A PILOT PLANT STUDY
OF THE ANAEROBIC DIGESTION OF SEMI-CHEMICAL MILL WASTES

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I. INTRODUCTION

Research at Virginia Polytechnic Institute on wastes from the wood-pulp industry was begun on June 1, 1947, under a contract with the National Council for Stream Improvement of the Pulp and Paper Industries. This research was directed toward the alleviation of stream pollution by semi-chemical pulp mill wastes.

Semi-chemical methods of pulping wood are explained briefly in the 1947 annual report to the National Council for Stream Improvement by the Virginia Polytechnic Institute Research Foundation, Incorporated, as follows: "The principle upon which these methods depend is digestion under relatively mild chemical conditions, which soften but do not fully pulp the wood with subsequent reduction to pulp by mechanical disintergration. The name 'semi-chemical' has generally been applied only to those cooks employing neutral sodium sulfite. However, there are a number of other processes operating under mild acid or alkaline conditions which should also be classed as 'semi-chemical'. Among these are semi-sulfate, semi-sulfite, semi-soda and others." (19)

The report further states that semi-chemical pulping was developed largely for the utilization of hardwood timber, since softwood is steadily becoming scarcer.

As a result of this development the paper industry is enabled to utilize an abundant supply of hardwood. Expansion of the industry, however, is hampered by the stream pollution problem presented, and it is urgent that a successful method be found for treatment of wastes from the semi-chemical process.

During the year 1948 research was begun at Virginia Polytechnic Institute by Mr. B. T. Sharp on the possibility of treating semi-chemical pulp mill wastes by anaerobic digestion. His work involved studies of gas production, biochemical oxygen demand removal, and nutrient requirements. During 1949-1950 Mr. G. J. Bruni extended the scope of this investigation to include digester capacities and efficiencies on a laboratory scale.

The author has furthered the investigation of Messrs. Sharp and Bruni by experimenting with a pilot plant instead of a laboratory-sized digester. Actual treatment plant conditions were, therefore, more nearly duplicated. In expressing results the author has used terms suggested by Gehm (20) as standard for the various projects financed by the National Council for Stream Improvement.

II. REVIEW OF THE LITERATURE

Considerable research has been done on the anaerobic digestion of sewage sludge, but little on the digestion of semi-chemical mill wastes. Since it was expected that the same principles would be involved in both digestions, the literature reviewed dealt chiefly with sludge digestion. This review is intended to bring to light a few of the many factors encountered in anaerobic digestion.

Digestion is the decomposition of substances by anaerobic micro-organisms. Fats, cellulose, lignins, and proteins are a few of the substances that occur in domestic sewage sludge. Faber, et al (1) stated that fats, which constitute one of the most important constituents of fresh solids in sludge, disappear rapidly in digestion. Celluloses, which are not present in sludge as abundantly as fats, are said to disappear just as readily, but lignins tend to accumulate. Proteins disappear to a lesser extent than fats, but thorough changes take place in their structure. Some are decomposed completely to ammonia, while others are partially hydrolyzed to amino acids and amides. Still others are resynthesized into the microbial protoplasm.

Metcalf and Eddy (2) state that sludge, being very complex in character, is able to support a variety of groups

of organisms capable of utilizing the different types of food substances as they occur originally in the sewage solids, or as they are modified by the activities of the various organisms developing in the sludge. They claim that the products of one group of organisms become available to other groups until the bulk of the nutritive element is consumed and the sludge is rendered stable. Some of the unicellular micro-organisms were believed to absorb their food through cell membrane. In order to obtain nourishment from solid matter which was not in a form to be absorbed, it was necessary that the matter be peptized by enzymes. The enzymes are catalytic agents carried in or secreted by living cells. From these beliefs it was concluded that correlation existed between the concentration of enzymes and the chemical nature of the substances decomposed.

Rudolfs (3) found in a laboratory experiment that by adding different enzymes to fresh solids, fine screenings and activated sludge, very little practical difference existed in regard to digestion time, gas production, drainability, material handled and odors, between the materials to which enzymes were added and those digested alongside without enzymes. Some of the experiments were conducted under as nearly as practicable optimum temperature and reaction conditions for the greatest activities of the

enzymes added.

Chamberlin (4) named the enzymes occurring during the digestion of a well seeded mixture and determined their activity. Lipase was found to have two peaks of activity; one was to digest the lower fats before maximum gasification. Diastase has a peak of activity coinciding with the peak of gasification. This activity causes the decomposition of starches. Rennet has its greatest activity at the beginning of digestion, coincident with the greatest decomposition of soluble nitrogenous matter. Pepsin has one peak coinciding with the gas peak. During this time it decomposes proteins to proteoses and peptones. Trypsin has two peaks, one coinciding with the first lipase peak during which proteins are decomposed, and the other occurring two weeks after maximum gasification, during which complex proteins, proteoses and peptones are decomposed.

Three major stages have been distinguished in digestion (35), (36). The first stage is characterized by intensive acid production and putrefactive odors. These are caused by organic acids, acid carbonates, and gas consisting chiefly of carbon dioxide with some hydrogen sulphide produced in the first stage. During this stage the pH can be expected to drop to six or lower. The second stage is acid regression. It is during this stage that organic acids and nitrogenous compounds are attacked and ammonia compounds

and acid carbonates are formed. Hydrogen and carbon dioxide are the gases of decomposition. The pH value rises slowly. As the acidity recedes, the third stage, or alkaline fermentation sets in, and the more resistant materials, including proteins and organic acids, are attacked. Methane and carbon dioxide are the gases liberated. The pH may rise above 7.0, and the sludge becomes well buffered. All three stages of digestion are operative at the same time in a balanced digester.

Temperature is an important factor affecting digestion since digestion is a biologically-activated process. Heukelekian (5) found that gas yield, volatile matter reduction, and decomposition of nitrogenous substances are greater in the temperature range 45° C to 60° C (thermophilic conditions) than at lower temperatures. He found that solids concentrated to a greater extent at high temperatures than at lower temperatures, and that digestion was more rapid between 55° C and 60° C than at 45° C to 55° C. When the temperature rose above 60° C digestion retarded.

Rudolfs and Miles (6) conducted experiments at Freehold, New Jersey, in two pilot digestion tanks. Attempts to produce thermophilic sludge from digesting mesophilic sludge, to which fresh solids were added, failed. Operation difficulties, scum formation, objectionable odors,

and little gas production resulted.

Relative to seeding, Rudolfs and Setter (7) found that in order to acclimate the organisms to industrial wastes, sludge that had been produced in previous experiments, should be used as seed. The organisms should then be fed again with the same sludge in increasing quantities.

Pruss (8) states that sludge may be rejuvenated by daily mixing of fresh solids with all the old sludge in the digestion tank. The rejuvenation is brought about by the bacteria living in the old sludge acquiring energy from the easily digestable substances in the fresh sludge. With the added energy the bacteria attack substances in the old sludge which they did not previously digest.

Imhoff and Fair (9) claim that seeding is one of the most important requisites for proper, rapid sludge digestion. They say that seeding is brought about by gas bubbles rising from the decomposing sludge in the bottom of a digestion tank, and lifting sludge particles to the water surface. At the surface a mixture takes place with other particles that have not decomposed. The particles sink back to the bottom when the bubbles escape at the surface. In this way, the mass of sludge is kept in circulation, and the old sludge is brought in contact with incoming fresh sludge particles. Burley (10) points out that a period of two hours between additions of solids should not be exceeded

if gas production is to be sustained.

The idea of mechanical stirring as an aid to digestion has resulted in much experimentation. Pruss (11) claimed that the rotation of sludge nearly doubles the efficiency of the digestion tank since circulation is assumed to provide favorable environment for the bacteria responsible for digestion, by bringing fresh organic material to the active bacteria and by removing the gaseous products of bacterial action.

Cleary (12) had another but equally important reason for agitation of sludge. He claimed it is not only of vital necessity to insure thorough heat distribution and to prevent the formation of any layer effects when a thickened sludge is added to the digestion tank, but with short digestion periods it becomes essential that the fresh solids be quickly seeded without dependence upon natural admixture. At high temperatures the lighter materials in the sludge have a tendency to float and eventually a dense mat of scum is formed, which not only retards the digestion but makes a partly impenetrable mass through which the escape of gas is greatly hindered. This scum becomes fibrous and tough and if allowed to remain for any length of time it is difficult to disperse. Gentle stirring practised from the beginning of operations not only prevents the development of this scum but also serves to release the entrained gasses at a

uniform rate, with the consequent production of a dense, ripe sludge.

Fischer (13) conducted a laboratory experiment to determine the effect of different rates of stirring on digestion. He found that a slow rate showed most beneficial results, in that scum was reduced and a denser sludge was obtained due to the release of entrained gas. This rapid release of gas (which contains an average of about 25 to 30 per cent carbon dioxide) also decreased the acidity and increased the alkalinity of the sludge.

Lime is said by Bachmann (14) to be beneficial to digestion when used in suitable quantities. An excess of lime causes detrimental effects such as retarding digestion, or even causing foaming.

Keefer and Kratz (15) experimented with lime and activated carbon on sludge digestion. It was found that activated carbon reduced the odors but lime was more effective in increasing the speed of digestion.

Schlenz (16) disagrees with the use of lime in digesters, in fact he makes the statement that "lime in any form or amount has no place in a digester". He believes that conditions for proper digestion are not obtained by mere adjustment of the hydrogen-ion concentration. It is his opinion that the proper reaction of the digestion tank contents is the result of proper digestion conditions.

Heukelekian (17) found the carbon-nitrogen ratio in sewage sludge undergoing digestion to be important for the metabolism of the micro-organisms concerned. The food serves to build up their cells and to furnish energy for their activities. The energy is obtained preferably from carbonaceous materials, but in their absence organic substances containing nitrogen can serve as a source of energy. In such cases much more nitrogen will be left in the solution as waste product than if carbonaceous materials are utilized as a source of energy. This is due to the fact that organisms consume more carbon than nitrogen for energy and store more carbon than nitrogen in their cells.

Straub (18) conducted an experiment to determine the effect of the addition of nitrogen on paper pulp digestion. Sodium nitrate was added to supply nitrogen in such quantities as to give a nitrogen to carbon ratio of one to twenty. This ratio approximated that of fresh raw sewage sludge. The sodium nitrate had a decided effect on the digestion of paper pulp in that it reduced the rate of gas production, reduced the methane content, increased the nitrogen content of the gas, and caused a more complete breakdown of the volatile solids.

