THE TREATMENT OF LIQUID WASTES
FROM THE CANE SUGAR INDUSTRY IN PUERTO RICO

Project A-033-PR

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ABSTRACT

This study was undertaken to determine the applicability of several waste treatment processes to the purification of the waste waters generated by the cane sugar industry in Puerto Rico. The specific processes considered were anaerobic digestion, activated sludge treatment, trickling filtration, oxidation using mechanically supplied air, and adsorption in activated charcoal. Pilot plant units which allowed determining the changes in efficiency brought about by changes in the variables of operation were constructed for each of the processes considered. Numerous runs were made in each of them, using as raw material waste waters brought into the laboratory from different sugar mills operating in the Western part of Puerto Rico.

As a first step in the study the physical and chemical parameters characterizing the waste were evaluated. The actual volumes of effluents discharged by the mills were also determined.

The results obtained show that cane sugar waste waters are amenable to treatment by each of the processes considered. In each of them efficiencies of the same order of magnitude as those corresponding to the industrial application of the same processes to other wastes were obtained. The reductions in pollutional load attained were in the order of 50 per cent for anaerobic digestion, 80 per cent for activated sludge treatment, 75 per cent for trickling filtration, 50 per cent for mechanical aeration methods, and 50 per cent for treatment with activated charcoal. Correlations were made which allow estimating the efficiency of each of these processes as a function of physical parameters of operation.
The pollutional load of waste waters from the cane sugar industry may therefore be significantly reduced by using the treatment processes evaluated in this study.
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THE TREATMENT OF LIQUID WASTES FROM
THE CANE SUGAR INDUSTRY

Introduction:

The disposal of untreated waste waters from cane sugar mills is the major environmental problem which this industry faces in Puerto Rico at present. About 200 million gallons of raw liquid wastes from sugar mills are discharged daily into the island’s streams and marine bodies during the cane grinding season, the pollutional load of these discharges having been estimated to be equivalent to that from a population of one million people (6).

There are now twelve sugar factories operating in the island. During the 1966-67 milling season there were 22 factories operating as compared to 40 in 1940-41. As may be seen, the sugar cane industry in Puerto Rico is no longer a dynamic one but rather an ailing manufacturing activity. The main factors that account for the financial difficulties of the industry are the following:

a) High production costs
b) Low sugar yield
c) Gradual disappearance of the "colonos", or small plantation’s owners
d) Gradual reduction of land availability
e) Labor problems

Up to a few years ago the major source of waste waters in the sugar mills of Puerto Rico was water used mainly for cooling purposes in evaporators, heat exchangers, and vacuum pans. Entrainment and washings from these equipments accounted for the organic matter present in the wastes. In most of the mills the cooling water was used in a once-through operation.
In recent years, to help solve its economic problems, most sugar mills have shifted, either in full or partially, to mechanical harvesting procedures. As a result the sugar cane brought into the mills contains much higher amounts of soil, leaves, debris, dirt, and miscellaneous organic substances than when hand-harvesting was used. The cooling water is now additionally used to wash the cane as it enters the process, the end result therefore being that the wastewaters generated at present carry a higher concentration of suspended and dissolved solids than they did in the past. The fraction of the total volume of cooling water which is additionally used in a mill for washing the incoming cane depends, of course, on the fraction of the total amount of the cane being processed which has been mechanically harvested. Normally, if the cane is entirely a mechanically harvested one, the water used for cooling also suffices for washing. No additional water needs are therefore imposed by mechanical harvesting and the subsequent washing requirements which it imposes.

This study was directed at determining the applicability of the following waste treatment processes to liquid sugar mill effluents:

a) Anaerobic digestion
b) Activated sludge treatment
c) Trickling filtration
d) Oxidation by direct aeration

As the experimental work was carried out it was considered desirable to extend its scope to include treatment by adsorption in activated charcoal. This would be mainly a tertiary treatment operation which, if used by industry, would process wastes which would have been previously treated by one of the four secondary treatment methods first mentioned.
Anaerobic digestion, activated sludge treatment, and trickling filtration are processes well established in the treatment of both sanitary and organic industrial wastes. They are essentially biological decomposition processes which require that bacteria feed on the organic matter of the wastes to convert it to gaseous products of assimilation.

In anaerobic digestion the dissolved organic solids are transformed into methane, carbon dioxide, small amounts of other gases, and stabilized residue through the action of bacteria which perform their metabolic processes in the absence of oxygen. In trickling filtration and in activated sludge treatment the microbial population utilizes oxygen dissolved in the wastewaters to oxidize the dissolved organic matter to carbon dioxide and water, and leaving also a solid residue which does not yield to additional biological degradation.

Oxidation by direct aeration may be carried out in a wide variety of modes and equipments. In essence the process involves adding air to the wastewaters being treated, by means of mechanical devices. The process may be carried out in the presence of aerobic bacteria or of algae to enhance its efficiency, or alternately biological decomposition of the dissolved organics may not be pursued in which case direct molecular oxidation would occur. Lagoons and tanks are usually employed in this type of process (9).
Anaerobic Biological Treatment Process:

1. General Theory:

Anaerobic decomposition of dissolved organic matter is effected by bacteria which utilize the oxygen present in the organic matter properly to decompose it. The presence of dissolved molecular oxygen is not required for the process, and, as a matter of fact, were it present anaerobic organisms would become extinct.

From a kinetic viewpoint anaerobic treatment may be described as three-step process involving: (a) the hydrolysis of complex organic substances, (b) the production of acids, and, (c) the fermentation of the organic acids into gaseous products, mainly methane and carbon dioxide. In the first step, complex organics are converted to less complex organic materials by enzymatic hydrolysis. In the second step, these hydrolysis products are fermented to simple organic compounds, predominantly volatile fatty acids, by the so called "acid forming bacteria". In the third step the simple organic compounds are fermented to methane and carbon dioxide by a group of strictly anaerobic bacteria called the "methane formers" (I).

Suitable measures of the amount of decomposable, or organic matter undergoing anaerobic decomposition in a given time, are (1) the volume and rate of gas production and (2) the reduction in weight of volatile organic material and its rate of loss. Gas production from a single batch of organic material traces an s-shaped curve as a function of time. This means that from the beginning of the process to a point in time near the halfway mark of total gas evolution, the yield of gas in a unit of time becomes progressively greater. After this point has been reached, the yield becomes constantly less and a limiting value is gradually approached. This behavior of organic materials that are under-
going anaerobic digestion is characteristic of processes in which the accumulation of enzymes plays a part. A lag period is usually observed before the onset of active anaerobic decomposition in processes utilizing this mode of treatment (11).

If anaerobic decomposition is interpreted as a first order reaction, the gasification curve may be formulated as a first order reaction that is catalyzed by the products of the reaction. As an approximation the following expression has been suggested (10):

\[
\frac{dy}{dt} = k (G - y)
\]

where \(G\) = total amount of gas generated

\(y\) = amount of gas produced in time \(t\)

The evaluation of the magnitude of the constant offers some difficulty, and its ranges have not as yet been sufficiently well established to be of practical service to the engineer (13). Simpon determined \(k\) to be 0.3/day for several digesting mixtures in which sludge was removed from active digestion and permitted to batch digest to completion (30). Fair and Moore found \(k\) to be 0.168/day at 95°F in digestion without mixing (13). Shulze determined \(k\) to be 0.14/day at 92°F, while Grune et al determined it to be 0.25/day at 90°F in digesting sewage sludge (14). It should be mentioned that \(G\), the total amount of gas generated, can not be determined exactly, since an infinite time would be required.

