydroxide by weight.

Figures 13 and 14 show the relationship between pH and the percent neutralizer added. In both cases the solution of sodium hydroxide yielded a higher pH than the lime when both were added at the same level. The values plotted in Figures 13 and 14 are given in Tables 36 and 37, respectively, of Appendix A.

Tables 20 and 21 show the pH and viscosity at one-week intervals after neutralization. Each value is the average from two samples. There was a tendency for the pH to decrease as time passed, especially at the higher pH values and for the samples neutralized with sodium hydroxide. There appeared to be no significant viscosity changes in the four-week period.

Effect of Neutralization on Degrees Brix. The hemicellulose extract from California had a specific gravity of 1.315, and the corresponding sodium hydroxide solution contained 29.6% sodium hydroxide by weight. The material from Mississippi had a specific gravity of 1.292, and the corresponding sodium hydroxide solution contained 26.7% sodium hydroxide by weight.

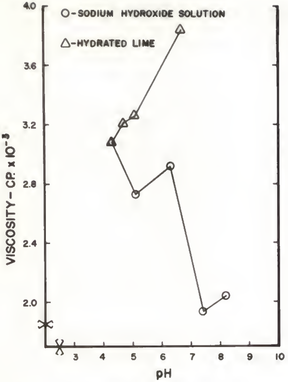

FIG. 11. RELATIONSHIP BETWEEN pH AND VISCOSITY OF 65% HEMICELLULOSE EXTRACT (MISS.).

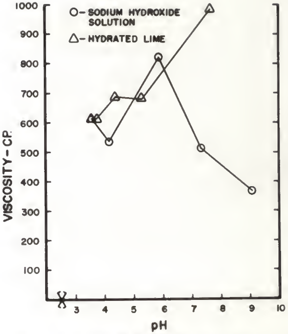

FIG. 12. RELATIONSHIP BETWEEN pH AND VISCOSITY OF 65% HEMICELLULOSE EXTRACT (CAL.).

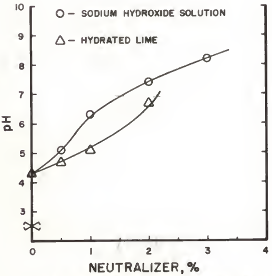

FIG. 13. RELATIONSHIP BETWEEN pH AND NEUTRALIZER ADDED TO 65 % HEMICELLULOSE EXTRACT (MISS.).

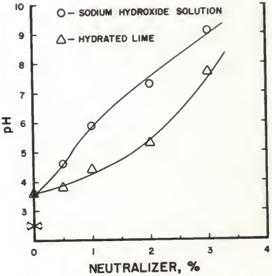

FIG. 14. RELATIONSHIP BETWEEN pH AND NEUTRALIZER ADDED TO 65% HEMICELLULOSE EXTRACT (CAL.).

Table 20. Viscosity at 30°C. in centipoises and pH of 65% hemicellulose extract from Mississippi at one-week intervals after neutralization with sodium hydroxide solution and hydrated lime.

% NaOH	% CaO	1 week		2 weeks		3 weeks		4 weeks	
		pH	Viscosity	pH	Viscosity	pH	Viscosity	pH	Viscosity
0.0	---	4.3	3,310	4.2	3,170	4.2	3,220	4.2	3,170
0.5	---	5.3	2,890	5.0	2,820	4.9	2,800	4.9	2,850
1.0	---	6.1	3,110	5.9	2,990	5.8	3,010	5.8	2,890
2.0	---	7.1	2,180	6.7	2,030	6.5	1,990	6.3	1,910
3.0	---	7.3	1,900	7.1	2,210	7.0	2,270	7.0	2,300
---	0.5	4.7	3,350	4.6	3,250	4.6	3,230	4.5	3,320
---	1.0	5.1	3,510	5.1	3,350	5.1	3,470	5.0	3,420
---	2.0	6.7	4,340	6.2	4,250	6.0	4,130	5.9	3,990

Table 21. Viscosity at 30°C. in centipoises and pH of 65% hemicellulose extract from California at one-week intervals after neutralization with sodium hydroxide solution and hydrated lime.