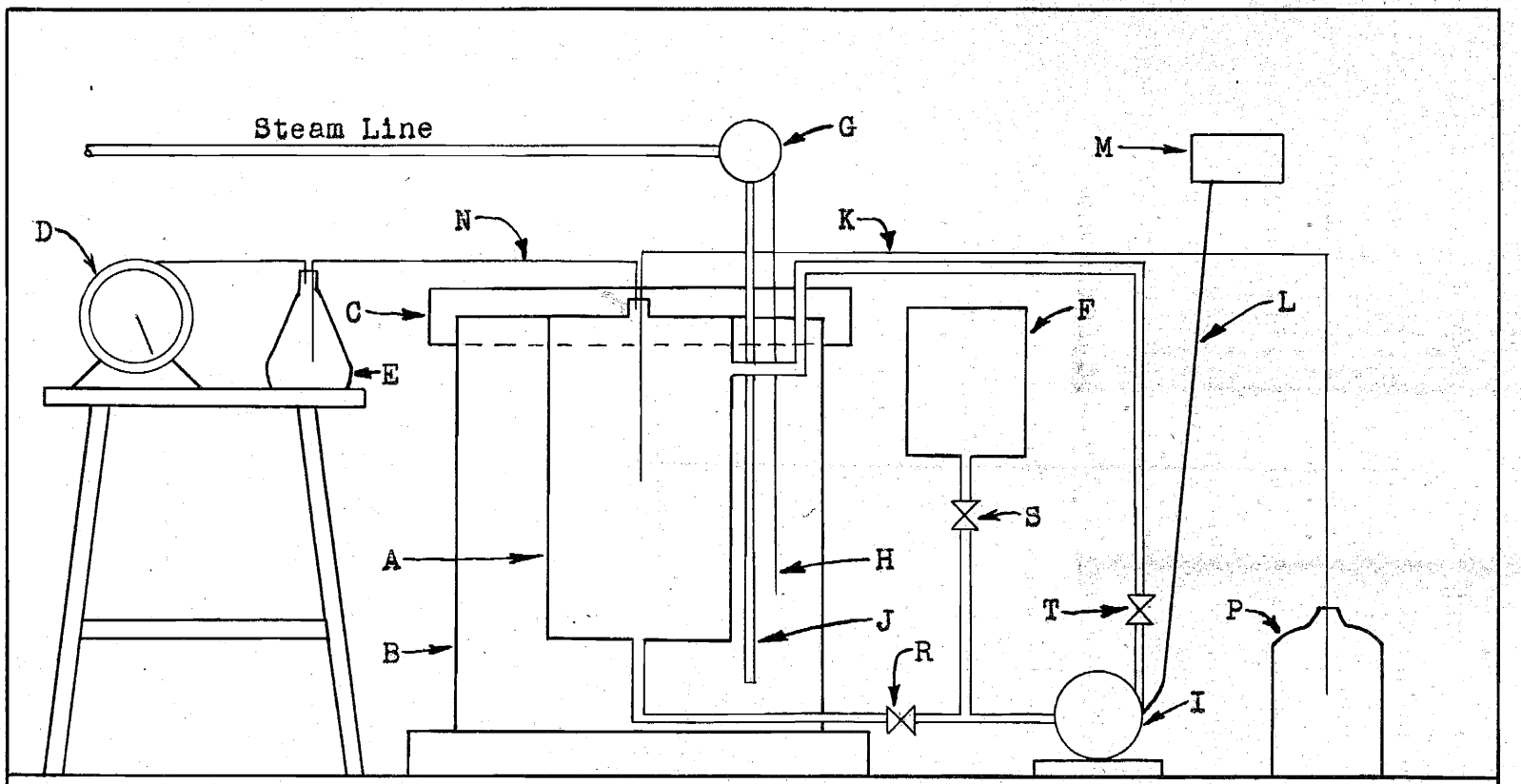
III. THE INVESTIGATION

General

The purpose of this investigation was to determine if high loadings of semi-chemical pulp mill waste could be processed in a pilot plant by anaerobic digestion as successfully as in the laboratory-sized digester investigated by Mr. Bruni.

A pilot plant, shown schematically in Figure I, was seeded with well digested sewage sludge obtained from the Virginia Polytechnic Institute Sewage Disposal Plant. Temperature of the digester was maintained at 37° C and the sludge was recirculated one minute every hour. Mixed hardwood waste was diluted before adding to the digester, so the biochemical oxygen demand (B.O.D.) would be between 1,000 and 3,000 parts per million (p.p.m.). Ammonium phosphate and ammonium hydroxide were added to the waste in order to give a phosphorous to carbon ratio and a nitrogen to carbon ratio of one to twenty.

Loadings on the digester were increased periodically, and the resulting effects on the gas production and biochemical oxygen demand (B.O.D.) were noted. Chemical tests were performed on both the digester influent and effluent so the performance of the digester could be determined. Tests performed were total solids, volatile solids,



A - Digester (15-gallon drum)
B - Water Bath (55-gallon drum)
C - Overflow Launder
D - Wet Test Meter
E - Trap (1 Liter Erlenmeyer Flask)
F - Feed Tank (5 gallons)
G - Thermoregulator
H - Thermoregulator Bulb
I - Centrifugal Pump
J - Steam Line
K - Supernatant Draw-off Line
L - Electric Cord to Motor
M - Recirculation Timer
N - Gas Line
P - Carboy
R, S, & T - Valves

Figure I

Anaerobic Digester for Semi-Chemical Wastes

sulfates, pH, alkalinity, phosphates, sugars and biochemical oxygen demand (B.O.D.).

Method of Procedure

The experimental procedures and the analytical methods employed in this investigation are discussed in the following paragraphs.

Digester Inoculation

The 15-gallon digestion tank was placed inside a 55-gallon drum water bath, with the temperature of the bath being maintained at 37° C. Ten gallons of well digested sludge from the college sewage disposal plant was used to seed the tank. The following day, a quantity of waste was added to the digester to give a loading of 0.045 pounds per cubic foot of digester capacity. The waste was diluted ten times with distilled water, since it was believed the residual chlorine in the tap water would kill the organisms. After a period of operation at this loading, the digester was operated at loadings of 0.055 and 0.065 pounds per cubic foot of digester capacity.

Reinoculation of the Digester

On several occasions the packing in the centrifugal

pump became worn, and air was pumped into the digester. On these occasions it became necessary to empty and reseed the digester with additional sludge obtained from the sewage treatment plant. Attempts to regain anaerobic conditions without emptying the digester, but by inoculating with one gallon of fresh sludge a day, proved unsuccessful. The digester seemed to react much quicker when the entire contents were emptied and replaced.

Addition of Phosphorous to Waste

Phosphorous was added at all times to the waste in the form of ammonium phosphate, so the ratio of phosphorous to carbon would be one to twenty. In order to calculate the amount of ammonium phosphate required for the correct ratio, it was assumed the organic matter was 40 per cent carbon. It is believed that this assumption is very close to being correct, since previous studies on modified kraft waste indicated the solids contained 35 per cent carbon (21). No correction was made for the phosphorous already present in the waste. The effect of adding phosphorous was not studied, as it was not a part of this investigation.

Addition of Nitrogen to Waste

Nitrogen in the form of ammonium hydroxide was added to the waste before adding to the digester. An amount of nitrogen was added to give a nitrogen to carbon ratio of

one to twenty. As with phosphorous, no correction was made for the nitrogen already present in the waste, and a study was not made of the effect of nitrogen.

Feeding of the Digester

Fresh portions of diluted waste were added to the digester A, as shown on Figure I, in the following manner. Recirculation was stopped by closing valves R and I, and the sludge was allowed to settle at least an hour in order to draw off a clear supernatant. The amount of diluted waste added was maintained at 9.12 liters, but the amount of concentrated waste was increased as the loading was increased. Since 9.12 liters were to be added to the digester an equal amount was siphoned through the rubber tubing K. The carboy P was marked at a volume of 9.12 liters for convenience in measurement. The diluted waste was poured into the feed tank F, and when the correct amount of supernatant had been siphoned valves R, S, and T were opened and the pump I was turned on. Valve S was closed as soon as the feed tank was empty in order to prevent the pumping of air into the digester. The feeding was completed by turning off the pump.

Methods Used to Increase Loading

It had originally been planned to begin with a loading of 0.03 pounds of biochemical oxygen demand (B.O.D.) per

cubic foot of digester capacity per day, maintain this loading for one week, then increase the loading 0.01 pounds per cubic foot. The biochemical oxygen demand (B.O.D.) of the diluted waste was greater than expected and it also varied somewhat every day, so that a constant loading could not be maintained. After using the same concentration of waste for a five day period, the biochemical oxygen demand (B.O.D.) of the feed was determined and instead of the loading being 0.03 pounds per cubic foot, the loading was 0.045 pounds per cubic foot. Since the objective was to use heavy loadings on the digester, the same concentration of waste was used for the feed. The loading was later increased to approximately 0.055 pounds per cubic foot, and then to 0.065 pounds per cubic foot.

Total Solids Determination

A volume of sample was pipetted into a tared porcelain dish and evaporated. It was then dried at 103° C to a constant weight. The weight of the solids in milligrams multiplied by 1,000 and divided by the volume of the sample in milliliters gave the total solids of the sample in parts per million (p.p.m.).

Volatile Solids Determination

After obtaining the weight of the above sample it was ignited at 600° C for one-half hour. The dish was cooled

to 103° C, then a few drops of ten per cent ammonium carbonate solution were used to moisten the solids. The dish was dried for one hour at 103° C, then placed in a dessicator before weighing. The loss in weight was the volatile matter, and the parts per million (p.p.m.) of volatile solids were calculated by dividing the volatile matter expressed in milligrams by the volume of the sample in liters.

Sulfates Determination

To a volume of sample was pipetted hydrochloric acid. After stirring, the mixture was heated to aid flocculation. The mixture was then filtered, and the filtrate was heated to boiling. A slight excess of barium chloride solution was then added with stirring, and the precipitate was digested for two hours. The precipitate was then filtered and washed until chloride-free, and ignited at 800° C to a constant weight.

pH Determination

The pH was determined with a Beckman pH meter. A glass and a calomel electrode were submerged in the sample and the meter was read.

Alkalinity Determination

The alkalinity was determined with a Beckman pH meter. If the pH was above 8.3, the sample was titrated with dilute

sulfuric acid until a pH of 8.3 was reached. The milliliters of acid required for the titration were recorded, then the sample was further titrated until a pH of 4.0 was reached. The milliliters of acid for this titration were recorded and from the titrations the alkalinity of the sample could be determined.

Sugars Determination

Copper sulfate solution and alkaline tartrate solution were pipetted into a beaker. A volume of the filtrate obtained in the sulfate determination was then pipetted into the beaker. The beaker was covered with a watch-glass and heated so that boiling began in four minutes. The boiling was then continued for two minutes. The hot solution was filtered at once through an asbestos mat in a porcelain Gooch crucible, using suction. The precipitate was washed thoroughly with water, and after drying at 103° C it was weighed. The weight obtained is the weight of cuprous oxide, and by using Munson and Walker's table the concentration of dextrose was calculated.

Phosphate Determination

Nitric acid solution was added to a volume of the filtrate obtained in the sulfate determination, and was heated to 80° C. Ammonium molybdate solution was added with stirring until precipitation began. The precipitate was

digested for one hour at 40° C, then washed with nitric acid solution. The precipitate was then dissolved with ammonium hydroxide, and hydrochloric acid was added until a yellow precipitate appeared. Magnesium chloride solution and ammonium hydroxide were added with stirring. The precipitate was digested for two hours, then filtered and washed with ammonium hydroxide. The precipitate was ignited, then cooled to 103° C prior to being placed in a dessicator before weighing.

Ammonia Nitrogen Determination

A volume of sample was placed in a Kjeldahl flask with phosphate buffer solution to keep the pH at 7.4 during the distillation. The mixture was diluted with ammonia-free water, then 200 milliliters were distilled. A portion of the distillate was Nesslerized, and comparison made on a disc comparator.

Organic Nitrogen Determination

A volume of sample was concentrated to about ten milliliters by boiling in a Kjeldahl flask. Copper sulfate solution, sodium sulfate, and concentrated sulfuric acid were added to the concentrate. After boiling under a hood until the digestate became clear, ammonia-free water was added. The solution was made alkaline by adding a few drops of phenolphthalein, then adding sodium hydroxide until a red

color appeared. About 200 milliliters of distillate were obtained, and a portion was Nesslerized as in the ammonia nitrogen determination.

Nitrate-Nitrite Nitrogen Determination

Sodium hydroxide was added to a volume of sample and concentrated to about 20 milliliters in a casserole to remove free ammonia. The concentrated sample was put in a test tube with ammonia-free water and a strip of aluminum foil. The tube was loosely covered with a stopper to protect the solution from dust. The reduction was allowed to take place overnight, then the sample was poured into a Kjeldahl flask and diluted with ammonia-free water. Two hundred milliliters of distillate were collected, and a portion Nesslerized as in the ammonia nitrogen determination.

Dilution Water

The dilution water used for the biochemical oxygen demand (B.O.D.) was made by adding to a carboy of distilled water 10 milliliters of ferric chloride solution, 50 milliliters of calcium chloride solution, 50 milliliters of magnesium sulfate solution and 25 milliliters of phosphate buffer solution. After adding the above solutions, the water was aerated for 24 hours before using.

Biochemical Oxygen Demand Determination

Various dilutions were made of the samples in approximately 300 milliliter bottles. The diluting water was seeded with 25 milliliters of cotton filtered raw sewage obtained from the pumping station at Virginia Polytechnic Institute. The sodium azide modification of the Winkler process was used for the biochemical oxygen demand (B.O.D.) determinations. After incubating for five days at 12° C, concentrated sulfuric acid and sodium azide were added to the bottles. After vigorous shaking the bottles were permitted to stand for ten minutes, then manganous sulfate solution and alkaline iodide solution were added well below the surface of the liquid. Again the bottles were shaken vigorously, then allowed to stand for ten minutes or longer. Concentrated sulfuric acid was again added and shaking repeated. Two hundred milliliters were poured into an Erlenmeyer flask and titrated with sodium thiosulfate until only a small amount of iodine remained. Starch solution was added to give a blue color, and titration was continued to the first disappearance of the blue color. Since the incubation was at 12° C instead of the standard 20° C, it was necessary to convert all calculations from 12° C to 20° C. This conversion was made by applying the factor obtained from a time and temperature relations table (22).