The gas produced from the digestion of sewage sludge and similar organic mixtures is composed primarily of \(CO_2\) and \(CH_4\), with small quantities of \(NH_3\), \(H_2S\), \(H_2\), \(N_2\), and \(O_2\) present. Composition ranges from 25-35 percent \(CO_2\) and 65-75% \(CH_4\), and the amount is in the order of 15 to 20 standard cubic feet per pound of volatile matter destroyed for the case of digesting raw sludge.
2. Previous Work on the Anaerobic Treatment of Cane Sugar Wastes:

Very little work is reported in the literature on the biological treatment of cane sugar wastes; industrial practice at present is not utilizing this scheme at all (33). Pollution control in the case of the filter muds ("cachaza") is usually achieved by using it as a fertilizer in the cane fields, but all other liquid effluent streams from the mills are now discarded in most installations into receiving bodies of water with no treatment whatsoever. This includes the water used for washing the cane, the condenser waters, acidic and caustic wastes, and floor-wash waters. In a few mills the practice of impounding for a short period of time to remove suspended solids is followed (33).

Bhaskaraw and Chakrabarty conducted recently an anaerobic treatment study on wastes from the cane sugar industry (5). The study was made using a pilot plant unit located in a mill which was under operation. The unit was made up of two digestion ponds having a total volume of 1100 cubic feet and a depth of 4 feet. The anaerobic ponds were operated in two stages having 1 and 6 days detention periods respectively, or at other detentions. The authors report BOD reductions from 60 to 70% for detention periods from 2 to 7 days. The loadings used were in the order of 0.02 lbs. BOD per day per cubic foot for operation at room temperature (30°C), and of 0.04 lbs. BOD per day per cubic foot for operation at 37°C, without any significant changes in efficiency.

Bevan reports the status of the Australian cane sugar industry as a source of pollution (3). Mention is made of mills treating their wastes in aerated lagoons and in facultative ponds operated on a rotational basis, but no operational data are presented in the report.

Biaggi had previously reported the pollutional load resulting from the operation of sugar mills in Puerto Rico, but although significant data on the amounts involved are
presented, no methods of treatment were defined as followed in practice (6). This, of

course, was due to the fact that no treatment processes other than brief lagooning were

followed in any of the mills in the island, as previously informed by Guzmán (15).

3. Experimental Procedures Followed in Anaerobic Treatment:

A. Characterization of wastes:

An essential part of this study on the treatment of the liquid wastes from the
cane sugar industry dealt with the characterization of the raw wastes and of the treated
products. All pertinent physical, chemical, and biological parameters were measured
using the equipments and procedures described in the corresponding standard procedures
(1). The proper characterization of the substances involved is an important design need.

In this respect a most significant point should be brought out. It concerns the
change in the properties of the wastes from the sugar industry which occurs as mechaniza-
tion of the harvesting operations is increased. The change is brought about by the
need to wash the cane effectively before it is ground, since the amount of foreign ma-
terial which it contains is larger in the case of mechanical harvesting than in that of
hand harvesting.

Waste streams from a sugar mill may be considered to fall into four major groups.
As previously mentioned, these are cane-wash waters, condenser waters, acidic and
caustic wastes, and floor-wash waters. The usual practice in Puerto Rico is to mix
the last three into a single effluent which may then be either rejected or used for
washing the incoming cane and then discarded. Depending on the mill considered,
the effluent wastes may be from one to four different streams, of characteristics which
will depend on its constituent wash waters. Cane wash water contributes to pollution
in a larger degree than do the other sources, since it carries large amounts of suspended and dissolved solids.

In conclusion, the wastes from sugar mills vary in composition from mill to mill depending on the uses and functions which the water has served, on the scheme used to dispose of it, and on the degree of mechanization of the harvesting operation carried out.

In studying the anaerobic decomposition of liquid wastes from the cane sugar industry, four different mills were considered in this project. These were Centrales Igualdad, Guánica, Eureka, and Coloso. Some of these companies did not carry out mechanical harvesting in 1972 whereas others did. Thus a rather wide fluctuation in the properties of the waste waters resulted from the laboratory measurements and determinations made.

Table 1 summarizes the ranges of values observed and includes, for purposes of comparison, data which had been reported in the literature at a time when mechanical harvesting was not practiced in Puerto Rico at all (6). As may be seen, significant differences are observed between the various data.
<table>
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<td>pH</td>
<td>5.3-8.8</td>
<td>6.8</td>
</tr>
<tr>
<td>BOD₅, mg/liter</td>
<td>112-225</td>
<td>180</td>
</tr>
<tr>
<td>COD, dichromate method, mg/liter</td>
<td>385-978</td>
<td>591</td>
</tr>
<tr>
<td>Total solids, mg/liter</td>
<td>500-1400</td>
<td>740</td>
</tr>
<tr>
<td>Suspended solids, mg/liter</td>
<td>100-700</td>
<td>375</td>
</tr>
<tr>
<td>Volatile solids, mg/liter</td>
<td>244-805</td>
<td>494</td>
</tr>
<tr>
<td>NH₃ nitrogen, mg/liter</td>
<td>2.2-9.2</td>
<td>4.4</td>
</tr>
<tr>
<td>Dissolved oxygen, mg/liter</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Temperature, °C</td>
<td>31-49</td>
<td>45°</td>
</tr>
<tr>
<td>Phosphates, ppm</td>
<td>0.19-C.38</td>
<td>0.47</td>
</tr>
</tbody>
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The total volume of liquid wastes discharged from a mill may also be a function of the mode of operation followed in harvesting, since the amount of water required to wash the cane which has been mechanically harvested may be much larger than that needed for cleaning hand-harvested cane; this depending on the operating scheme used. Reliable data on the amounts involved in one case or the other are either unavailable or very difficult to obtain in the field. Most data available correspond to the time when mechanical harvesting was not done at all (6), the literature being totally devoid of information on the amounts actually used in Puerto Rican mills for washing mechanically harvested cane.

Two typical mills will be used as an illustration of the various trends now found in practice. The water requirements for sugar mills in Puerto Rico are 2 gallons per minute for each ton of cane processed per day. This was water used mainly for cooling purposes but since the advent of mechanical harvesting this same water is used for washing the cane before it is finally rejected. Central Plata, located in the town of San Sebastián, processes as raw material cane harvested by both mechanical and hand-cutting methods. About 90% of the total cane processed is hand-harvested and 10% is mechanically cut. However, about one third of the water used for cooling purposes is further used to wash the cane which has been mechanically harvested, the remaining two thirds being rejected directly as waste. Central Coloso, located in the town of Aguada, processes cane from mechanical harvesting operations almost entirely. This mill still uses a total amount of water of 2 gpm. per day per ton, but the cooling water is totally used and is enough for proper washing of the cane (2).

On this basis it may be stated that the amounts of waste waters generated in
This process mechanically harvested cane are at present the same as those which use hand-harvested cane. However, the pollution loads of the two types of waste waters differ considerably as shown previously in Table 1.

3. Experimental Procedures:

Figure 1 shows the experimental anaerobic digestion unit used in this study. It consisted essentially of a 5 gallon polyethylene bottle used as digester, with provisions for gravity—feeding—and—withdrawal of the wastes at rates such that the liquid level in the bottle remained constant at all times. The digester could therefore be operated at varying detention times by merely changing the wastes feed rate and maintaining the discharge rate constant and equal to the feed rate. The amount of gas generated was measured using a wet test gas meter, and its composition determined by Orsat analyses. The digester was maintained completely mixed at all times by pumping back into it a fraction of the gas generated by the digestion process. A peristaltic varying-volume pump was used for this purpose. All runs were made in a room kept at a constant temperature of 25°C.

During actual operation fresh waste water from the mill being considered was brought in daily to the laboratory, the source and the effluent stream from which it was drawn being maintained unchanged throughout the duration of each run (10 to 15 days). The feed bottle was filled with this waste water, and used to supply the digester until the next day when it was substituted by the new waste water brought in fresh from the mill. The unit was allowed to run uninterrupted until steady state was reached, this being established by a constant rate of gas generation. At this time 6-hour composite samples of both the fresh feed and the effluent stream were taken and analyzed, the gas
Flow rate was measured, and a composite sample of the gas was analyzed.