% NaOH	% CaO	1 week		2 weeks		3 weeks		4 weeks	
		pH	Viscosity	pH	Viscosity	pH	Viscosity	pH	Viscosity
0.0	---	3.6	645	3.6	645	3.5	645	3.5	660
0.5	---	4.7	580	4.5	590	4.4	565	4.4	585
1.0	---	5.8	860	5.6	845	5.5	785	5.4	780
2.0	---	6.8	625	6.6	595	6.4	550	6.2	570
3.0	---	8.4	370	8.3	345	8.1	345	8.2	330
---	0.5	3.7	650	3.9	670	3.9	655	3.9	705
---	1.0	4.4	700	4.4	725	4.5	715	4.5	775
---	2.0	5.2	705	5.3	685	5.3	710	5.3	710
---	3.0	7.3	995	7.0	1,000	6.6	980	6.2	1,020

Figure 15 shows the relationship between the amount of sodium hydroxide used and the pH of 65% hemicellulose extract from Mississippi and from California. The relationship is not linear, but there is a similarity between the two curves.

Figures 16 and 17 show the relationship between the pH and the viscosity. These tests contain more closely spaced points than those shown in Figures 11 and 12 and again show the significant viscosity increase at the 1.0% level of sodium hydroxide neutralization. The viscosity at this point was greater than the original viscosity in the material from California, whereas it was not greater for the material from Mississippi. The behavior of the 65% hemicellulose extract from Mississippi in the pH 7 to 9 range was extremely irregular with regard to viscosity.

The values plotted in Figures 15, 16, and 17 are tabulated in Tables 22 and 23. The viscosity and pH values are averages from two samples. The degrees Brix and specific gravity values are a single determination from a composite of the two samples.

Tables 22 and 23 give the degrees Brix of the samples at the different levels of neutralization. The material from California suffered a decrease in Brix of 1.2 degrees at the 4.0% sodium hydroxide level of neutralization. This corresponded to 54.1 ml. (13.5%) of solution in 400 gm. of hemicellulose extract. Referring to Figure 3, a similar dilution with water would have produced a much greater reduction in degrees Brix.

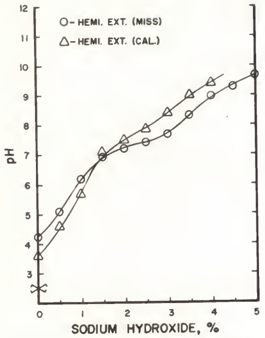

FIG. 15. RELATIONSHIP BETWEEN pH AND SODIUM HYDROXIDE ADDED TO 65% HEMICELLULOSE EXTRACT.

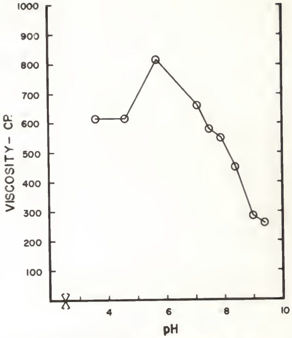

FIG. 16. EFFECT OF pH ON VISCOSITY OF 65 % HEMICELLULOSE EXTRACT (CAL.) NEUTRALIZED WITH SODIUM HYDROXIDE SOLUTION.

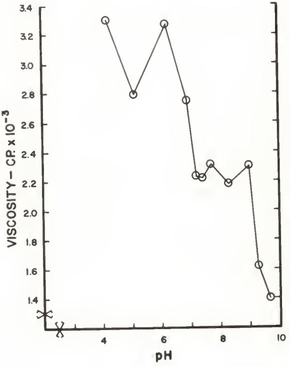

FIG. 17. EFFECT OF pH ON VISCOSITY OF 65% HEMICELLULOSE EXTRACT (MISS.) NEUTRALIZED WITH SODIUM HYDROXIDE SOLUTION.

Table 22. Effect of neutralization with sodium hydroxide solution on degrees Brix of 65% hemicellulose extract from California.