IV. DATA AND RESULTS

General

The data and results obtained from the investigation of the anaerobic digestion of the waste liquor from semi neutral-sulfite pulping of mixed hardwoods are tabulated and graphed on the following pages.

At various times throughout the experiment the centrifugal pump, used for adding feed and recirculating the contents of the digester, pumped air. When this occurred the anaerobic conditions of the digester were naturally eliminated, and it was necessary to reseed the digester with sludge. Whenever reseeding took place the results of the effluent chemical tests were found to vary considerably from results obtained at other times. The variation is evidently due to greater proportions of sludge than mixed hardwood waste being drawn from the digester for testing purposes. The effluent chemical results for the 17th and 25th day of April seem to be influenced by reseeding, therefore, they are not considered reliable. The biochemical oxygen demand (B.O.D.) for the effluent on April 26 also offers a doubt as to its reliability.

Concentrated Semi-Chemical Waste

General

An analysis of the concentrated mixed hardwood waste was made April 20, 1951, the day it was received from the Mead Corporation, Lynchburg, Virginia. The following table contains the results of the analysis.

TABLE I

Analysis of Mead Mixed Hardwood Waste

Total Solids (ppm)	109,816
Volatile Solids (ppm)	52,560
Ammonia Nitrogen (ppm NH_3)	17.6
Organic Nitrogen (ppm NH_3)	260
Nitrate-Nitrite Nitrogen (ppm NH_3)	22
Sulfates (ppm SO_4)	1,860
Alkalinity (ppm $CaCO_3$)	16,760
pH	7.8
Phosphates (ppm PO_4)	683
Sugars (ppm Dextrose)	5,700
B.O.D. (ppm)	25,550

Ammonia Nitrogen

General

When organic nitrogenous matter is decomposed by bacterial activity, a portion of the organic matter is used for food by the bacteria and a part is converted into ammonia. Although there are some variations in the process of ammonification, depending on the organisms and the conditions involved, the successive products are usually proteoses, peptones, amino acids, and ammonia.

Buswell and Hatfield (27) found the minimum ammonia nitrogen concentration suitable for the anaerobic fermentation of cellulosic material to be 100 parts per million (p.p.m.). Greater concentrations do not aid materially nor have they been found bacteriostatic. T hey say the bacteria seemingly utilize the ammonia nitrogen to build up their own structure. Lysis of these bacteria, following death, releases the nitrogen which under anaerobic conditions is again reduced to ammonia nitrogen. Because of this cycle it is necessary to add ammonia nitrogen only to raise the initial content of the tank to 100 parts per million (p.p.m.) or greater.

The following table which gives the results of ammonia nitrogen tests is plotted on plate I.

TABLE II

Results of Ammonia Nitrogen Tests (ppm NH_3)

Date	Influent	Effluent
April 12	220	-
April 17	1	120
April 19	220	-
April 25	140	200
May 1	120	100
May 3	250	250
May 8	300	325
May 10	500	450
May 15	200	150
May 17	150	250

Discussion of Results

As previously reported ammonium phosphate and ammonium hydroxide were added as nutrients to the digester feed. On April 17, 1951, instead of using the above nutrients, potassium phosphate and sodium nitrate were used to increase the phosphorous and nitrogen. This explains the sharp decrease in ammonia nitrogen as found in the feed for April 17, 1951.

The results of this test seem to bear out the findings of Buswell and Hatfield that the ammonia nitrogen is utilized by bacteria to build up their structure, and after their

death the nitrogen is reduced again to ammonia nitrogen. Plate I shows little variation in ammonia nitrogen between the feed and the effluent.

Organic Nitrogen

General

The organic nitrogen determination is a measure of the nitrogen in the form of proteins or intermediate decomposition products (28). Nitrogen was added to the feed in the forms of ammonium hydroxide and ammonium phosphate to fulfill the requirements of the bacteria carrying on decomposition.

Plate II furnishes a graphic picture of the data in the table below.

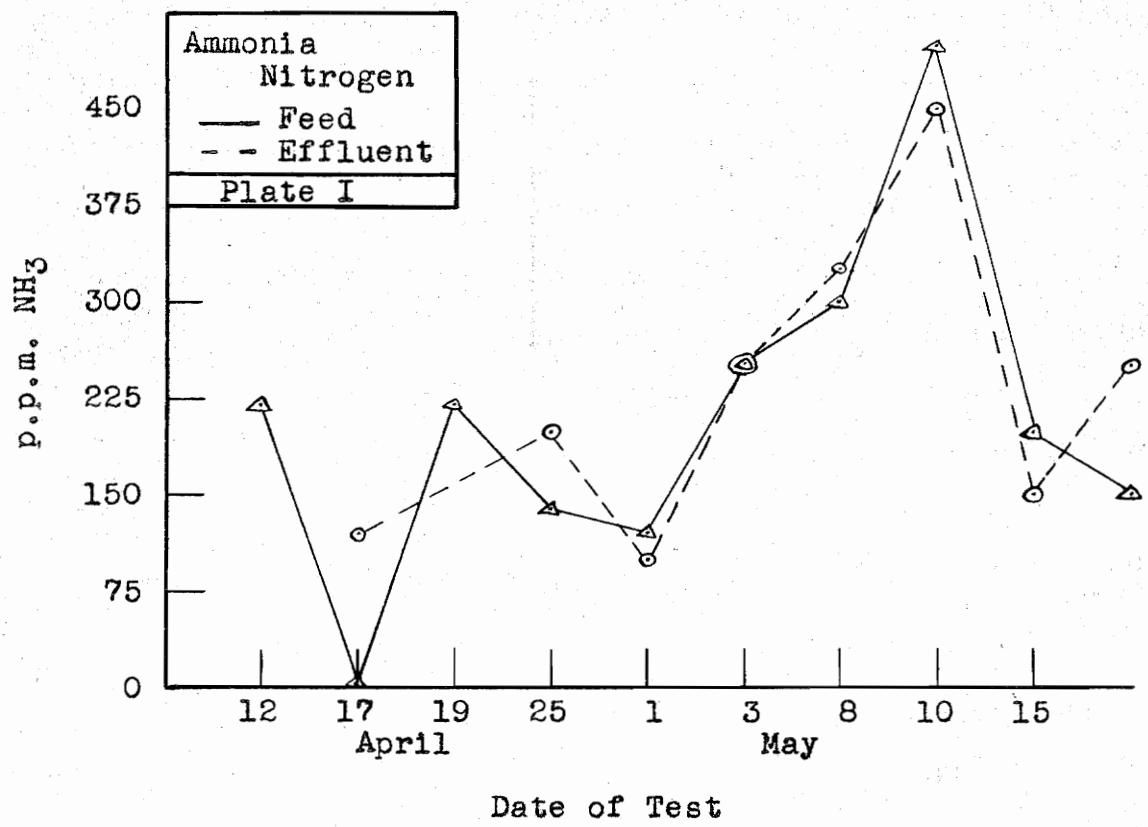
TABLE III

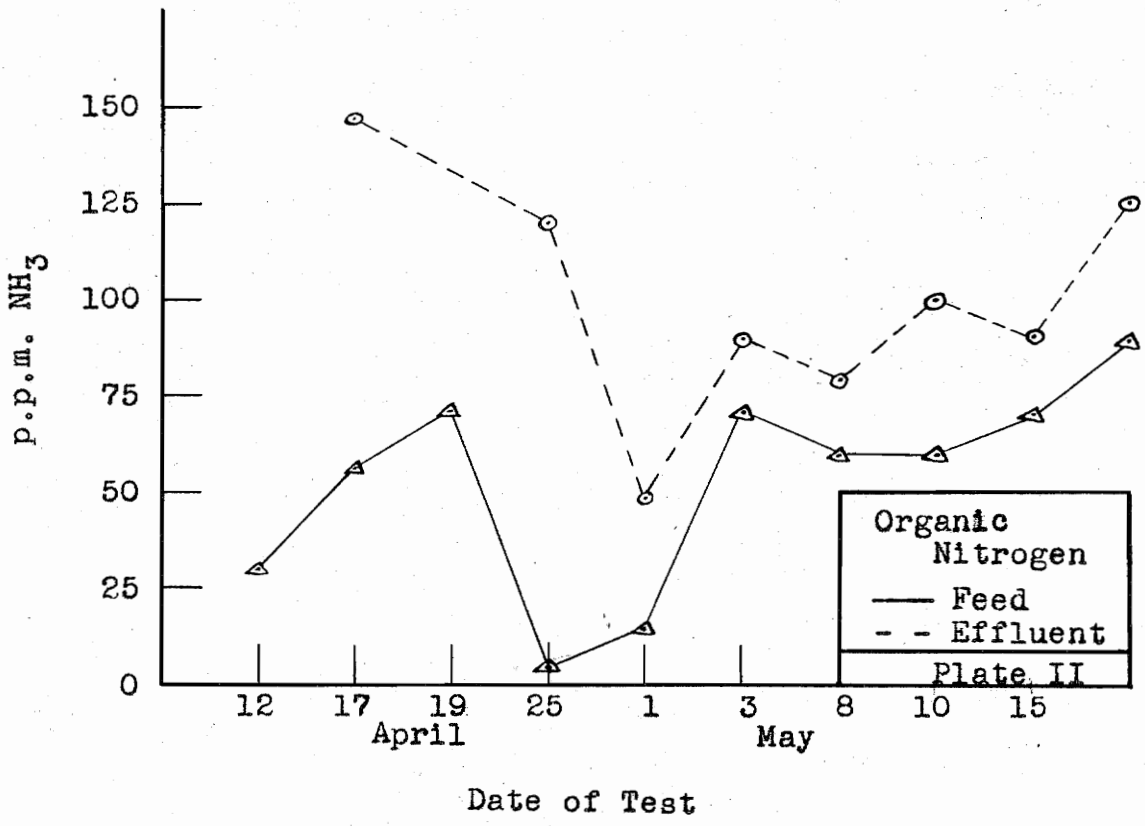
Results of Organic Nitrogen Tests (ppm NH_3)

Date	Influent	Effluent
April 12	30	-
April 17	56	146.3
April 19	72	-
April 25	4	120
May 1	14	48
May 3	72	90
May 8	60	80
May 10	60	100
May 15	70	90
May 17	90	125

Discussion of Results

Plate II shows an increase in organic nitrogen as the waste is passed through the digester. The increase in organic nitrogen indicates a growth of bacteria in the digester, which in turn, suggests the digester is operating successfully.


Ammonia Nitrogen
Feed
Effluent
Plate I
p.p.m. NH_3
450
375
300
225
150
75
0
12
17
19
25
1
3
8
10
15
April
May
Date of Test


Organic Nitrogen
Feed
Effluent
Plate II
p.p.m. NH_3
150
125
100
75
50
25
0
12
17
19
25
1
3
8
10
15
April
May
Date of Test

Nitrate-Nitrite Nitrogen

General

Nitrates in a waste may be changed to other compounds by the reduction processes of anaerobic bacteria. In order for this reduction to take place, three conditions are required in addition to the necessary organisms: (1) a lack of oxygen, (2) a supply of nitrates, and (3) a supply of food such as carbohydrates to furnish the necessary energy.

Numerous bacteria reduce nitrates - some are facultative anaerobes and some strict anaerobes. Escherichia coli, Aerobacter aerogenes, and Clostridium perfringens are a few of the bacteria reducing nitrates.

There are several products of nitrate reduction. Often there is formed in succession nitrous acid or a nitrite, hyponitrous acid, hydroxylamine, and ammonia. Some of the nitrate may be used for food by the bacteria and changed into their proteins. The oxygen of the nitrate is used in bacterial respiration (25).

Nitrites are very unstable and are found when a change is taking place. They are either being reduced to ammonia or oxidized to nitrates.

The "nitrous" bacteria oxidize the ammonia to nitrites, and the "nitric" bacteria oxidize the nitrites to nitrates. No species of bacteria is known to do both. The bacteria

obtain their energy by the oxidation processes involved. Nitrosomonas and Nitrosococcus are the "nitrous" bacteria, and Nitrobacter is the "nitric" bacteria (26).