In each run a one-liter volume of seed was initially placed in the digester to provide an active population of anaerobic bacteria to help minimize the induction period required for decomposition to occur. The seed was prepared at the start of the project by filling a five-gallon plastic bottle with waste water from a sugar mill and adding to it one liter of sewage from a digester in a municipal sewage treatment plant. Air was totally excluded by sealing the bottle with a tight-sealing stopper with two holes drilled in it. Short pieces of glass tubing were inserted in the holes, and these had one foot lengths of rubber tubing connected to them and leading to small beakers full of water. This arrangement provided a way for the gaseous products of decomposition to leave the bottle while at the same time preventing air from reaching the liquid in the bottle. Each and every day during the duration of the study one liter of the material in the seed bottle was withdrawn and rejected, while at the same time one liter of fresh waste water was added to occupy the volume corresponding to the amount drawn out. The bottle was shaken at frequent intervals to mix its contents as thoroughly as possible.

Runs were made using detention periods up to seven days and as short as two hours, these corresponding to maximum loadings in the order of 0.04 lbs. of volatile solids per day cubic feet of digester volume.

4. Results:

The data gathered in the study on anaerobic treatment appear summarized in tables 2, 3, and 4, pages 91, 92 and 93 of the appendix. Figures 2, 3, 4, 5, and 6 additionally illustrate the significant data in graphical form.

The results shown represent runs made using waste waters which had not been
...ed for washing cane as well as others which had been first used for cooling purposes and subsequently employed to wash the cane reaching the mill. In some cases the results for the two types of wastes could be integrated into a single graphical expression whereas in others this could not be done. A total of fifteen runs were made using as feed in occasions water which had been used for washing the cane, and in others waste water coming directly from the condensers and other heat exchange units. As previously mentioned, the duration of each run was from 10 to 15 days, due allowance thus provided for the system to reach steady state in each of the detention times considered.

Table 2 shows the measurements made on the rate of generation of gases from waste waters which had been used to wash cane. The data appear plotted in Figures 2 and 3. As seen in them, the rate at which gas is produced increases with time for digestion periods up to about 4 days, and then declines to nearly no change at all for 7 day digestion periods. The BOD values corresponding to the waste waters which yielded these gas generation data were in the upper band of the values reported in Table 1, and ranged from 160 to 225 ppm. The gas generation rate was not therefore a function of BOD contents within the range of values considered. The average gas composition for all the runs made was 4% carbon dioxide and 60% methane.

A similar trend is observed in the change in total acids contents occurring during digestion. These data are tabulated in Table 3 and appear graphically in Figure 4. The acidity is seen to increase percentagewise much more in the case of waste water from the cane washing operation than in that of waste water from the manufacturing operation. At the end of 7 days of digestion the cane washing wastes contained an average acidity of 450 mg. Ca CO₃/liter while the process wastes had a maximum acidity of 290 mg. Ca CO₃/liter.
Figure 2: Progress of gas production by gas sugar biotreatement undergoing in-situ coal decomposition.
REDUCTION IN CONCENTRATION OF SUSPENDED SOLIDS DURING ANAEROBIC DIGESTION

Reduction in Concentration of Suspended Solids

Digestion Time, Days

MANUFACTURING WASTEWATER

JULIAN'S WASHING WASTEWATER
The data plotted in Figure 5 show that a larger decrease in concentration of suspended solids occurs during anaerobic digestion of waste water from the cane washing operation than in that of waste waters from the manufacturing operation. Prior to digestion the concentration of suspended solids in the first type of waste water is in the order of 500 to 700 ppm, while in the second type it ranges from about 50 to 100 ppm, as previously shown in Table 1. The observed difference between the decreases in suspended solids in one case and in the other is consistent with that found in the increases in acidity. The larger increase in acidity occurring during digestion of the waste waters from the cane washing operation corresponds to a larger increase in the amount of dissolved and suspended solids which disappears through biochemical decomposition processes.

The rate of BOD reduction observed during anaerobic digestion increases rapidly during the first day of digestion and then levels off with time, as shown in Figure 6. At digestion periods of 7 days no significant change in BOD occurred with time.

5. Conclusions:

The anaerobic digestion of sugar cane waste waters produces reductions in their pollutional loads which increase as the periods of digestion increase. Reductions in the order of 50% are attainable for digestion periods of 7 days, this being in agreement with the data report by Bhaskaran et al (5). The percentage reductions in BOD corresponding to different digestion periods are shown in Figure 6.

As anaerobic decomposition occurs the acidity of the waste waters increases, the concentration of suspended solids in them goes down, and the amount of gas generated increases. This is in agreement with the theories and principles applicable
Trickling Filtration:

1. Principles of Operation:

One of the methods used to treat organic waste waters is that of biological oxidation in fixed-bed units. These units are known as trickling filters. They are employed in removing the colloidal and dissolved organic matter present in the waste waters fed to them.

A trickling filter generally consists of a circular vessel containing a bed of suitable depth of crushed stone, crushed slag or other reasonably hard and insoluble media. Conventional filters contain a packing of rocks from 2 1/2 to 4 inches in size and of depth from 3 to 8 feet. Waste water is sprayed over the bed either by fixed nozzles or rotary distributors. An underdrain with suitable ventilation is supplied for proper filter operation. As organic wastes continuously trickle over the bed, a gela- tinous film of micro-organisms develops on the surface of the medium. This slime is composed of microbial cells of different degrees of activity, of organic matter in various stages of decomposition and of organic and inorganic residues produced by the decomposition process.

Biological filters may be broadly designated as either standard (low-rate) or high rate trickling filters. Low rate filters operate with hydraulic loadings of 2 to 6 gal/acre/day, with organic loadings from 1500 to 7500 lb/acre-ft/day and with depths of about 6 ft.

Plastic packings such as Surfaced are employed in depths up to 40 ft, with hydraulic loadings as high as 4.00 gpm/ft².
The mechanism of BOD removal in a trickling filter is similar to that of the aerated sludge process. A large portion of the liquid applied to the surface of a filter passes rapidly through the filter and the remainder trickles slowly over the surface of the slime growth on the rocks. Removal occurs by biosorption and coagulation from that portion of the fluid which passes rapidly through the filter and by progressive removal of soluble constituents from that portion with large residence time. As waste passes through the filter, nutrients and oxygen diffuse into the slime, where assimilation takes place. At the same time, by-products and CO₂ diffuse out into the flowing liquid. As oxygen diffuses into the biological film, it is consumed by microbial respiration, so that a defined depth of aerobic activity is developed. Slime activity below this depth is anaerobic as shown in figure 7 (10).

FIGURE 7

SCHEMATIC REPRESENTATION OF TRICKLING FILTER OPERATION

<table>
<thead>
<tr>
<th>Filter Media</th>
<th>Effective Film Depth</th>
<th>BOD Nutrients</th>
<th>Organic Acids, etc</th>
<th>Anaerobic</th>
<th>Aerobic by-products</th>
<th>Air</th>
</tr>
</thead>
<tbody>
<tr>
<td>H₂S</td>
<td></td>
<td>CO₂</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

BOD removal through a trickling filter is related to the available biological slime surface and to the mean residence time of the waste in the filter. Naturally it is expected that the rate of BOD removal be dependent also upon waste temperature, nutrients availability, oxygen availability and liquid distribution inside the filter.
The mean residence or contact time of the liquid with the filter surface is related to the filter depth, the hydraulic loading, and the nature of the filter packing. A general relationship was developed by W.E. Howland (16) and K.L. Shulze (26):

\[ t = \frac{C D}{Q^n} \]  \hspace{1cm} (2)

where \( t \) = mean detention or contact time
\( D \) = filter depth
\( Q \) = hydraulic loading

The constants \( C \) and the exponent \( n \), incorporate surface and viscosity effects and will vary with the type of filter packing. Theoretical analysis indicates that the exponent, \( n \), will approach 1/3 for turbulent flow and 2/3 for laminar flow as per Howland et al (16).