NaOH Percent	NaOH Ml.	Viscosity at 30°C. (centipoises)	pH	Specific Gravity	Degrees Brix
0.0	0.0	615	3.6	1.315	64.9
0.5	6.8	615	4.6	1.316	65.1
1.0	13.5	815	5.7	1.314	64.8
1.5	20.3	660	7.1	1.312	64.6
2.0	27.0	580	7.5	1.312	64.3
2.5	33.8	550	7.9	1.313	64.5
3.0	40.5	450	8.4	1.312	64.2
3.5	47.3	285	9.0	1.307	63.9
4.0	54.1	260	9.4	1.305	63.7

Table 23. Effect of neutralization with sodium hydroxide solution on degrees Brix of 65% hemicellulose extract from Mississippi.

NaOH Percent	NaOH Ml.	Viscosity (cp. at 30°C.)	pH	Specific Gravity	Degrees Brix	Degrees Brix by Double Dilution
0.0	0.0	3,300	4.2	1.292	60.7	30.3
0.5	7.5	2,790	5.1	1.291	60.8	30.3
1.0	15.0	3,270	6.2	1.290	60.7	29.9
1.5	22.5	2,750	6.9	1.291	60.7	30.3
2.0	30.0	2,230	7.2	1.291	60.6	30.2
2.5	37.5	2,220	7.4	1.291	60.7	30.4
3.0	45.0	2,310	7.7	1.292	60.8	30.2
3.5	52.5	2,180	8.3	1.289	60.3	30.3
4.0	60.0	2,300	9.0	1.288	60.1	30.2
4.5	67.5	1,620	9.3	1.287	60.1	30.1
5.0	75.0	1,400	9.7	1.286	59.8	29.9

The hemicellulose extract from Mississippi suffered a decrease in Brix of 0.6 degrees at the 4.0% sodium hydroxide level of neutralization, and 60.0 ml. of solution was used. The degrees Brix by double dilution was also determined on the samples from Mississippi. The values by double dilution agree very closely with the original values for degrees Brix.

Pelleting Studies

The results of the pelleting tests are given in Table 24. Each result is the average of only one pelleting test. However, the amount of pellets produced in each test was large enough to attach some significance to the single observation. Each pellet durability index shown is the average of three samples of cold pellets drawn from a single test run.

Table 24. Results of pelleting studies.

Type of Urea	Formula	KWH/Ton[1]	P.D.I.[2]
Feed Urea A	AH-65-C	21.70	9.38
	AH-65-D	21.20	9.64
Feed Urea B	AH-65-C	27.91	9.40
	AH-65-D	29.43	9.48
Feed Urea C	AH-65-C	19.90	9.50
	AH-65-D	17.35	9.22
Small Fertilizer Urea	AH-65-C	23.02	9.40
	AH-65-D	21.83	9.20
Large Fertilizer Urea	AH-65-C	24.24	9.54
	AH-65-D	24.93	9.52

[1]Kilowatt-hours per ton.
[2]Pellet durability index.

The only difficulty encountered during the pelleting operation was an occasional plugging of the pellet die. This was thought to be due to the additional stickiness of the ration caused by the combination of the urea and the moisture added in the conditioning chamber of the pellet mill. The plugging of the die could not be attributed to any one kind of urea. Temperature rises in the conditioning chamber of the pellet mill ranged from 26°C. to 34°C. Temperature rises in the pellet mill die ranged from 18°C. to 30°C.

Examination of the data presented in Table 24 showed an obvious difference in the energy requirements of the rations containing the different types of urea. A t-test of the differences of the pairs indicated no significant difference (0.01 level) in energy requirements or pellet durability index between the rations containing cane molasses and the rations containing hemicellulose extract.

CONCLUSIONS

Temperature and Blending Studies

The viscosity of 65% hemicellulose extract from Mississippi increased very rapidly as the temperature decreased, but the viscosity was not a problem at temperatures above approximately 30°C. The viscosity of 65% hemicellulose extract from Mississippi can be effectively reduced by blending it with cane molasses or 65% hemicellulose extract from California. The viscosity of 65% hemicellulose extract from Mississippi can be reduced drastically by dilution with less than 10% water.

Surface Acting Agents

Sirlene effectively reduced the viscosity of 65% hemicellulose extract from Mississippi when it was used at levels of 1% or 2% of the sample weight, and it was more effective on the material which had been treated with a lipase enzyme. Kem-Wet did not produce a marked reduction in the viscosity of cane molasses or 65% hemicellulose extract from Mississippi when it was used at the recommended treatment levels.