The data obtained from this test is shown below and is plotted on Plate III.

TABLE IV

Results of Nitrate-Nitrite Nitrogen Tests (ppm NH_3)

Date	Influent	Effluent
April 12	1.8	-
April 17	18	14
April 19	1.4	-
April 25	7	64
May 1	8	10
May 3	12.6	12
May 8	6.0	25
May 10	10	36
May 15	28	16
May 17	19.2	20

Discussion of Results

The sharp rise in nitrate-nitrite nitrogen in the digester feed for April 17 is probably due to the use of sodium nitrate as a nutrient, on that particular day. The great increase in the effluent nitrate-nitrite nitrogen for April 25, as previously stated, is evidently influenced by the reseeding of the digester on April 23.

It is questionable as to what degree, if any, an increase of nitrate-nitrite nitrogen in the digester will have upon the operation of the digester. Heukelekian (31) states the production of nitric acid by nitrification may affect the pH value in poorly buffered substrate or in large quantities. If the pH of the digester was affected by an increase of nitric acid, it is believed a favorable affect took place since the pH values were within the ranges usually associated with favorable digestion.

Sulfates

General

In a waste containing sulfates, the sulfates can be broken down by reduction in the absence of oxygen (23). Hydrogen sulfide is produced by this reduction which is the decomposition of sulfur-bearing organic matter under

anaerobic conditions.

The following table is plotted on Plate IV in order to furnish a relationship between the sulfates present in the feed and effluent.

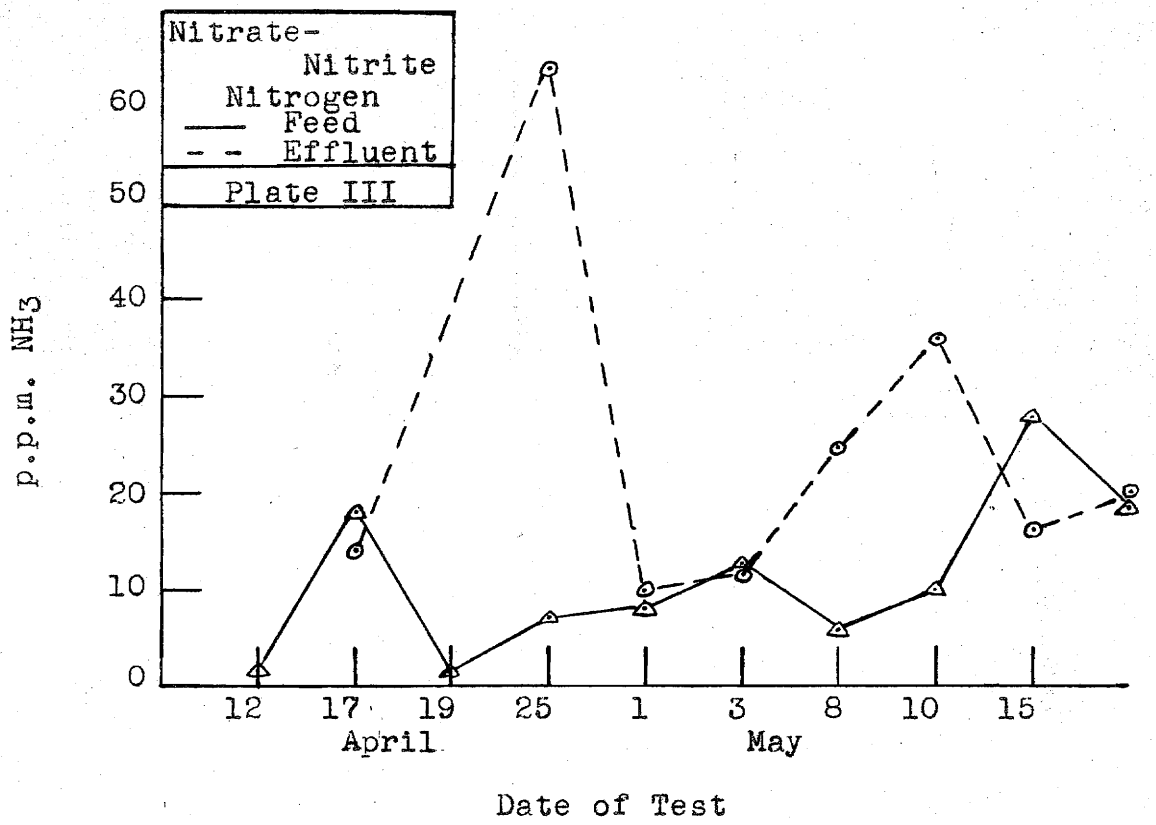
TABLE V

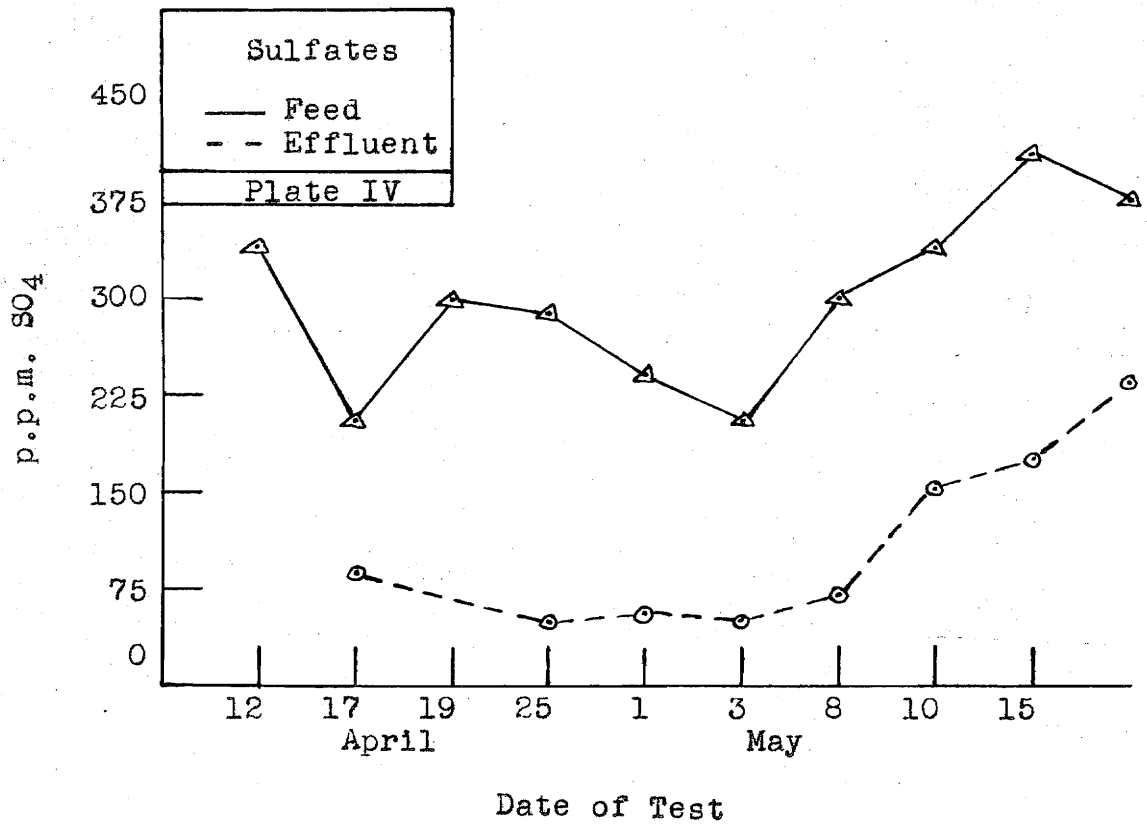
Results of Sulfates Tests (ppm SO_4)

Date	Influent	Effluent
April 12	344	-
April 17	208	88
April 19	300	-
April 25	290	50.2
May 1	244	55
May 3	207	49.4
May 8	301	70.9
May 10	341	152
May 15	414	176
May 17	379	236

Discussion of Results

Plate IV indicates the digester was successful in reducing the sulfates. No tests were made to determine the presence or actual quantity of hydrogen sulfide given off


Nitrate-
Nitrite
Nitrogen
Feed
Effluent
Plate III
p.p.m. NH_3
60
50
40
30
20
10
0
12
17
19
25
1
3
8
10
15
April
May
Date of Test


Sulfates
Feed
Effluent
Plate IV
p.p.m. SO_4
450
375
300
225
150
75
0
12
17
19
25
1
3
8
10
15
April
May
Date of Test

during digestion, but as samples were drawn from the digester the odor of hydrogen sulfide was prevalent. The decrease in the sulfates as the waste passed through the digester, with the resulting odor of hydrogen sulfide, indicated the waste was in the first stage of digestion. It is during this stage that the materials easily available for food are attacked by the bacteria. The materials would be sugars, starches, carbohydrates and soluble nitrogenous compounds. The first stage is characterized by acid production and putrefactive odors (24).

Total Solids

General

Total solids, or residue on evaporation, are important as an index of the concentration of the waste. Total solids include both the suspended and the dissolved solids, and are broken down into volatile (loss on ignition) and fixed mineral matter. Generally it is assumed that the volatile portion represents organic matter, and since this portion may putrefy it is much more significant in regards to the concentration of the waste.

On plate V is plotted the results of the total solids tests given in the following table.

TABLE VI

Results of Total Solids Tests (ppm)

Date	Influent	Effluent
April 12	9,168	-
April 17	12,108	21,672
April 19	8,772	-
April 25	8,956	29,328
May 1	11,240	10,968
May 3	10,788	9,580
May 8	12,316	10,100
May 10	11,172	10,792
May 15	12,964	10,636
May 17	13,752	12,972

Discussion of Results

The high values for the effluent on the 17th and 25th of April are probably due to the reseeding with digested sludge on the 11th and 23rd of April. Otherwise the results agree with what one would expect to take place. The total solids decreased as the waste passed through the digester, indicating that successful operation of the digester was in progress.

Volatile Solids

General

As previously stated, the volatile solids are approximately a measure of the organic matter present. The determination of volatile solids gives an idea of the quantity, but not of the character of the organic matter present.

Results of the volatile solids tests are given below and are plotted on plate VI.

TABLE VII

Results of Volatile Solids Tests (ppm)

Date	Influent	Effluent
April 12	2,672	-
April 17	4,092	10,756
April 19	2,312	-
April 25	2,600	12,944
May 1	3,840	3,388
May 3	3,416	2,544
May 8	4,104	2,536
May 10	3,900	3,288
May 15	3,580	2,224
May 17	4,568	3,852

Discussion of Results

As would be expected, the results of the volatile solids tests are very similar to the results of the total solids examinations. In comparing the two plates (V and VI) it is interesting to note that as the total solids in the feed increase, the volatile solids increase, and the same occurs with a decrease. This would indicate that a ratio definitely exists between the total and volatile solids for this particular waste.