Dimensional analysis was used by Sinkoff et al (31) to develop a relationship for residence time in a trickling filter:

\[ t = \frac{C D v^a}{g^{1/3}} \left( \frac{S}{Q} \right)^b \]  \hspace{1cm} (3)

in which \( S \) is the specific surface and \( D \) is the filter depth, \( v \) stands for kinematic viscosity, \( g \) is the gravitational constant, and \( C \) is a constant. Exponents \( a \) and \( b \) depend upon the material employed as filter media. The data of Sinkoff et al indicated a variation in mean residence time of 7.5 min. to 0.5 min. over a range of hydraulic loading of 8.9 Mgal/acre/day to 276 Mgal/acre/day. By contrast studies by the Water Pollution Research showed that at low hydraulic loadings (3-5 Mgal/acre/day)
Average residence times of 40 - 60 min, were obtained. The residence time is also increased due to capillary water storage according to the research work of Howland and Shulze (18).

The effect of residence time in a filter on BOD removal has been described by Howland (16) and Shulze (26) and is defined by:

\[
\frac{L_e}{L_0} = e^{-kt}
\]  

(4)

In which \(L_e\) is the BOD remaining in filter effluent, \(L_0\) is the BOD applied to the filter after mixing with recycle, \(t\) is the contact time and \(k\) is a function of the active film per unit volume. The equation is based on the assumption that the process follows first order kinetics. For complex wastes, the equation may require a modification.

The fraction of BOD remaining in solution can also be approximately represented by the relationship:

\[
\frac{L_e}{L_0} = C \cdot Q^n
\]  

(5)

A general relationship can be developed by combining these two equations (4, 5) and applying a modification for slime distribution in the filter:

\[
\frac{L_e}{L_0} = e^{-kD_m/Q^n}
\]  

(6)

Howland and Shulze (16) have shown that \(m = 1.0\) in cases where the film is approximately uniformly distributed through the filter depth. However, for most cases \(m\) is less than unity.
The above equation (6) presumes that all components of the organic waste are removed at the same rate. There is considerable evidence, however, that in complex cases the removal rate decreases with concentration or time since the more easily volatile components will be removed faster. This requires that a retardant form of equation be employed to describe the overall removal process, namely:

$$\frac{L_e}{L_0} = \frac{1}{1 + \frac{C_f Dm}{Qn}}$$

(7)

1. **Experimental Procedures followed:**

The material used for filter packing was rocks of 2 to 3 inches in size. These were obtained from a trickling filter operating with sanitary sewage at the Mayaguez area. The slime already deposited on these rocks was thick and grayish color, these being signs of a "healthy" trickling filter where nutrients are available in adequate amounts.

The filter itself was constructed from a surplus stainless steel vessel from the Chemical Engineering Department's laboratory. The head of the vessel was cut-off and several 3/8 inch holes were drilled around its circumference, at 42 1/2 inches from the top of the vessel. The purpose of these holes was to accommodate the support rods for the filter media. Ventilation was accomplished through twenty three - 1/4 in. holes drilled around the vessel and below the support rods. These ventilation holes were fitted with 2 in. long pieces of plastic tubing to prevent effluent leakage through the holes. Figure 8 shows schematically the configuration of the trickling filter with its corresponding measurements.
The trickling filter system was composed of:

- a 55 gal fresh feed tank,
- a feed pump, (200 volts)
- two rotameters,
- a 55 gal combined feed receiving tank,
- a Plastic distributor,
- the Trickling filter itself,
- a 55 gal effluent receiver,
- two recycle pumps, and a
dual pass - multichamber settler.
The system layout is shown in figure 9, page 29. Photographs showing part
the installation are shown on pages 94, 95, and 96.

In order to acclimate the slime-covered rocks to the new feed, the system was
allowed to operate on a closed loop for some time. The fresh feed (water wastes) from
the sugar mill was dumped into the receiver drum and recirculated through the system
for 24 hrs. After this period part of the spent waste water was discarded to the sewer
and a fresh batch was loaded. Having in mind that sugar mill waste waters are deficient
in nutrients (6) as shown in table 1, page 9, one liter of an aqueous solution of 3.615 gm
\( \text{Cu(NO}_3\text{)}_2 \) and 0.44 gm \( \text{KH}_2 \text{PO}_4 \) was added once a week. This practice was allowed to
continue for about five weeks. After this period the unit had become acclimated and was
ready for continuous operation.

In the continuous operation mode, the fresh process waste water feed was brought
in from the mill and loaded into the fresh feed tank. From there it was pumped through
the rotameter by the feed pump. The desired amount of recycle waste water was pumped
through the other rotameter. Both the fresh feed and the recycle were allowed to mix in
the combined feed receiving drum and drained into the plastic distributor. This distributor
in turn fed the trickling filter uniformly. The filter discharged into a receiving drum from
which the recycle pump was fed, and the remaining effluent travelled on to the multi-
chamber settler.

Perhaps the most pressing problem that was faced in the operation of this unit was
the plugging tendency of the discharge nozzles of the various tanks, of the discharge
hoses of pumps and even of the rotameters. In order to overcome the problem a fine mesh
screen was installed on the receiver drum. The screen was cleaned periodically to keep
it from accumulating large amounts of settleable matter.
The period of continuous operating mode on this filter was limited to 14 hours. The limiting factor was the amount of waste water that could be brought from Igualdad or Mill on one day due to the 8-hr. working day of the campus personnel and the capacity of the vehicle used. However, steady state in a given run was reached rather fast, in all cases in less than an hour. At the conclusion of each run the filter was kept wet at all times to keep active the biological population in the slime.

Results and Findings:

The table that follows summarizes the most important characteristics of the trickling filter used in this research work.

<table>
<thead>
<tr>
<th>TABLE 5</th>
</tr>
</thead>
</table>

TRICKLING FILTER CHARACTERISTICS

| Filter diameter, inches | 13.0 |
| Filter depth, inches | 41.5 |
| Packing material | Rocks, 2-3 in mesh size |
| Filter area, sq. ft. | 0.9216 |
| Filter volume cu. ft. | 3.1974 |
| Environment Temperature, °C | Indoors, 23 °C |