Enzymatic Treatment of 4% and 48% Hemicellulose Extract

The viscosity of 4% and 48% hemicellulose extract from Mississippi was reduced through treatment with enzymes, and a lipase enzyme produced the best results. Although most of the reduction in viscosity occurred within 4 hours, the enzymes continued to be active for much longer reaction times. Neutralization of 45% hemicellulose extract with lime inhibited the activity of a lipase enzyme, although the percent reduction in viscosity increased as the pH of the neutralized samples increased to 6.5.

Enzymatic Treatment of 65% Hemicellulose Extract

A food grade lipase enzyme and its crude counterpart were effective at economic treatment levels in reducing the viscosity of unneutralized 65% hemicellulose extract from Mississippi. The gumase and cellulase enzymes, which were effective on 4%

hemicellulose extract, were not as effective as the lipase enzymes in reducing the viscosity of 65% hemicellulose extract.

Neutralization Studies

Preliminary research on the neutralization of 45% hemicellulose extract showed that lime produced a greater increase in the viscosity than reagent grade sodium hydroxide. Furthermore, the sodium hydroxide produced a greater increase in the pH than the lime when equal levels were used. However, after a period of about three months, the pH of the samples neutralized with sodium hydroxide had decreased more than the pH of the samples neutralized with lime.

Neutralization of 65% hemicellulose extract with hydrated lime produced an increase in the viscosity. Neutralization with sodium hydroxide solution produced an increase in the viscosity at about pH 6, but at lower or higher pH values there was a decrease in the viscosity without a serious change in the specific gravity of the hemicellulose extract. There was a decrease in the pH of the samples neutralized to higher pH values after a period of four weeks. There were no apparent trends in the viscosity during this time.

Pelleting Studies

There was no significant difference (0.01 level) in the energy requirements or pellet durability of pellets produced from a beef cattle ration containing 10% of either 65%

hemicellulose extract from Mississippi or cane molasses. Five commercial grades of urea produced obvious differences in energy requirement in these tests.

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APPENDICES

APPENDIX A

Tabulated Values of Data Plotted in Graphs

Table 25. Effect of temperature on viscosity of hemicellulose extract and cane molasses.

Cane Molasses		Hemicellulose Extract from Mississippi		Hemicellulose Extract from California	
Temp. (°C.)	Viscosity (cp.)	Temp. (°C.)	Viscosity (cp.)	Temp. (°C.)	Viscosity (cp.)
57.0	340	60.0	1,750	51.5	140
41.0	810	43.5	2,970	41.0	274
31.0	1,090	33.0	6,000	29.5	502
26.0	1,780	27.0	10,800	20.0	980
17.0	8,600	19.0	20,600	8.0	2,920
0.0	26,700			1.0	6,350

Table 26. Description of the samples used in Table 25.

Test Material	pH	Specific Gravity[1]	Degrees Brix
Cane Molasses	5.22	1.414	80.0[2]
Mississippi Hemicellulose Extract	----	1.315	64.4[2]
California Hemicellulose Extract	3.83	-----	63.4[3]

[1]Specific gravity determined volumetrically. Each result is the average of four trials.

[2]Converted to degrees Brix from the value given for specific gravity.

[3]Determined with a Brix hydrometer.

Table 27. Effect of water dilution on the viscosity at 30°C. of hemicellulose extract from Mississippi.

Water Added (ml.)	Water Added (%)	Viscosity (Centipoises)	% Reduction in Viscosity	Specific Gravity[1]	Degrees Brix[2]
0	0	8,720	0	1.315	64.4
10	2.85	5,680	34.86		
20	5.71	3,680	57.80		
25	7.14	3,030	65.25		
30	8.57	2,640	69.72		
35	10.00	2,110	75.80		
40	11.43	1,840	78.90		
45	12.86	1,570	82.00		
50	14.29	1,350	84.52		
60	17.14	990	88.65	1.260	55.0

[1]Specific gravity determined using a volumetric flask and a torsion balance.
[2]Specific gravity converted to degrees Brix.