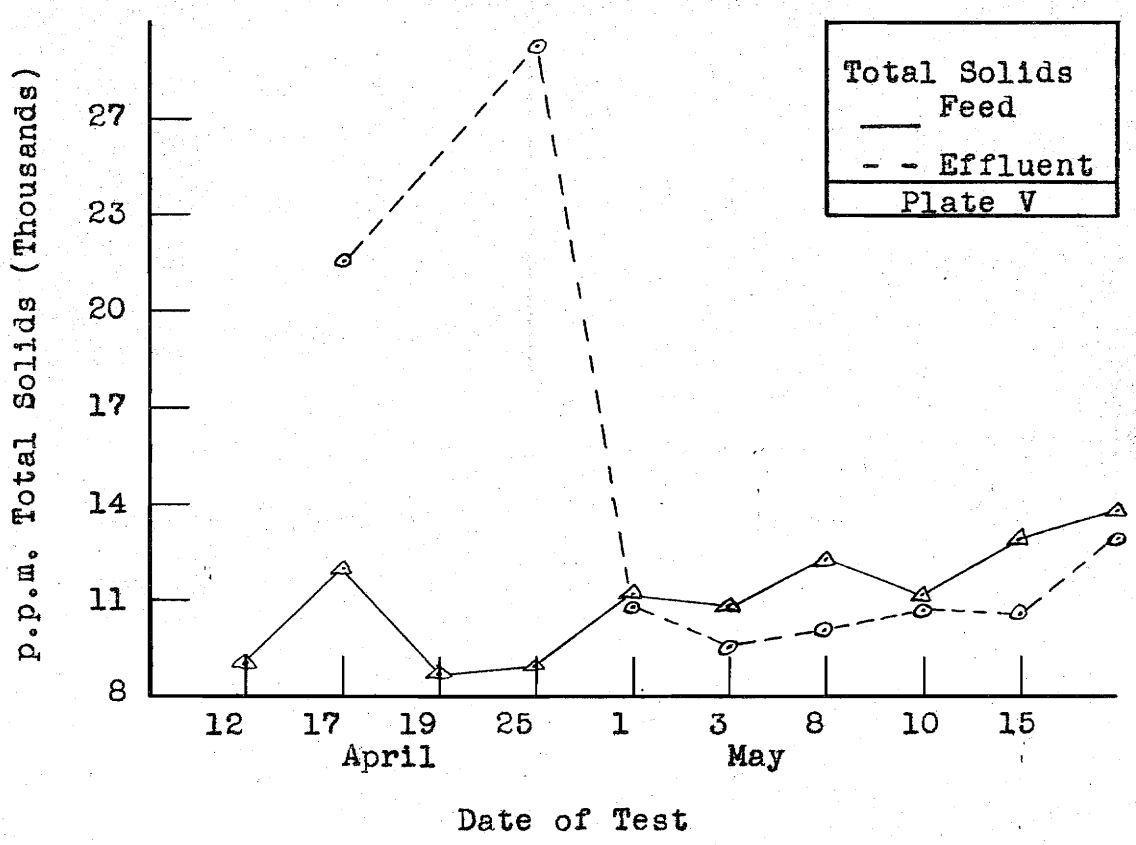
From plate VI it is found that the volatile solids are being reduced by the organisms in the digester, indicating favorable operation.

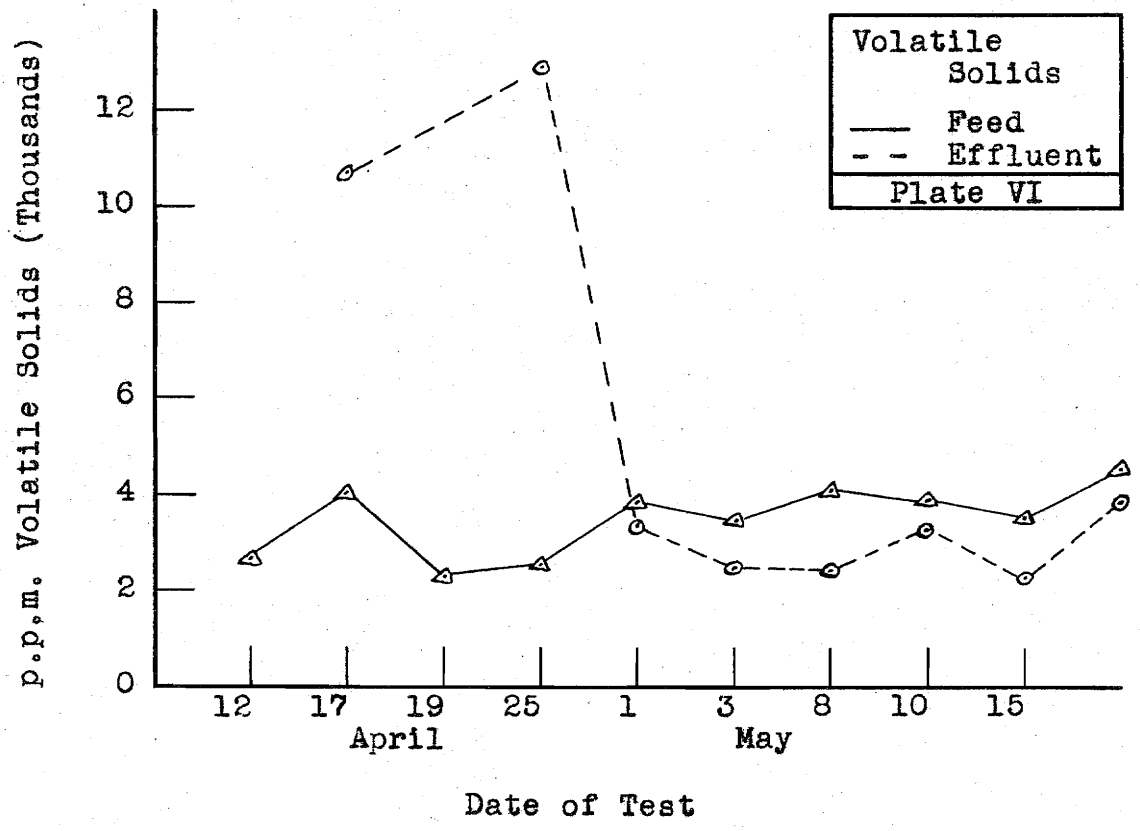
pH

General

pH is a method of expressing the alkalinity or acidity. The pH is defined as the logarithm of the reciprocal of the hydrogen-ion concentration when that concentration is expressed as moles of hydrogen-ion per liter of solution. pH ranges from 0 to 14 with 7 being the neutral point. From 0 to 7 indicates acidity and 7 to 14 alkalinity.

Whipple (29) says the effect of different hydrogen-ion values upon the growth of microscopic organisms is


Total Solids
Feed
Effluent
Plate V
p.p.m. Total Solids (Thousands)
27
23
20
17
14
11
8
12
17
19
25
1
3
8
10
15
April
May
Date of Test


Volatile Solids
Feed
Effluent
Plate VI
p.p.m. Volatile Solids (Thousands)
12
10
8
6
4
2
0
12
17
19
25
1
3
8
10
15
April
May
Date of Test

not known. However, it is well known that hydrogen-ion concentration will change with fluctuating plankton growth. The hydrogen-ion concentration may considerably affect the absorption of food by bacteria and its ultimate utilization.

The following table gives the results of the pH tests which are plotted on plate VII.

TABLE VIII

Results of pH Tests

Date	Influent	Effluent
April 12	8.8	-
April 17	7.9	7.7
April 19	8.7	-
April 25	8.2	7.5
May 1	8.7	6.7
May 3	8.9	7.5
May 8	8.2	7.5
May 10	9.7	8.0
May 15	9.4	7.4
May 17	9.2	7.2

Discussion of Results

The results of this test signify conditions inside the digester are favorable for organic activity. Only one test gave an acid pH for the effluent, and two days later the pH had adjusted itself without the addition of lime. The pH of the feed is high, but falls within a satisfactory range after being in the digester a short time.

Alkalinity

General

Alkalinity may be due to the presence of the carbonates or bicarbonates of potassium, sodium, calcium, magnesium, or to silicates, borates and phosphates (30). The measurement of alkalinity serves as another index of biological activity.

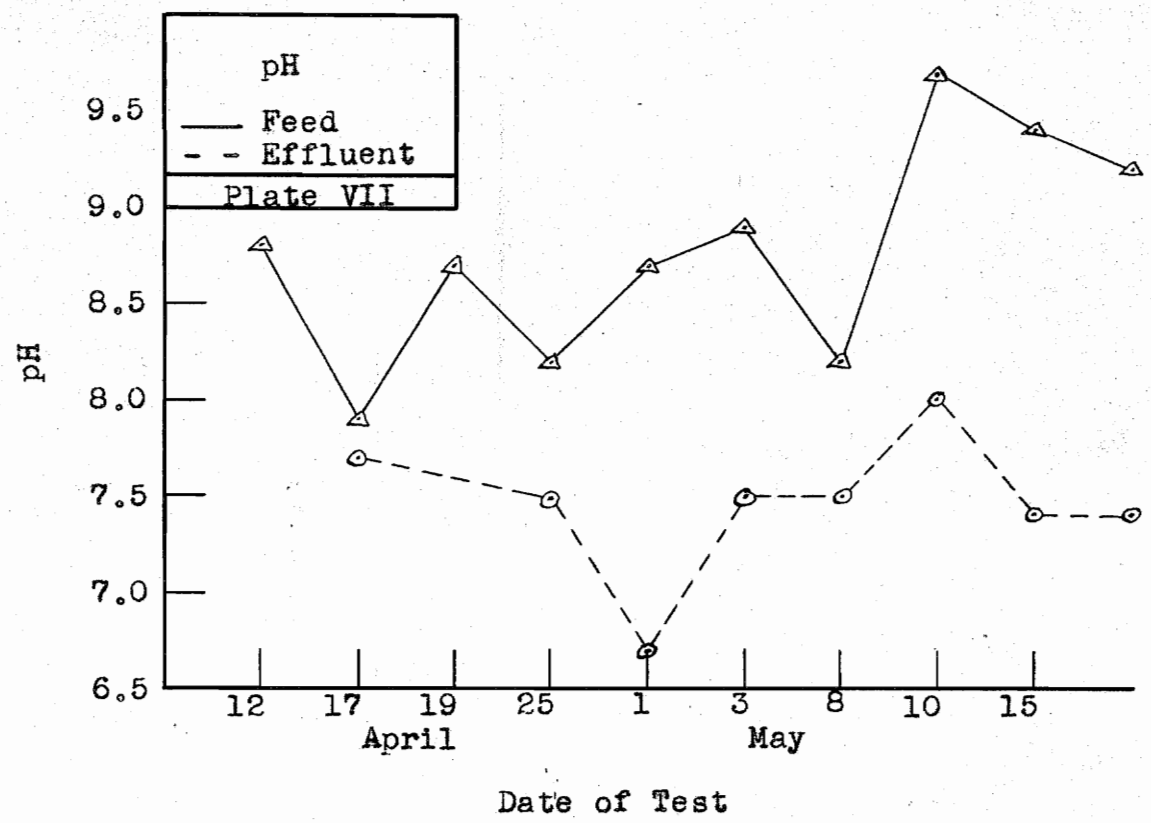
TABLE IX

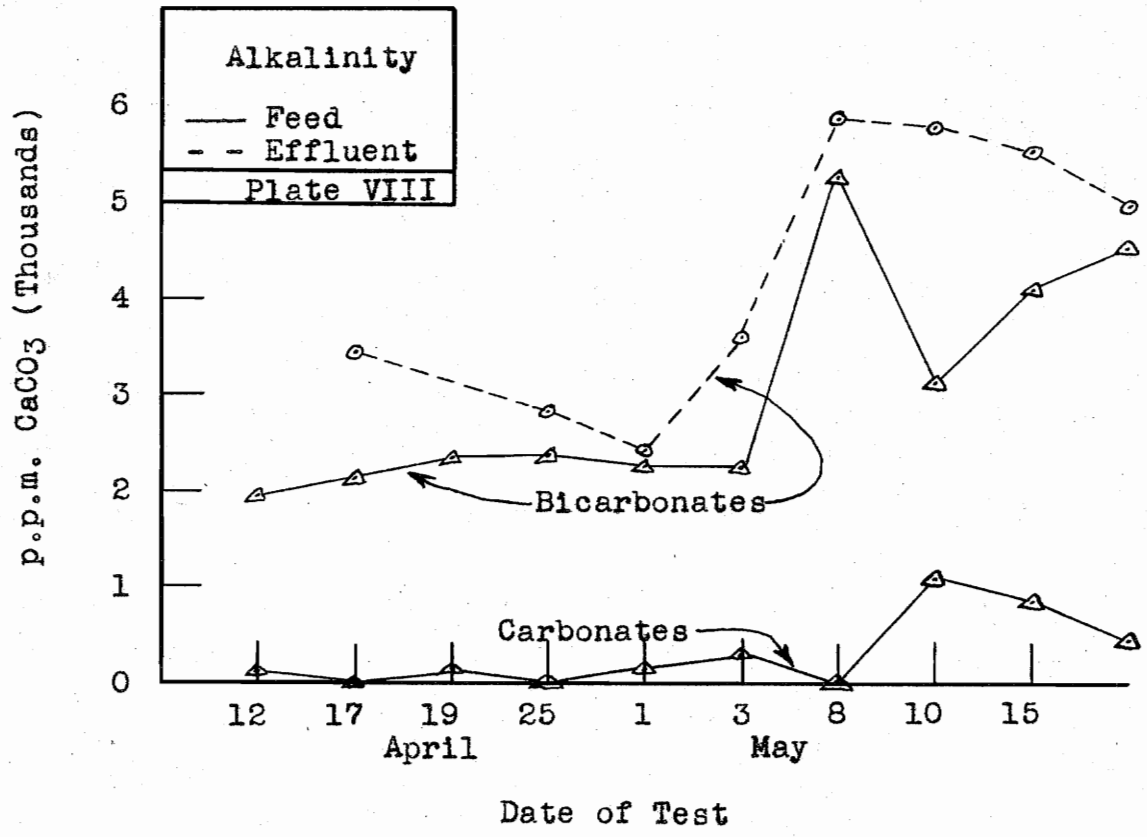
Results of Alkalinity Tests (ppm $CaCO_3$)