The results of the experiments performed on the trickling filter are presented in tables 6, 7, and 8, on pages 31, 32, and 33 respectively. A total of thirty runs were performed on the filter to cover a wide range of operating conditions. Recycle ratios were varied from 0.0 to 10.0 while hydraulic loadings were maintained on the "high-rate" classification most of the time. Figure 10 shows the filter performance using
<table>
<thead>
<tr>
<th>Feed Rate GPM</th>
<th>Recycle Rate GPM</th>
<th>Recycle Ratio</th>
<th>Combined Feed Rate GPM</th>
<th>Total Hydraulic Rate GPM/FT²</th>
<th>% BOD₅ Reduction</th>
<th>Fresh Feed BOD₅</th>
<th>Filter Inlet BOD₅</th>
<th>% BOD₅ Reduction Based on Fresh Feed</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.2</td>
<td>0.5</td>
<td>2.5</td>
<td>0.7</td>
<td>0.760</td>
<td>13.3</td>
<td>75.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.3</td>
<td>0.6</td>
<td>2.0</td>
<td>0.9</td>
<td>0.977</td>
<td>10.0</td>
<td>185.0</td>
<td>65.0</td>
<td>68.3</td>
</tr>
<tr>
<td>0.3</td>
<td>0.6</td>
<td>2.0</td>
<td>0.9</td>
<td>0.977</td>
<td>0.0</td>
<td>70.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.5</td>
<td>0.5</td>
<td>1.0</td>
<td>1.0</td>
<td>1.085</td>
<td>5.4</td>
<td>65.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.0</td>
<td>0.0</td>
<td>0.0</td>
<td>1.0</td>
<td>1.085</td>
<td>4.3</td>
<td>355.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.2</td>
<td>1.0</td>
<td>5.0</td>
<td>1.2</td>
<td>1.302</td>
<td>19.1</td>
<td>55.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.3</td>
<td>0.9</td>
<td>3.0</td>
<td>1.2</td>
<td>1.302</td>
<td>15.4</td>
<td>65.0</td>
<td></td>
<td></td>
</tr>
<tr>
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<td>1.2</td>
<td>6.0</td>
<td>1.4</td>
<td>1.519</td>
<td>0.0</td>
<td>45.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.5</td>
<td>0.0</td>
<td>0.0</td>
<td>1.5</td>
<td>1.628</td>
<td>0.0</td>
<td>50.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Feed Rate (GPM)</td>
<td>Recycle Rate (GPM)</td>
<td>Recycle Ratio</td>
<td>Combined Feed Rate (GPM)</td>
<td>Hydraulic Rate (GPM/ft^2)</td>
<td>% BOD 5 Reduction</td>
<td>Fresh Feed BOD 5 (mg/L)</td>
<td>Filter BOD 5 (mg/L)</td>
<td>No BOD Reduction Based on Fresh Feed (mg/L)</td>
</tr>
<tr>
<td>----------------</td>
<td>--------------------</td>
<td>--------------</td>
<td>--------------------------</td>
<td>---------------------------</td>
<td>---------------------</td>
<td>------------------------</td>
<td>----------------------</td>
<td>--------------------------------</td>
</tr>
<tr>
<td>Feed Rate (GPM)</td>
<td>Recycle Rate (GPM)</td>
<td>Recycle Ratio</td>
<td>Combined Feed Rate (GPM)</td>
<td>Total Hydraulic Rate (GPM/FT²)</td>
<td>% BOD Reduction</td>
<td>Fresh Feed BOD₅</td>
<td>Filter Feed BOD₅</td>
<td>% BOD Reduction Based on Fresh Feed</td>
</tr>
<tr>
<td>-----------------</td>
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<td>-------------------------------</td>
<td>-----------------</td>
<td>------------------</td>
<td>-----------------</td>
<td>-----------------------------------</td>
</tr>
<tr>
<td>0.06</td>
<td>0.2</td>
<td>3.3</td>
<td>0.26</td>
<td>0.282</td>
<td>71.4</td>
<td></td>
<td></td>
<td>35.0</td>
</tr>
<tr>
<td>0.02</td>
<td>0.2</td>
<td>10</td>
<td>0.22</td>
<td>0.239</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.1</td>
<td>0.1</td>
<td>1.0</td>
<td>0.2</td>
<td>0.217</td>
<td>22.2</td>
<td>147</td>
<td>45.0</td>
<td>75.5</td>
</tr>
<tr>
<td>0.2</td>
<td>0.0</td>
<td>0.0</td>
<td>0.2</td>
<td>0.217</td>
<td>18.5</td>
<td></td>
<td>325.0</td>
<td></td>
</tr>
<tr>
<td>0.04</td>
<td>0.16</td>
<td>4.0</td>
<td>0.2</td>
<td>0.217</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.1</td>
<td>0.0</td>
<td>0.0</td>
<td>0.1</td>
<td>0.109</td>
<td>26.7</td>
<td></td>
<td>300.</td>
<td></td>
</tr>
<tr>
<td>0.05</td>
<td>0.05</td>
<td>1.0</td>
<td>0.1</td>
<td>0.109</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.02</td>
<td>0.06</td>
<td>3.0</td>
<td>0.08</td>
<td>0.087</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.02</td>
<td>0.04</td>
<td>2.0</td>
<td>0.06</td>
<td>0.065</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
...recycle ratio as the parameter.

When comparing the performance of the experimental filter with the data published in the literature, it is found that the performance appears to be poor. The apparent anomaly can be explained based on the facts that the data reported in the water treatment literature include primary and final settling tanks. The percent BOD reductions cited by the literature are in fact the reduction accomplished by the filters plus the reduction contribution of the primary and secondary settling tanks used in connection with the filters. According to Eckenfelder and O'Connor (10) and Fair et al., plain sedimentation on primary settling tanks is capable of removing from 25 to 30% of the incoming BOD. It is also known that due to the good sedimentability characteristics of trickling filter effluents, secondary settling tanks remove a significant percentage of the BOD load which reaches them, as the primary tanks do. It is then evident that BOD reductions accomplished in trickling filter installations due to settling tanks alone are in the order of magnitude of 40 to 60%. Two tests made in this work showed that the average removal attained in the multichamber settler used in this study is in the order of 20%. This cipher, together with that corresponding to a primary settler, makes the efficiency of the experimental filter equivalent to those reported in the literature.

The filter installation was totally assembled indoors for convenience. The fact that the filter did not receive any direct sunlight is partly responsible for its non-optimum performance (9). The indoor temperature was about 23 °C.

The performance of the filter was correlated based on the BOD reduction accomplished by the filter only. It does not includes settling tanks. Figure 11, page 36
FIGURE 1

EFFECT OF THE FILTER DEPTH TO HYDRAULIC LOAD RATIO ON THICKLING FILTER PERFORMANCE USING SUGAR MILL WASTEWATERS

Filter Depth / Hydraulic Loading, \( \text{Ft. / GPH / ft}^2 \)
As the filter performance using recycle ratio as a parameter. The procedure that allows was used to obtain the performance correlation of the filter used in this study.

As suggested from Figure 11, page 36, the filter performance could be expressed by an equation of the form:

$$\frac{L_e}{L_0} = C_r \left( \frac{D}{Q} \right)^{S_r}$$

where $L_e$ = BOD remaining in filter effluent

$L_0$ = BOD applied to the filter (including recycle)

D = filter depth, ft.

Q = hydraulic loading, GPM/Sq ft.

$C_r$ = constant that is a function of the recycle ratio (R)

$S_r$ = exponent that is a function of the recycle ratio (R)

$C_r$ and $S_r$ can be expressed as follows:

$$C_r = C \cdot f(R)$$

$$S_r = S \cdot g(R)$$

where $C$ and $S$ are constants.

Expressing equation 6 in logarithmic form,

$$\log \frac{L_e}{L_0} = S_r \log \frac{D}{Q} + \log C_r$$

The determination of $C_r$ is most easily done when $(D/Q) = 1.0$; at this time $C_r = L_e/L_0$.

Reading directly from Figure 12, page 39, the following ciphers are obtained:
is now assumed that when the recycle ratio is zero (R=0.0), the influence of f(R)
row-existing, and therefore $C_r = C = 0.94$. With the value of $C$ already established,
values of f(R) are obtained accordingly; as shown below:

<table>
<thead>
<tr>
<th>R</th>
<th>0</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>≥ 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>$C_r$</td>
<td>0.94</td>
<td>0.91</td>
<td>0.88</td>
<td>0.86</td>
<td>0.83</td>
</tr>
</tbody>
</table>

Plotting R vs. f(R) (graph 12) yields the equation for the function f(R); namely:

$$f(R) = 1.0 - 0.03(R) \tag{10}$$

The determination of the function g(R) is more complex since it involves the
calculation of the slopes for the various curves. The calculations involved for the
termination of the slope $S_r$ when R = 0.0, are included as an example:

When $R = 0.0$, from Figure 11, page 36

$$S_r = \frac{\log 0.94 - \log 0.98}{\log 1.0 - \log 10.0}$$

$$S_r = \log 1.0561$$

$$S_r = 0.02366$$

The remainder of the calculations is presented below in tabular form:

<table>
<thead>
<tr>
<th>R</th>
<th>0</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>≥ 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>$S_r$</td>
<td>-0.02366</td>
<td>-0.03981</td>
<td>-0.05805</td>
<td>-0.07115</td>
<td>-0.07188</td>
</tr>
</tbody>
</table>
FUNCTION f(R) VERSUS RECYCLE RATIO IN INJECTION FILTRATION TESTS

Recycle Ratio, R
since when \( R = 0 \) the influence of the function \( g(R) \) is non-existing, the value of \( S_r = -0.02366 \). The different values of the function \( g(R) \) are obtained in a similar fashion as the values for \( f(R) \); namely:

\[
\begin{array}{cccccc}
R & 0 & 1 & 2 & 3 & \geq 4 \\
g(R) & 1.0 & 1.683 & 2.453 & 3.007 & 3.038 \\
\end{array}
\]