Table 28. Effect of temperature on viscosity of hemicellulose extract from Mississippi diluted with water.

Sample A		Sample B		Sample C		Sample D		Sample E	
Temp. (°C.)	Viscosity (cp.)	Temp. (°C.)	Viscosity (cp.)	Temp. (°C.)	Viscosity (cp.)	Temp. (°C.)	Viscosity (cp.)	Temp. (°C.)	Viscosity (cp.)
49.0	980	60.0	1,750	0.5	34,300	14.0	57,200	57.0	340
42.0	1,350	43.5	2,970	8.0	13,660	18.0	37,400	41.0	810
36.0	1,650	37.0	4,810	16.0	6,530	22.0	20,200	31.0	1,090
27.0	2,890	33.0	6,000	21.0	4,200	27.0	11,800	26.0	1,780
16.0	4,660	27.0	10,800	26.0	2,800	34.5	6,800	17.0	8,600
11.0	8,700	19.0	20,600	31.0	1,710	41.0	3,870	0.0	26,700
3.0	35,500	9.0	42,000	41.0	1,190	59.0	1,440		
				57.0	530				

Table 29. Description of the samples used in Table 28.

Sample and Treatment	Specific Gravity[1]	No. Trials	Degrees Brix[2]
A and C--Diluted Hemicellulose Extract	1.285	4	59.4
B and D--Hemicellulose Extract	1.315	4	64.4
E--Cane Molasses	1.414	2	80.0

[1]Specific gravity determined volumetrically.
[2]Specific gravity converted to degrees Brix.

Table 30. Viscosity of blends of hemicellulose extract from Mississippi and California.

Percent Hemicellulose Extract from California	Temp. (°C.)	Viscosity (cp.)	Temp. (°C.)	Viscosity (cp.)
0	2.0	114,600	58.0	995
10	2.0	88,800	57.0	865
20	2.0	63,800	58.0	795
30	2.0	57,900	58.0	665
40	2.0	51,400	57.5	530
50	2.0	37,800	57.5	450
60	3.0	28,700	57.5	355
70	3.0	23,250	57.5	270
80	3.0	16,750	56.5	220
90	3.0	15,050	57.0	175
100	3.0	11,100	56.5	125

Table 31. Effect of temperature on viscosity of blends of hemicellulose extract from Mississippi and cane molasses.

0% Cane Molasses		5% Cane Molasses		10% Cane Molasses		25% Cane Molasses	
Temp. (^{o}C.)	Viscosity (cp.)	Temp. (^{o}C.)	Viscosity (cp.)	Temp. (^{o}C.)	Viscosity (cp.)	Temp. (^{o}C.)	Viscosity (cp.)
60.0	1,750	54.5	2,060	54.5	1,950	53.5	1,660
43.5	2,970	36.5	4,430	37.0	3,470	37.0	2,720
33.0	6,000	29.0	9,500	29.0	8,730	29.0	6,490
27.0	10,800	20.0	25,150	20.0	21,100	20.0	16,680
19.0	20,600	4.0	190,800	4.0	167,400	1.0	151,600
9.0	42,000						
2.0	211,000						

Table 32. Effect of temperature on viscosity of blends of hemicellulose extract and cane molasses.

50% Cane Molasses		75% Cane Molasses		100% Cane Molasses	
Temp. (°C.)	Viscosity (cp.)	Temp. (°C.)	Viscosity (cp.)	Temp. (°C.)	Viscosity (cp.)
54.0	1,210	53.5	810	57.0	340
40.0	4,090	36.0	2,030	41.0	810
29.0	4,840	29.0	3,190	31.0	1,090
20.0	10,800	20.0	6,860	26.0	1,780
0.0	96,600	4.0	48,000	17.0	8,600
				0.0	26,700

Table 33. Effect of Sirlene and of water on the viscosity of unneutralized hemicellulose extract.

	Viscosity (Centipoises) at 30°C.						
Sample	No Treatment	1% Sirlene	1% Water	2% Sirlene	2% Water	4% Sirlene	4% Water
A	5,820	5,420	- -	4,980	- -	4,660	- -
B*	5,320	4,190	- -	3,760	- -	3,470	- -
C	5,340	- -	4,870	- -	4,450	- -	3,490
D*	3,150	- -	2,720	- -	2,620	- -	1,690

*Enzymatically treated with a lipase enzyme prior to utilization in this experiment.