Date	Influent		Effluent
	Carbonates	Bicarbonates	Bicarbonates
April 12	100	1,976	-
April 17	-	2,154	3,460
April 19	152	2,360	-
April 25	-	2,390	2,800
May 1	190	2,295	2,410
May 3	310	2,250	3,605
May 8	-	5,270	5,890
May 10	1,100	3,120	5,800
May 15	880	4,100	5,510
May 17	488	4,550	4,990

Discussion of Results

It is believed the sharp rise in alkalinity on May 8 for both the feed and effluent was probably due to the increase in loading on the digester May 7. When the load was increased a greater quantity of ammonium phosphate was added to the feed, which in turn evidently increased the alkalinity. The bicarbonate increase in the effluent is probably due to the neutralization of carbonate alkalinity either by carbon dioxide or organic acid produced during the fermentation.


pH
Feed
Effluent
Plate VII
9.5
9.0
8.5
8.0
7.5
7.0
6.5
pH
12
17
19
25
1
3
8
10
15
April
May
Date of Test


Alkalinity
Feed
Effluent
Plate VIII
6
5
4
3
2
1
0
p.p.m. CaCO3 (Thousands)
Bicarbonates
Carbonates
12
17
19
25
1
3
8
10
15
April
May
Date of Test

Phosphates

General

Phosphorous was added to the feed in the form of ammonium phosphate. It was not known if the organisms would utilize this phosphorous for their structure, but it was believed the phosphorous would have no detrimental affect on the organisms.

TABLE X

Results of Phosphate Tests (ppm PO_4)

Date	Influent	Effluent
April 19	930	-
May 1	922	905
May 3	990	1,050
May 8	1,185	1,150
May 10	1,390	1,260
May 15	1,475	1,260
May 17	1,363	1,300

Discussion of Results

Plate IX indicates an almost continuous increase in the phosphorous content of both the feed and the effluent. The increase in the feed content is partially due to the increase in the digester loadings beginning on the 7th and 14th of May. The amount of ammonium phosphate added was increased when the loading was increased. The effluent increase indicated the organisms were not using the phosphorous in sufficient quantities to compensate for the additional phosphorous in the feed. Since the phosphorous in the effluent was in all but one case less than the phosphorous in the feed, it is assumed the organisms utilized a certain quantity for their cell structure. Once again the results of a test signify conditions inside the digester are favorable for organic activity.

Sugars

General

Buswell and Hatfield (32) found that when dextrose is fermented a rapid evolution of gas occurs in the beginning, but later subsides to a slower, more steady evolution as the reaction proceeds. During the initial, rapid period acids are formed, and during the later evolution of gas the acids are being decomposed.

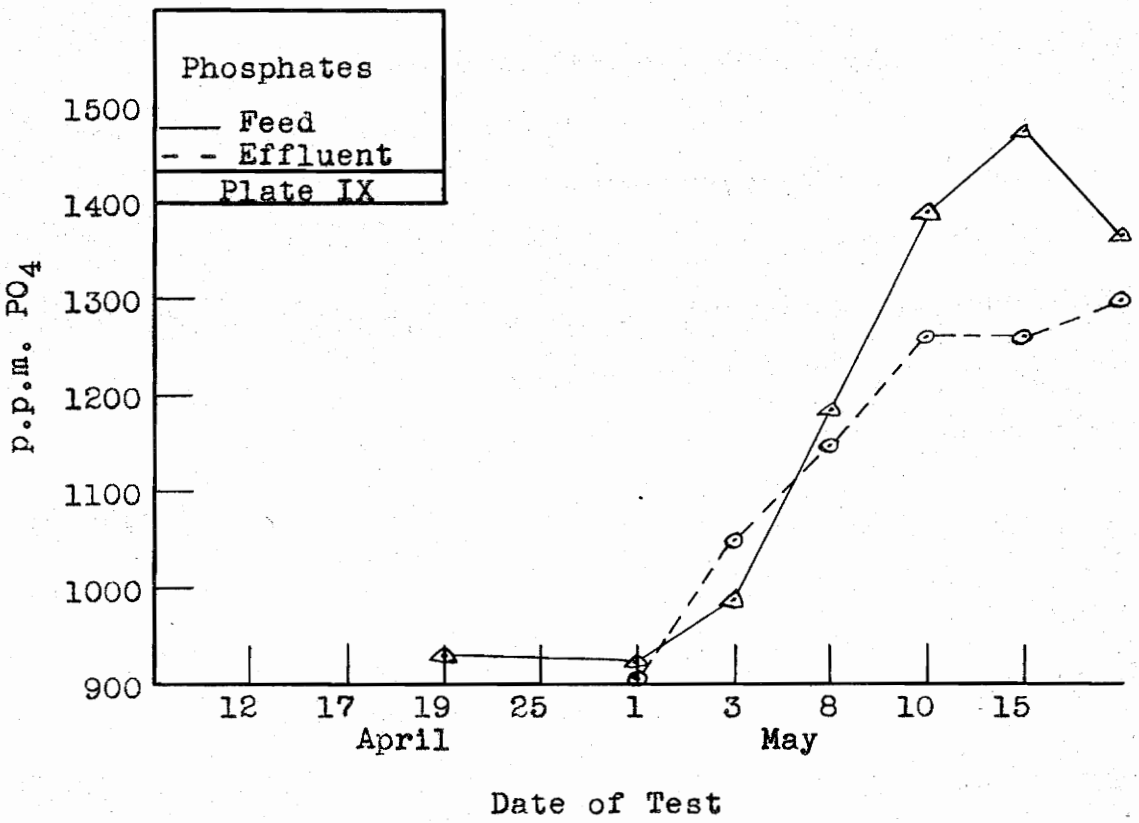
TABLE XI

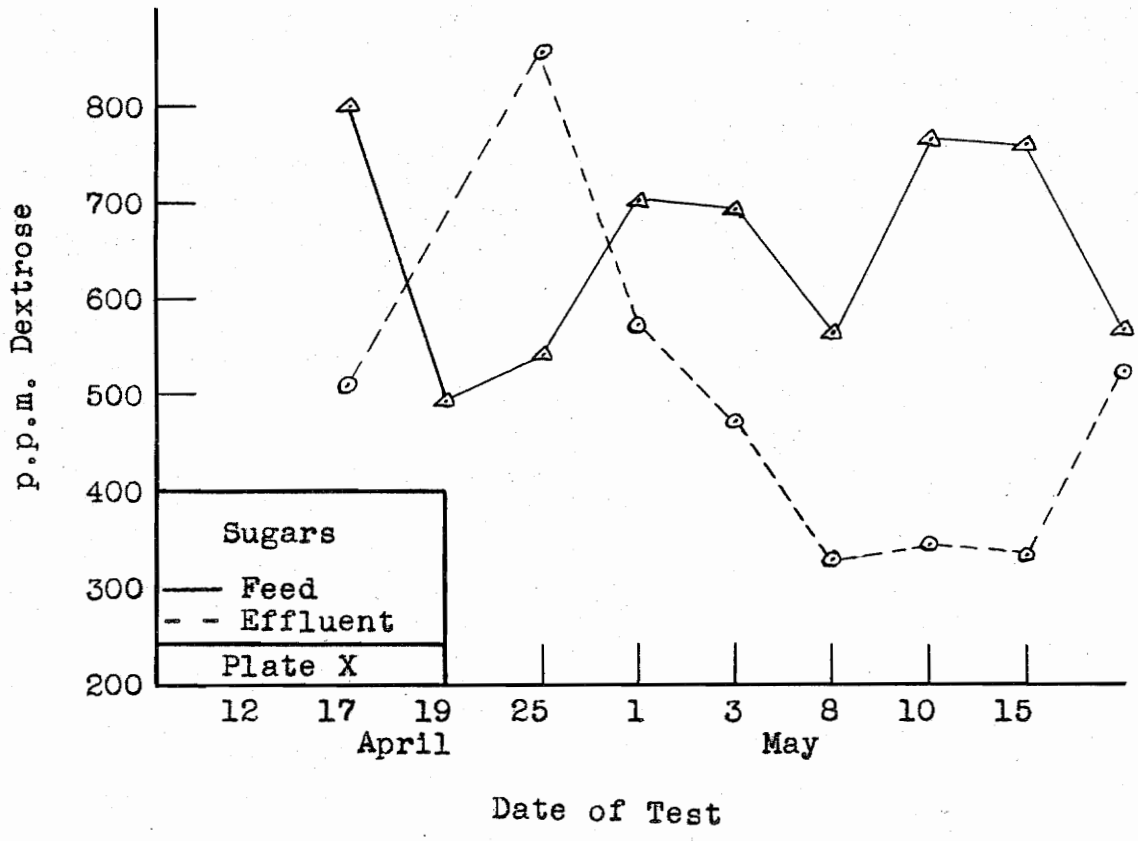
Results of Sugars Tests (ppm Dextrose)

Date	Influent	Effluent
April 17	800	514
April 19	494	-
April 25	544	858
May 1	706	576
May 3	696	472
May 8	564	330
May 10	769	348
May 15	760	334
May 17	570	526

Discussion of Results

The sugars in the effluent were high on April 25 but this is probably due to the digester being reseeded on the 23rd of April. Since then the sugars have always been less in the effluent than in the feed. A reduction in sugars indicates they are being decomposed by the bacteria and probably broken down into acetic and formic acids.


Phosphates
Feed
Effluent
Plate IX
1500
1400
1300
1200
1100
1000
900
p.p.m. PO_4
12
17
19
25
1
3
8
10
15
April
May
Date of Test


800
700
600
500
400
300
200
p.p.m. Dextrose
Sugars
Feed
Effluent
Plate X
12
17
19
25
1
3
8
10
15
April
May
Date of Test

Biochemical Oxygen Demand

General

The biochemical oxygen demand (B.O.D.) determination is a measure of the oxygen required to oxidize the organic matter in a sample, through the agency of microscopic organisms (33). Phelps (34) states that it represents the oxygen that will be demanded by materials in the course of its complete oxidation biochemically. He further states that it is determined by the availability of the material as a bacterial food and by the amount of oxygen utilized by the bacteria during its oxidation. The organic substance must not only be a bacterial food, but must also be a complete food or be combined with other essential food requirements. Various industrial wastes have exceedingly high food value of such limited character as to make them unavailable without some supplementary material. It has been found that dilution water for paper mill wastes required the addition of mineral salts which did not exert any direct oxygen demand themselves. The mineral salts were essential accessory food requirements.

The data obtained from this test are given in table XII and are plotted in their entirety on plate XI. The same data are plotted on plates XII through XV. The gas production per day is plotted on the above mentioned

plates in order that a relation, if one exists, may be detected between the gas production and biochemical oxygen demand (B.O.D.).

Discussion of Results

The biochemical oxygen demand (B.O.D.) of the effluent was on many occasions higher than that of the digester influent. This is contrary to what one would expect to find, but is probably due to the more complex material being broken down. This material has a higher biochemical oxygen demand (B.O.D.) once it is broken down to less complex, more available form than it had in the original form. The organisms decomposed the material that appealed to them, and in so doing released other less complex materials which evidently had a greater biochemical oxygen demand (B.O.D.). This new material, being waste products of these organisms, remains without being further decomposed in the digester.

Assuming the above interpretation is correct, the author believes that some type of organism will thrive on this undecomposed matter. If this organism is anaerobic, it will in time find its way to this material, thus reducing the biochemical oxygen demand (B.O.D.). If the organism is aerobic, additional treatment of the waste will be necessary.