The plot of these figures appears on graph 13, page 41. In order to obtain the equation for the function \( g(R) \), the curve was divided into two sections. One linear portion, and an approximate quadratic portion. The contribution of both were added and the following equation was obtained:

\[
g(R) = 1.0 + 0.725(R) - 0.033(R)^2
\]  
(11)

The general filter performance equation, based on the filter alone, is therefore:

\[
L_o = (0.94) (1.0 - 0.03R) (D/Q)^a
\]

where \( a = (-0.02366) (1.0 + 0.725R - 0.033R^2) \)  
(12)

There is no doubt that one of the most important uses of the above equation (12) is for determining the limiting hydraulic load. The limiting hydraulic load for a particular filter is defined as that value of \( Q \) (hydraulic load) at which no BOD reduction occurs. At this condition the ratio \( L_o/L_o = 1.0 \) and the filter equation now is

\[
C_r \left( \frac{D}{Q_L} \right) S_r = 1.0
\]  
(13)

from which the value of \( Q_L \) is calculated:

\[
Q_L = D \left( C_r \right)^{1/S_r}
\]  
(14)

Using a recycle ratio of 3.0 as an example, the value of \( Q_L \) obtained is 34.2 GPM/Sq. Ft., which agrees with the value given by Figure 11.
FIGURE 13
FUNCTION $g(R)$ VERSUS RECYCLE RATIO ON THICKLING FILTRATION TESTS

$g(R) = 1.01 + 2.75 R + 0.033 R^2$

Recycle Ratio, $R$
Conclusions:

Sugar mills waste waters can be treated efficiently by trickling filters. The molar ratios of BOD/N/P remain to be determined and further investigation in this aspect is required.

The performance of trickling filters alone, can be correlated by an equation of the form:

\[
\frac{L_e}{L_o} = C_r \left( \frac{D_r}{Q} \right)^{1/r}
\]

where the subscript \( r \) denotes a recycle ratio dependency. This correlation in turn can be used to predict the limiting hydraulic load to a particular filter. Equations for calculating \( C_r \) and \( S_r \) were found in this study. These are applicable to sugar cane waste waters only.

The fact that the original rock slime has to be acclimated prior to its use in continuous trickling filters may be a disadvantage due to the seasonal characteristic of the cane sugar milling activity.

BOD \(_5\) reductions as high as 75% (based on fresh feed) can be accomplished with recycle ratios of 1.0. Higher reductions are possible when using a secondary settler for the filter effluent.
activated Sludge Treatment:

General Theory:

The activated sludge process may be defined as a system in which flocculated biological growths are continuously circulated and contacted with organic waste waters in the presence of oxygen. The oxygen is usually supplied from air bubbles injected into the sludge liquid mass under turbulent conditions. The process involves an aeration step followed by a solid liquid separation one from which the separated sludge is recirculated back for re-mixing with the influent waste. The aeration step may be considered composed of three functional phases:

a) a rapid absorption of waste substrate by the active sludge,

b) progressive oxidation and synthesis of the absorbed organics and the concurrent removal of organics from solution.

c) further aeration resulting in oxidation and dispersion of the sludge particles.

The different phases of the process are shown in Figure 14.

Several modifications to the conventional activated sludge process have been developed. The purpose of the different modifications is to achieve economic advantage in construction and operation of these units.

The conventional activated sludge process as carried out in practice involves the following steps:

1) Primary sedimentation of the wastes to remove settleable organic and inorganic solids.

2) Aeration of mixture of wastes and biologically active sludge.
PHASES INVOLVED IN
THE ACTIVATED SLUDGE PROCESS

FIGURE 14

CONCENTRATION

AERATION TIME

Dispersed Growth

Range of Effective\nFlocculation

Flow Dispersion.

Organic Substrate

Biomass

\[ t_1 \]

\[ t_2 \]
3) Separation of the biologically active sludge from the effluent wastes by sedimentation.
4) Return of settled sludge to re-mix with the raw waste.

The process is schematically illustrated in Figure 15.

**FIGURE 15**

**CONVENTIONAL ACTIVATED SLUDGE PROCESS**

Conventional activated sludge treatment of domestic sewage has been shown to accomplish 90-95 per cent BOD reduction (9). This process can operate over a wide range of loading conditions varying from an active sludge with high synthesis yields to extended aeration in which most of the sludge synthesized in the process is destroyed by oxidation. The loading limitation on the process is that required to effect flocculation and permit settling and separation of the biological flocs. Bess and Conway (4) have reported BOD reductions in the order of 80% in an aerated stabilization process with no recirculation.

The conventional process, to yield high-degree treatment, will operate over the range AB of the curves shown in Figure 14. There will be a high biological sludge yield from synthesis $\Delta S$, which will require subsequent treatment and disposal. The oxygen-utilization rate will remain high throughout the process.
In the extended aeration process, sufficient aeration time is provided to oxidize substantially all of the sludge synthesized from the waste water as indicated by time $t_2$ in Figure 14. The mean oxygen-utilization rate is at or near the endogenous respiration level. Figure 16 shows the flow diagram for this type of process.

**FIGURE 16**

EXTENDED AERATION PROCESS

The contact stabilization process is applicable to the treatment of wastes containing a large proportion of the BOD in suspended or colloidal form. A high percentage of the BOD is rapidly removed by biosorption after contact with well-aerated activated sludge. In this process, waste is aerated with stabilized sludge for a short contact time (15-60 min.). The mixed liquor is then separated by sedimentation. The settled sludge is transferred from the clarifier to a sludge stabilizer where aeration is continued to complete the oxidation and to prepare the sludge for BOD removal in fresh incoming waste. Figure 17 illustrates this process.
Effective removal in the contact period requires sufficient active sludge to remove the colloidal and suspended matter and a portion of the soluble organics. The retention time in the stabilizer must be sufficient to stabilize these organics; if it is insufficient, unoxidized organics will be carried back to the contact tank and the BOD removal efficiency will decrease. If on the other hand, the stabilization period is too long, the sludge will undergo excessive autoxidation and part of its high initial removal capacity will be lost in the contact tank. There is no doubt that by increasing the contact period substantially, almost complete oxidation of the absorbed organics will occur, thereby eliminating the need for the stabilization step.

The contact stabilization process has been employed successfully by Ullrich and Smith (9) in Texas for the treatment of domestic sewage. The literature also reports that Zablatsky (37) used this process to treat wastes in New Jersey. Eckenfelder and Grich (10) conducted pilot plant studies on cannery wastes using this process. Bevan (3), has casually reported the use of this type of installation for treating sugar mill...
In practice, autoxidation of biological sludge does not follow first order kinetics, but rather follows as slowdown trend in which the rate of oxidation decreases with time or concentration. This is due to the fact that the various cellular constituents are in their ease of oxidation or biodegradability. A portion of the cellular material is only resistant to oxidation and it is thereby accumulated. McKinney (23) and others have reported that non-oxidizables solids build-up from autoxidation of biological sludge may amount to 25% of the sludge formed.

The application of the activated sludge process or any of its modifications, to the treatment of sugar mills waste waters is non-existing at this time. Waste water treatment and disposal in sugar mills range from no treatment at all, to complete land application. End-of-line treatment, when practiced, generally consists of primary settling before discharge or impoundage. Biaggi (6) and Guzmán (15) have reported sugar mill effluent characteristics for many sugar factories in Puerto Rico and stress the need for adequate treatment of the wastes.

2. Experimental Procedures Followed:

A bench scale bio-oxidation unit (activated sludge) was used in this study. It was composed of the following elements:

1) glass bio-oxidation reservoir with support stand, volume equal to 4800 ml.
2) feed metering pump
3) three-spokes air diffuser
4) air pump
5) air metering rotameter
6) sludge and recycle metering pump
7) effluent metering pump
8) effluent clarifying tubes
9) vacuum pump

This type of unit assembly was chosen to perform this phase of the study because it offers the following advantages:

1) It provides for continuous flow and for uniform organic loading.
2) Metered aeration and inlet-outlet flows possible.
3) Control of suspended solids is easily achieved.