Table 34. Effect of neutralization with sodium hydroxide on the pH and viscosity of 45% hemicellulose extract from Mississippi.

% NaOH Added	pH after Neutralization			Viscosity (centipoises) at 30°C.	% Increase in Viscosity
	1 day	34 days	80 days	1 day after Neutralization	
0.4	5.0	4.8	4.8	61.0	6.1
0.8	5.6	5.1	5.0	63.0	9.6
1.2	7.5	5.9	5.8	81.0	39.7
1.6	8.8	5.9	6.0	69.5	20.9
2.4	10.0	6.2	6.1	69.5	20.9
3.2	10.4	6.8	6.6	71.0	23.5
4.0	10.6	8.3	7.9	82.0	42.6

Table 35. Effect of neutralization with lime on the pH and viscosity of 45% hemicellulose extract from Mississippi.

% CaO Added	pH after Neutralization: 1 day	36 days	82 days	Viscosity (centipoises) at 30°C. 1 day after Neutralization	% Increase in Viscosity
0.4	4.9	4.8	4.7	66.0	12.8
0.8	5.8	5.2	5.4	79.5	34.7
1.2	6.6	5.8	5.8	82.0	37.8
1.6	7.3	5.7	5.9	99.0	67.8
2.4	8.3	6.1	6.2	119.5	102.5
3.2	8.9	6.7	6.7	124.5	109.2
4.0	9.2	8.0	7.6	164.5	176.5

Table 36. Viscosity and pH of 65% hemicellulose extract from Mississippi one day after neutralization with sodium hydroxide solution and hydrated lime.

% NaOH	Ml. NaOH Solution	% CaO	Gm. Hydrated CaO	pH	Viscosity at 30°C. (centipoises)
0.0	0.0	0.0	0.0	4.3	3,080
0.5	7.2	---	---	5.1	2,730
1.0	14.4	---	---	6.3	2,920
2.0	28.8	---	---	7.4	1,930
3.0	43.1	---	---	8.2	2,040
---	---	0.5	4.4	4.7	3,210
---	---	1.0	8.9	5.1	3,260
---	---	2.0	17.8	6.7	3,840

Table 37. Viscosity and pH of 65% hemicellulose extract from California one day after neutralization with sodium hydroxide solution and hydrated lime.

% NaOH	Ml. NaOH Solution	% CaO	Gm. Hydrated CaO	pH	Viscosity at 30°C. (centipoises)
0.0	0.0	0.0	0.0	3.6	615
0.5	6.8	---	---	4.6	535
1.0	13.5	---	---	5.9	820
2.0	27.0	---	---	7.3	515
3.0	40.5	---	---	9.1	370
---	---	0.5	4.4	3.8	615
---	---	1.0	8.9	4.4	685
---	---	2.0	17.8	5.3	680
---	---	3.0	26.7	7.7	985

Table 38. Formulation of rations used in pelleting tests.

Ingredient	Percent of ration by weight	
	AH-65-C	AH-65-D
Soybean meal	12.0	12.0
Rolled milo	40.4	40.4
Rice hulls	34.6	34.6
Cane molasses	10.0	----
Hemicellulose extract	----	10.0
Urea	1.0	1.0
Dicalcium phosphate	1.0	1.0
Vitamin-drug premix	1.0	1.0
	100.0	100.0

Table 39. Percent nitrogen, fineness modulus (F.M.), and modulus of uniformity (M.U.) of the urea used in the pelleting studies.

Type of Urea	Percent Nitrogen	F.M.	M.U.
Feed Urea A	42	3.33	0:9:1
Feed Urea B	42	3.42	0:9:1
Feed Urea C	42	3.38	0:9:1
Small Fertilizer Urea	45	5.47	10:0:0
Large Fertilizer Urea	45	5.79	10:0:0

APPENDIX B

Viscosity, pH, and Degrees Brix of Commercial Samples of Cane Molasses and Various Samples of Hemicellulose Extract

Several samples of cane molasses and corn molasses were collected from commercial sources in the process of this research. The pH, specific gravity, and viscosity of these samples were measured and recorded for reference with regard to pH and viscosity. This data has been tabulated in the following table. Included with this information is the description of samples of hemicellulose extract which were received at various times from the plant locations in Mississippi and California. The trend has been toward a much lower viscosity in hemicellulose extract from Mississippi since production was shifted from a pilot plant operation to a full-scale plant operation in the fall of 1964.