TABLE XII

Data on Digestion of Mixed Hardwood Waste

Date	INFLUENT BOD ppm	INFLUENT lb BOD / cu ft, day	INFLUENT Liters Waste / Day	GAS PROD. cu ft, day	EFFLUENT BOD ppm	Remarks
1951 March 7	-	-	11	.485	-	
8	-	-	11	.573	-	
9-11	-	-	-	-	-	
12	-	-	11	.550	-	
13	-	-	11	.442	-	
14-15	-	-	-	-	-	
16	-	-	11	.378	-	
17-19	-	-	-	-	-	
20	-	-	11	.701	-	
21	-	-	11	.570	-	
22	2,700	.0405	11	.568	2,377	
23	1,858	.0278	11	.579	1,669	
24-26	-	-	-	-	-	Pumping Air

TABLE XII

Data on Digestion of Mixed Hardwood Waste (Continued)

Date	INFLUENT BOD ppm	INFLUENT lb BOD / cu ft, day	INFLUENT Liters Waste / Day	GAS PROD. cu ft, day	EFFLUENT BOD ppm	Remarks
1951 March 27	1,693	.0339	14.62	.103	1,630	
28	-	-	-	-	1,464	
29	-	-	-	-	1,990	
30	-	-	-	-	1,936	
31	-	-	-	-	-	
1951 April 1	-	-	-	.153	-	
2	2,180	.0327	11	.205	1,455	
3-4	-	-	-	-	-	
5	-	-	-	-	-	Pumping Air
11	-	-	-	-	-	Reseeded
12	3,000	.0449	9.12	1.073	-	

TABLE XII

Data on Digestion of Mixed Hardwood Waste (Continued)

Date 1951 April	INFLUENT BOD ppm	INFLUENT lb BOD / cu ft, day	INFLUENT Liters Waste / Day	GAS PROD. cu ft, day	EFFLUENT BOD ppm	Remarks
13	3,305	.0494	9.12	.658	1,410	
14	-	-	9.12	.752	1,359	
15	-	-	-	-	-	
16	-	-	9.12	.159	1,708	
17	2,525	.0377	9.12	5.349	1,610	Pumping Air
18	-	-	9.12	.086	-	
19	2,930	.0437	9.12	.042	-	Reseeded
20	-	-	-	.145	-	
21-22	-	-	-	-	2,593	Reseeded
23	-	-	9.12	-	-	Reseeded
24	-	-	-	.710	-	
25	1,476	.0221	9.12	.376	-	

TABLE XII

Data on Digestion of Mixed Hardwood Waste (Continued)

Date	INFLUENT BOD ppm	INFLUENT lb BOD / cu ft, day	INFLUENT Liters Waste / Day	GAS PROD. cu ft, day	EFFLUENT BOD ppm	Remarks
1951 April						
26	-	-	9.12	.365	296	
27	-	-	9.12	.264	1,186	
28	-	-	9.12	.209	1,696	
29	-	-	-	.172	-	
30	-	-	9.12	.168	1,729	
1951 May						
1	3,068	.0461	9.12	.172	2,305	
2	-	-	9.12	.180	2,260	
3	3,075	.0461	9.12	.161	2,398	
4	-	-	9.12	.211	3,140	
5	2,630	.0394	9.12	.202	2,537	
6	-	-	-	.214	3,343	

TABLE XII

Data on Digestion of Mixed Hardwood Waste (Continued)

Date	INFLUENT BOD ppm	INFLUENT lb BOD / cu ft, day	INFLUENT Liters Waste / Day	GAS PROD. cu ft, day	EFFLUENT BOD ppm	Remarks
1951 May						
7	-	-	9.12	.147	2,737	Increased Loading
8	1,847	.0276	9.12	.184	2,007	
9	-	-	9.12	.218	2,892	
10	2,783	.0416	9.12	.219	3,407	
11	-	-	9.12	.215	2,893	
12	-	-	9.12	.222	3,990	
13	-	-	-	.219	2,990	
14	3,993	.0600	9.12	.278	2,677	Increased Loading
15	-	-	9.12	.315	-	
16	-	-	9.12	.250	-	
17	-	-	9.12	.259	-	

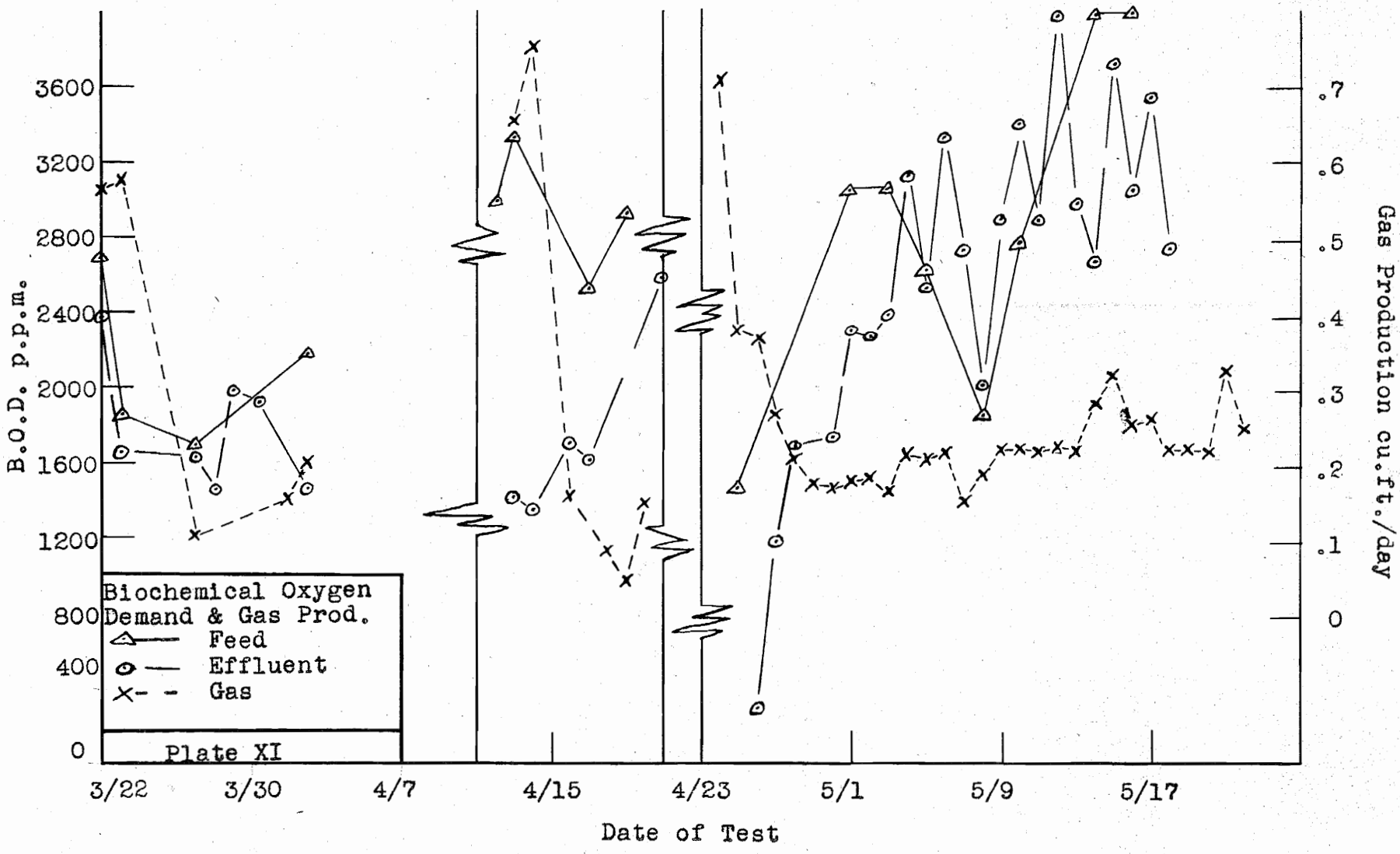
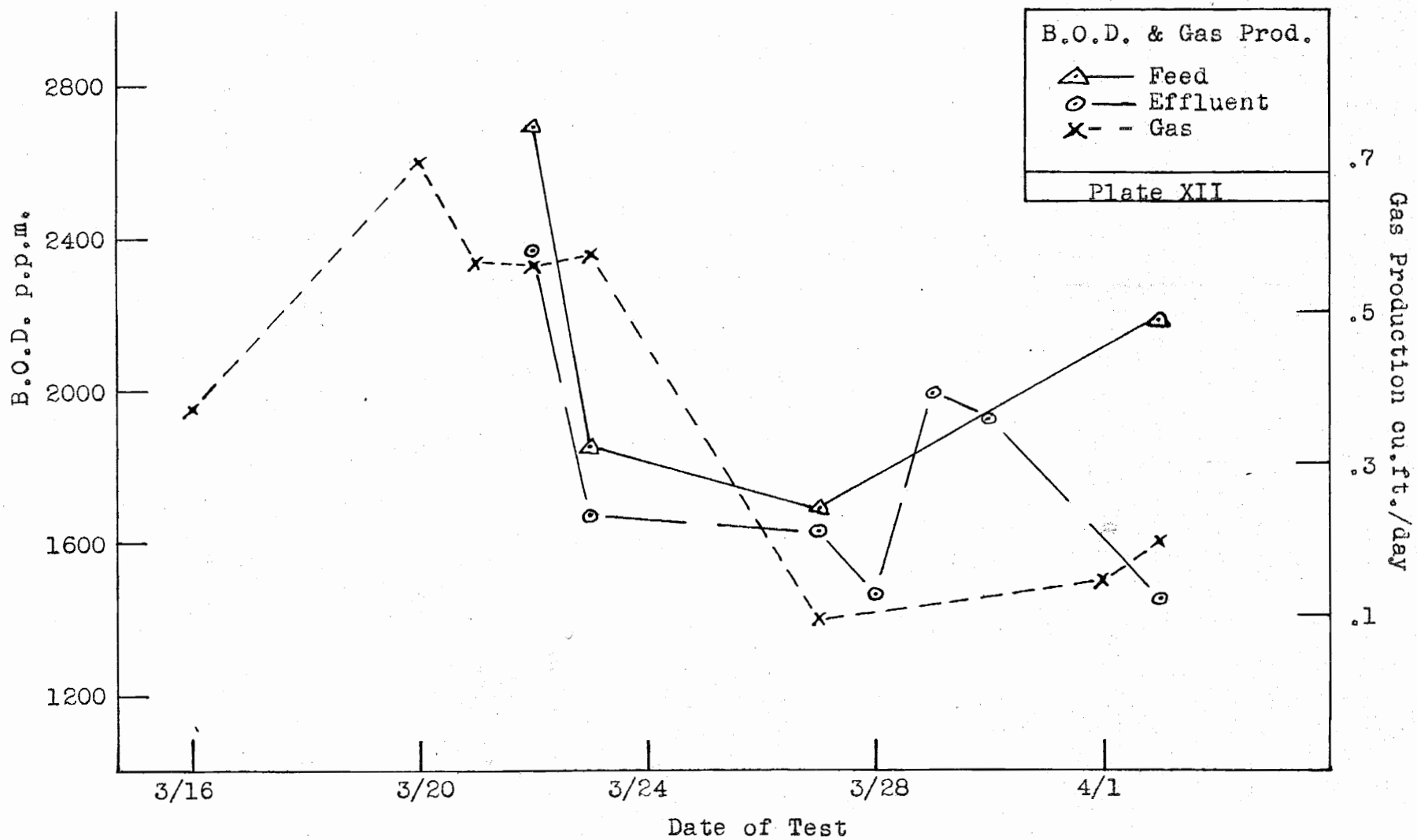
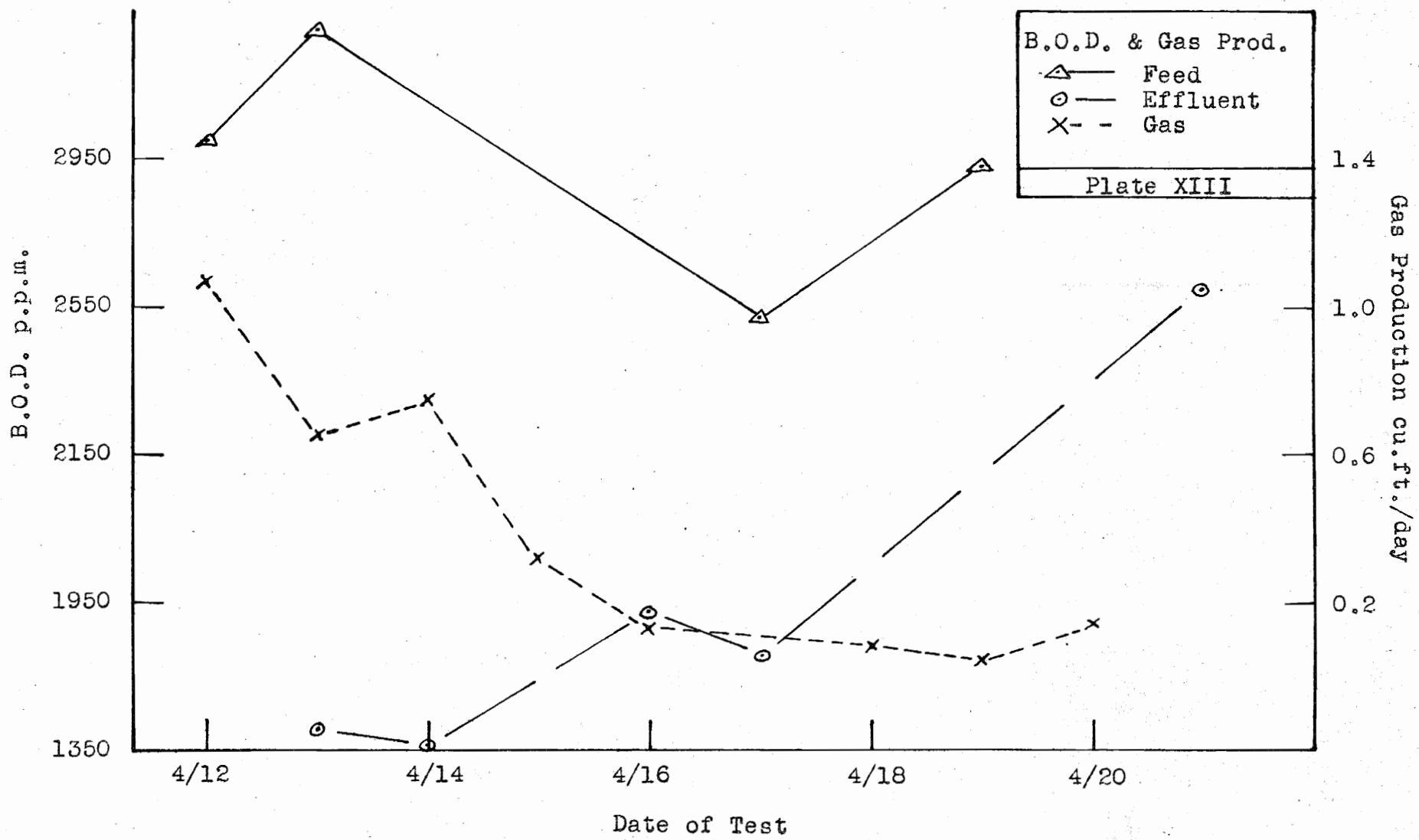