Although exact duplication of full scale results is for all practical purposes impossible, this unit provided useful data through maximum control of the three process variables: liquid residence time, sludge residence time, and rise velocity in the central mixing tube.

Fresh waste water samples were brought in daily from the sugar factories in 5 and 13 gal. plastic containers.

The original seeding material for the activated sludge unit was obtained from a sewage treatment plant’s digester. It was brought to the laboratory and continuously supplied with air by means of a portable air pump. The acclimation of the seed was accomplished by removing one liter of this liquor daily and adding to it an equal amount of sugar mill waste water. The temperature was constantly monitored and maintained at 25°C. The procedure was allowed to continue for about four weeks. Adequate amounts of this seed were transferred to the activated sludge unit whenever this was to start operating.
The unit accomplished complete mixing almost instantly. The influent feed was continuously pumped into the outer cone (see figure 18). Metered air, distributed through special glass porous diffusers, carried mixed liquor up, between the concentric inner and outer cones. The released air bubbled through the liquid thus keeping the sludge mixed. The mixed liquor flowed down the inner cone to continue recirculating. Effluent rose in the clarifying tube in the center of the inner cone under quiescent conditions allowing the biological mass to separate and settle back into the recirculating stream. Effluent was aspirated from the surface of the clarifying tube into the effluent stage bottle. Sludge concentration was controlled by discarding part of the mixed liquor to the sludge bottle and by adjusting the pumping rate. The amount of sludge recycled was controlled in analogous manner.

The activated sludge unit was operated intermittently at the beginning of this study, with run periods of 4 to 6 hours. The achievement of steady state conditions was perhaps the problem of most difficulty during this initial testing period. Once enough experience was acquired, reaching steady state conditions was an easy task although the problem was recurrent at times when lines became plugged up with sludge. This imposed a limitation on the range of flow conditions with which the unit could be operated.

Since it has been established that sugar mill effluents are deficient in nutrients \( \delta \), \( 10 \text{ml. of } 3.615 \text{ gm/l } \text{KNO}_3 \text{ and } 0.44 \text{ gm/l } \text{KH}_2 \text{PO}_4 \text{ nutrient solution were added in some of the runs to the mixed liquor to enhance the BOD removal efficiency.}\)

3. Results and Findings:

The data gathered on the performance of the activated sludge unit appear summarized in Table 9, page 62 of this report. Three of the runs were made using no recycle of sludge at all. Nutrients were added to the waste water in three other runs,
Diagram of the aeration process.
...cations of runs were varied from two to ten hours.

To best analyze the data the following definitions are introduced:

a) Recycle ratio \( R = \frac{\text{recycle rate}}{\text{total feed flow rate}} \) \hspace{1cm} \hspace{1cm} (15)

b) Loading \( L_t = \frac{L_0 + R L_e}{1 + R} \) \hspace{1cm} \hspace{1cm} (16)
in which \( L_0 = \text{initial BOD} \)

and \( L_e = \text{final BOD} \)

c) Detention time \( T_d = \frac{V}{Q} \)
in which \( V = \text{tank volume, ml.} \)

and \( Q = \text{total feed rate, ml./min.} \)

d) BOD removal constant \( r \)

\[ r = \frac{L_0 - L_e}{T_d} \] \hspace{1cm} \hspace{1cm} (18)

Running out these calculations on the experimental data obtained in the study

the results shown in the following table are obtained:

\begin{center}
\begin{tabular}{|c|c|c|c|c|c|c|c|c|c|}
\hline
\textbf{RUN} & 1 & 2 & 3 & 4 & 5 & 6 & 8 & 9 & 10 \\
\hline
\textbf{Lt} & 65 & 292 & 175 & 327 & 233 & 212 & 73 & 38 & 251 \\
\hline
\textbf{r} & 0.05 & 0.906 & 1.00 & 2.144 & 1.225 & 0.671 & 0.192 & 0.134 & 0.695 \\
\hline
\end{tabular}
\end{center}
...en the BOD removal constant is plotted versus the total loading in log-log paper, as in Figure 19, a straight line is seen to describe the results adequately. The equation of this line is

\[ L_t = 240 \times r^{1.02} \]  

(19)

This type of correlation was first developed by Weston (36). He defined the BOD removal constant as \( (L_0 - L_e) / (I_d - L_e) \), and a loading the same as that presented in Section 16. Operational data for over 20 plants could be correlated adequately when the parameters were plotted in log-log paper, different straight lines resulting for each of the various types of treatment. In other words, the conventional process yielded a different straight line from that corresponding to contact stabilization, this in turn differed from that for the completely mixed process, and so forth.

The BOD reductions for these tests are shown as a function of the time of operation of the units in Figures 20, 21, 22, 23 and 24, in pages 97 to 101 of the Appendix.

- **Conclusions:**

Cane sugar waste waters can be treated efficiently via the conventional activated sludge process. BOD reductions as high as 80% were obtained in the pilot plant studies conducted.

When no recirculation of sludge was done the BOD reductions attained were considerably smaller than those corresponding to the use of recycle. The use of nutrients did not have an effect on the efficiency of the process. These points are illustrated by the operational data reported in Table 9.

A correlation which permits determining the overall efficiency of the process as a function of detention time and recirculation ratio was obtained in this study.
<table>
<thead>
<tr>
<th>Run Number</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
<th>9</th>
<th>10</th>
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<tr>
<td>Duration, Hours</td>
<td>2</td>
<td>6</td>
<td>5</td>
<td>10</td>
<td>5</td>
<td>7.5</td>
<td>6</td>
<td>5</td>
<td>6</td>
<td>8.5</td>
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<tr>
<td>Initial BOD, PPM</td>
<td>65</td>
<td>292</td>
<td>175</td>
<td>275</td>
<td>180</td>
<td>185</td>
<td>--</td>
<td>60</td>
<td>35</td>
<td>10</td>
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<tr>
<td>Final BOD, PPM</td>
<td>59.4</td>
<td>205</td>
<td>75</td>
<td>65</td>
<td>60</td>
<td>45</td>
<td>--</td>
<td>20</td>
<td>7</td>
<td>65</td>
</tr>
<tr>
<td>Per Cent Reduction</td>
<td>8.7</td>
<td>29.8</td>
<td>57.2</td>
<td>76</td>
<td>66.5</td>
<td>75.6</td>
<td>75.6</td>
<td>67</td>
<td>80</td>
<td>69.4</td>
</tr>
<tr>
<td>Effluent Flow Rate, MI/Min.</td>
<td>48</td>
<td>48</td>
<td>44</td>
<td>43</td>
<td>43</td>
<td>19</td>
<td>19</td>
<td>19</td>
<td>19</td>
<td>19</td>
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<td>Sludge Flow Rate, MI/Min.</td>
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<td>4</td>
<td>5</td>
<td>5</td>
<td>4</td>
<td>5</td>
<td>6</td>
<td>6</td>
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<td>6</td>
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<tr>
<td>Total Feed Rate, MI/Min.</td>
<td>51</td>
<td>50</td>
<td>48</td>
<td>49</td>
<td>49</td>
<td>23</td>
<td>23</td>
<td>23</td>
<td>23</td>
<td>23</td>
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<td>Recycle Rate, MI/Min.</td>
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<td>0</td>
<td>2.5</td>
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<td>2.5</td>
<td>3</td>
<td>3</td>
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<td>3</td>
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<td>Fresh Feed Rate, MI/Min.</td>
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<td>50</td>
<td>48</td>
<td>46.5</td>
<td>47</td>
<td>20.5</td>
<td>20</td>
<td>20</td>
<td>20</td>
<td>20</td>
</tr>
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<td>Air Flow Rate, SCFM</td>
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<td>6.0</td>
<td>5.0</td>
<td>3.0</td>
<td>3.0</td>
<td>5.0</td>
<td>5.0</td>
<td>5.0</td>
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</tr>
<tr>
<td>Temperature, °C</td>
<td>26</td>
<td>25</td>
<td>28.5</td>
<td>28.5</td>
<td>24.5</td>
<td>25</td>
<td>25</td>
<td>25</td>
<td>24.5</td>
<td>26.0</td>
</tr>
<tr>
<td>Addition of Nutrients</td>
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<td>No</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>Yes</td>
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</tbody>
</table>
FIGURE 19

BOD REMOVAL CONSTANT VERSUS TOTAL LOADING IN ACTIVATED SLUDGE
Evaporation using Mechanical Aeration:

General Theory:

Aeration has been used for many years for transferring oxygen to biological treatment processes; for solvent stripping from wastes, and for removing volatile gases such as 

\( \text{CO}_2 \) and \( \text{HCN} \) from liquids. This process is a gas - liquid mass transfer one in which

interphase diffusion occurs when a driving force is created at non - equilibrium conditions.