Sample Description	pH	Degrees Brix	Temp. (°C.)	Viscosity (Centipoises) Spindle Number 2	3	4
Cane Molasses						
Sample I.	5.28	----	29.5	750	778	910
			1.0	7,530	5,480	3,410
Sample II.	5.24	----	29.5	1,980	1,850	2,140
			1.0	32,400	24,320	18,140
Sample III.	5.10	----	29.5	1,420	1,580	1,720
			1.0	26,600	16,260	11,760
Sample IV.	5.22	----	57.0	353	----	----
			30.0	2,140	2,030	2,240
			3.0	----	----	33,500
Corn Molasses						
Sample I.	4.89	----	29.5	334	336	----
			1.0	3,600	2,620	1,720
Sample II.	6.14	----	57.0	87	----	----
			30.0	----	----	630
			3.0	----	----	12,200
Hemicellulose Extract (Mississippi)						
Sample I. Not neutralized.	4.26	60.1	30.0	----	----	4,340
Sample II. Neutralized with ammonium hydroxide.	5.71	62.1	30.0	----	----	5,190
Sample III. Neutralized with hydrated lime.	5.54	70.0	30.0	----	----	5,400
Hemicellulose Extract (California)						
Sample I. Not neutralized.	3.83	63.4	30.0	----	502	----
Sample II. Neutralized with ammonium hydroxide.	6.19	65.1	30.0	----	816	----
Sample III. Neutralized with sodium hydroxide.	6.87	67.6	30.0	----	748	----

PHYSICAL PROPERTIES OF HEMICELLULOSE EXTRACT

AS A FEED INGREDIENT

by

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Hemicellulose extract was recently introduced as a feed ingredient for an energy source, and it is used in the dry or liquid form in much the same manner as other molasses products. Hemicellulose extract is the concentrated soluble product obtained from the steam treatment of wood at elevated temperatures and pressures without the use of acids, alkalis, or salts. It is produced at plant locations in California and Mississippi as a by-product in the manufacture of hardboard by Masonite Corporation.

The results of temperature studies showed that hemicellulose extract from Mississippi had a higher viscosity than cane molasses, especially at low temperatures. The viscosity of hemicellulose extract from California was less than or equal to the viscosity of cane molasses at all temperatures from 0°C. to 60°C. The viscosity of hemicellulose extract from Mississippi was effectively reduced by blending it with cane molasses or hemicellulose extract from California.

Two surfactants were tested. Sirlene reduced the viscosity of hemicellulose extract from Mississippi at levels of 1% and 2% by weight. Kem-Wet did not effectively reduce the viscosity of hemicellulose extract or cane molasses when it was used at levels prescribed by the manufacturer.

Several enzymes were tested for their effect on viscosity. A food grade lipase enzyme product and its crude counterpart were found to be the most effective in reducing the viscosity of hemicellulose extract from Mississippi.

Sodium hydroxide and lime were used to neutralize hemicellulose extract. The relationship between the amount of neutralizer and acidity and viscosity was determined over a four-week period. Neutralization of hemicellulose extract with hydrated lime produced an increase in the viscosity. Neutralization with a sodium hydroxide solution produced a decrease in the viscosity of hemicellulose extract from Mississippi. Neutralization of hemicellulose extract from California with a sodium hydroxide solution produced an increase in the viscosity at about pH 5 to pH 7.

A beef cattle ration containing 10% of either hemicellulose extract or cane molasses was pelleted, and the energy requirements and the pellet durability were compared for the two rations. There were no significant differences (0.05 level) in the pellet durability and energy requirements. Five commercial grades of urea were also examined in these tests. There were distinct differences in the energy required to pellet the rations containing the various grades of urea.