B.O.D. p.p.m.
3600
3200
2800
2400
2000
1600
1200
800
400
0
Gas Production cu.ft./day
.7
.6
.5
.4
.3
.2
.1
0
Biochemical Oxygen Demand & Gas Prod.
Feed
Effluent
Gas
3/22
3/30
4/7
4/15
4/23
5/1
5/9
5/17
Date of Test

Plate XI


B.O.D. & Gas Prod.
Feed
Effluent
Gas
Plate XII
B.O.D. p.p.m.
2800
2400
2000
1600
1200
Gas Production cu.ft./day
.7
.5
.3
.1
3/16
3/20
3/24
3/28
4/1
Date of Test


B.O.D. & Gas Prod.
Feed
Effluent
Gas
Plate XIII
B.O.D. p.p.m.
2950
2550
2150
1950
1350
Gas Production cu.ft./day
1.4
1.0
0.6
0.2
4/12
4/14
4/16
4/18
4/20
Date of Test

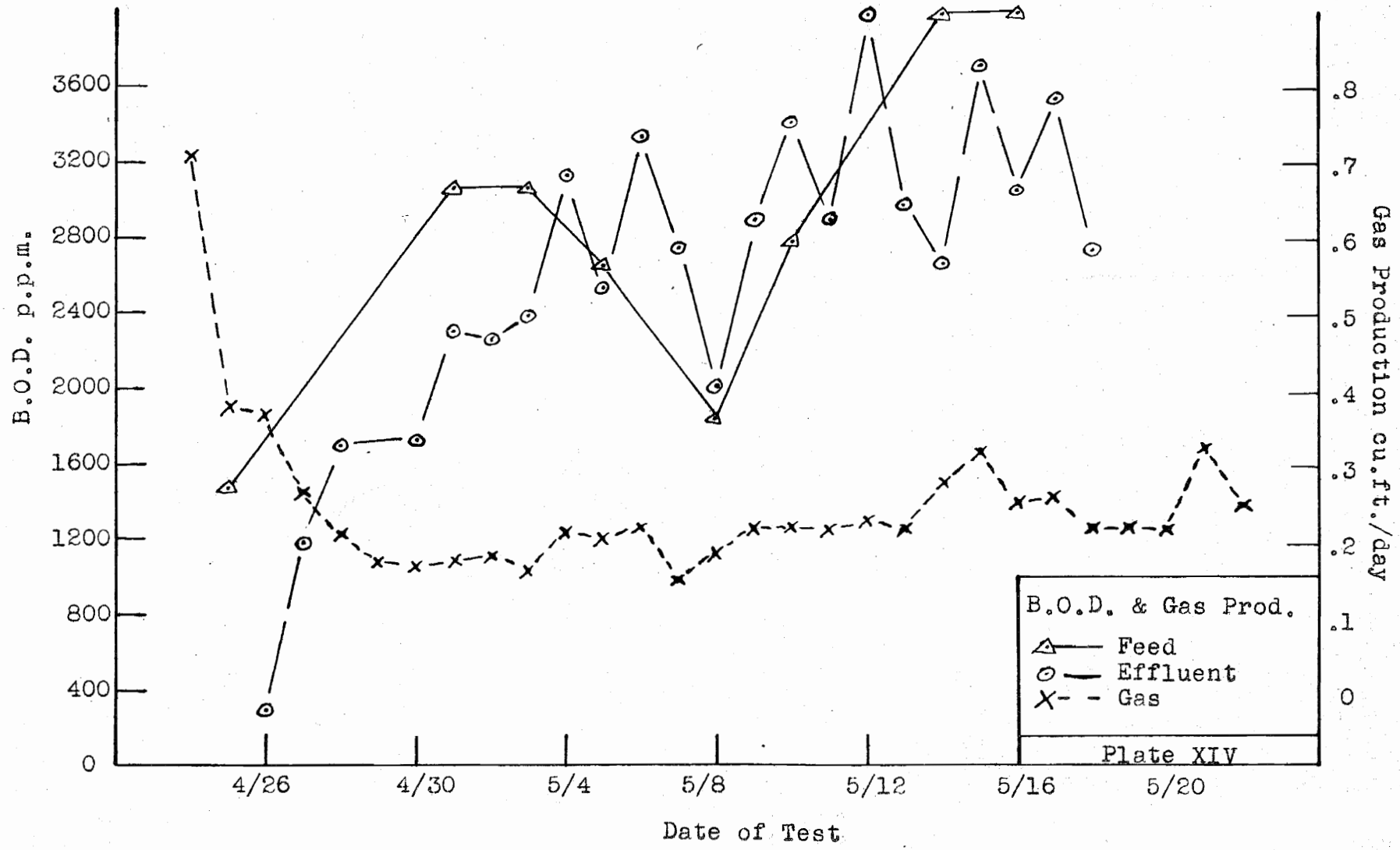

B.O.D. p.p.m.
3600
3200
2800
2400
2000
1600
1200
800
400
0
Gas Production cu.ft./day
.8
.7
.6
.5
.4
.3
.2
.1
0
4/26
4/30
5/4
5/8
5/12
5/16
5/20
Date of Test
B.O.D. & Gas Prod.
Feed
Effluent
Gas

Plate XIV

V. CONCLUSIONS

The conclusions resulting from the investigation of the digestion of mixed hardwood wastes at 57° C are presented in the following paragraphs.

1. At loadings of 0.0221 to 0.0600 pounds of biochemical oxygen demand (B.O.D.) per cubic foot per day the chemical tests indicated good biological activity.

2. Agitation appears advantageous in the digestion of mixed hardwood waste.

3. Average reduction in biochemical oxygen demand (B.O.D.) was 10.4 per cent.

4. The high gas production (46.44 cubic feet) per pound of biochemical oxygen demand (B.O.D.) destroyed, indicates material is being decomposed readily which does not have a very high biochemical oxygen demand (B.O.D.).

5. Digestion under the conditions of this investigation is not beneficial for an adequate reduction in the biochemical oxygen demand (B.O.D.) of mixed hardwood wastes.

6. The slight reduction (10.4 per cent) in biochemical oxygen demand (B.O.D.), indicates secondary treatment is necessary.

VI. SUMMARY

The purpose of this investigation was to determine if high loadings of semi-chemical pulp mill waste could be processed in a pilot plant by anaerobic digestion. The waste was obtained from the Lynchburg, Virginia, plant of the Mead Corporation, where mixed hardwood was being cooked. A neutral sulfite cooking liquor was used in the plant.

The waste was anaerobically digested in a 15-gallon tank placed in a 55-gallon water bath with the temperature maintained at approximately 37° C. The digester contents were recirculated one minute every hour. Ammonium phosphate and ammonium hydroxide were added to the feed to give nitrogen to carbon and phosphorous to carbon ratios of one to twenty.

The digester was seeded with well digested sludge from an anaerobic digester processing domestic sewage. After seeding, portions of diluted waste were added to the digester daily. The concentration of the diluted waste was increased periodically. Since the biochemical oxygen demand (B.O.D.) of the waste varied daily a constant loading could not be maintained. In future investigation, it it suggested the ratio of oxygen consumed (O.C.) to biochemical oxygen demand (B.O.D.) be determined, and used to maintain constant loadings.

On several occasions the packing in the centrifugal pump became worn and air was pumped into the digester. In order to restore bacterial activity in the digester, it was necessary to reseed with sludge.

Chemical tests performed on the digester feed and the digester effluent, showed the digester to be biologically active between loadings of 0.0221 and 0.600 pounds of biochemical oxygen demand (B.O.D.) per cubic foot per day.

The biochemical oxygen demand (B.O.D.) of the feed varied from 1,847 parts per million (p.p.m.) to 3,993 parts per million (p.p.m.), with an average of 2,899 parts per million (p.p.m.).

The biochemical oxygen demand (B.O.D.) of the effluent varied from 1,186 parts per million (p.p.m.) to 3,990 parts per million (p.p.m.), with an average of 2,599 parts per million (p.p.m.).

The average pounds of biochemical oxygen demand (B.O.D.) destroyed during the reliable testing period was 0.0045 pounds per day.

The average gas production for the period was 0.209 cubic feet per day.

The biochemical oxygen demand (B.O.D.) of the effluent showed very little reduction (10.4 per cent) over the reliable period of study.

The gas production per pound of biochemical oxygen

demand (B.O.D.) destroyed was very high (46.44 cubic feet), indicating material was being decomposed readily which did not have a very high biochemical oxygen demand (B.O.D.).

Agitation of the digester contents appears advantageous in the digestion of mixed hardwood waste.

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IX. VITA

Mr. Joseph Charles Hild was born in Baltimore, Maryland, on February 21, 1923. After attending elementary school in Baltimore and Newport News, Virginia, the author graduated from Holy Trinity High School, Norfolk, Virginia, in June, 1941.

In September, 1941, the author enrolled at Virginia Polytechnic Institute. In December, 1942, the author enlisted in the United States Army Air Force, where he studied navigation. After completing his training, he was sent to England to navigate with the Eighth Air Force, the destroyers of Germany. After completing the number of flights required by the Army Air Force rotation plan, the author was returned to the United States in January, 1945. After returning, he served as Transportation Officer at Langley Field, Virginia.

In January, 1947, the author was separated from the service, and accepted employment with the Crown Fastener Corporation, Newport News, Virginia.

In September, 1947, the author returned to Virginia Polytechnic Institute where he received a B. S. degree in Civil Engineering in June, 1950. After graduation he continued his studies and became a candidate for the M. S. degree in Sanitary Engineering.