In the gas phase, the driving force is the partial pressure gradient; in the liquid phase, a

concentration gradient.

The rate of molecular diffusion of a dissolved gas in a liquid is dependent upon the characteristics of the gas and the liquid, the temperature, the concentration gradient, and the cross-sectional area across which diffusion occurs. The diffusional process is defined by Ficks law as:

\[
N = -DLA \frac{dc}{dy}
\]  

(20)

where \( N \) = mass transfer per unit time

\( A \) = cross sectional area through which diffusion occurs

\( \frac{dc}{dy} \) = concentration gradient perpendicular to cross sectional area

\( DL \) = diffusion coefficient.

If it is assumed that equilibrium conditions exist at the interface, the mass transfer process can be expressed as:

\[
N = (-D_g A \frac{dp}{dy})_1 = (-DL A \frac{dc}{dy})_2 = (-D_e A \frac{dc}{dy})_3
\]  

(21)

where \( D_g \) = coefficient of diffusivity through the gas film

\( D_e \) = eddy diffusion coefficient of the gas in the body of the liquid

and \( \frac{dp}{dy} \) = partial pressure gradient through the gas film.
...the system dealt with in waste treatment involves high degrees of turbulence, the diffusivity will be several orders of magnitude greater than the coefficient of molecular diffusivity, and this need not be considered as a rate - controlling step.

Lewis and Whitman (20) developed the two film concept which considers stagnant gas and liquid interfaces through which mass transfer must occur. Equation 17 can then be expressed in terms of liquid and gas film coefficients as follows:

\[ N = K_L A (C_s - C) = K_g A (P_e - P) \]  \hspace{1cm} (22)

where \( C_s \) = oxygen saturation value

\( K_L \) = liquid film coefficient defined as \( D_L / Y_L \)

\( K_g \) = gas film coefficient defined as \( D_g / Y_g \)

and \( Y_L, Y_g \) are the film thickness of the liquid and gas respectively. The transfer process is shown schematically in figure 25 below.

Most of the mass transfer applications in waste water treatment are liquid-film controlled. Increasing the fluid turbulence will decrease the film thickness and hence increase \( K_L \). Danckwert (8) has defined the liquid film coefficient as:

\[ K_L = D_L r \]  \hspace{1cm} (23)

where \( r \), the rate of surface renewal, can be considered as the frequency with which a fluid with a solute concentration \( c \) is replacing fluid from the interface with concentration \( C_s \).

For liquid film controlled processes, equation 22 can be re-expressed in terms of concentration units as:

\[ \frac{N}{V} = \frac{dc}{dt} = K_L A \frac{A}{V} (C_s - C) \]  \hspace{1cm} (24)

where \( K_L \frac{A}{V} \) is equal to \( K_L a \), \( K_L a \) being an overall film coefficient.
In practice it is usually impossible to measure interfacial areas in aeration, the diffusion coefficient $K_\text{La}$ is usually used to compute the transfer rate.

The oxygen transfer coefficient $K_\text{La}$ is affected by the chemical and physical characteristics of the aeration system. These variables are the temperature, degree of mixing, liquid depth and waste characteristics. The liquid-film coefficient will increase with increasing temperature according to the following relationship:

$$K_L(t) = K_L(20^\circ \text{C}) \times 1.02^{(t-20)}$$  \hspace{1cm} (25)

where $t$ is the temperature expressed in degrees Centigrade. When air bubbles are involved, changes in liquid temperature will also affect the size of bubbles generated. For such a system, the effect of temperature on $K_\text{La}$ is given by:

$$K_\text{La}(t) = K_\text{La}(20^\circ \text{C}) \times 1.02^{(t-20)}$$  \hspace{1cm} (26)

Increasing the degree of mixing or turbulence will increase the overall transfer coefficient due to the film thickness reduction accompanying the process.

The effect of liquid depth on $K_\text{La}$ will depend very much upon the aeration method. For most types of bubble-diffusion systems $K_\text{La}$ will vary with depth according to the relationship,

$$\frac{K_\text{La}(H_1)}{K_\text{La}(H_2)} = \left(\frac{H_1}{H_2}\right)^n$$  \hspace{1cm} (27)

The exponent $n$ has a value of about 0.7 for most systems.

The presence of surface active agents and other organic contaminants will affect both $K_\text{La}$ and $A/V$ significantly. Molecules of surfactants will orient themselves on the interfacial surface thus creating a barrier to oxygen diffusion.

In diffused aeration systems, air bubbles are formed at an orifice from which they are released and rise through the liquid, finally bursting at the liquid surface. The
shape and size of the air bubbles are related to a modified Reynolds number; namely:

\[ R_e = \frac{dv \rho}{\mu} \tag{28} \]

Eckenfelder (9) has developed a general correlation for oxygen transfer from air bubbles through a still water column. This relationship is:

\[ K_L d_B H^{1/3} = C \left( \frac{d_B v}{V} \right) \left( \frac{V}{D_L} \right)^{1/2} \tag{29} \]

An expression relating \( K_L a \) and the air flow was also developed by Eckenfelder, assuming that the tank liquid surface was very small as compared to the interfacial bubble surface.

An expression is:

\[ K_L a = \frac{C H^{2/3} G_s (1-n)}{V} \tag{30} \]

where \( G_s \) = air flow rate

\( H \) = liquid depth

\( V \) = liquid volume

Numerous investigators (9, 10, 12) use a BOD removal constant to express their results in this type of process, assuming that the reaction is a first order one which may be described by the equation

\[ \frac{L_t}{L_0} = \frac{1}{(C_1 + K_{BOD} t)} \tag{31} \]

2. Experimental Procedures Followed:

The experimental equipment on oxidation by direct aeration used in this research study consisted of the following pieces:

1. Aeration tank; 20 inches diameter, 3 feet depth.
2. Aeration tank; 15 inches diameter, 3 feet depth.
3. Aeration tank; 10 inches diameter, 3 feet depth.
4- Air compressor.
5- Three spiral copper tubing assemblies.
6- Rotameter.
7- Three variable-speed electric stirrers.

The spiral assemblies were made out of several feet of copper tubing through which numerous 1/16 inch holes were drilled. Each assembly was wound and placed so to cover about 75% of the bottom of the vessel in each case.

Since the cylindrical vessels were not interconnected, three independent batch tests could be run at the same time. The only setback was the inability of the compressor to deliver the necessary amount of air to run all three units with air injection at the same time. Figure 34 schematically presents a typical batch test set up for this oxidation process.

The primary objective of this phase of the research work was to determine the effect of aeration in sugar mills' waste waters with minimum biochemical reactions occurring. Mention whatsoever is done in the literature concerning this type of oxidation process for sugar mill effluents; this being the first time that such a process is used to treat these wastes.

A measured amount of sugar mill waste water was placed in the selected vessel and its temperature, ph and general aspect logged in. Air supply was connected to the aeration spiral tube by means of a piece of rubber hose. The mechanical stirrer propeller was adjusted at a predetermined depth and a sample of waste water was collected at this time from the vessel (50 ml, approximately). The system was then started and samples collected at definite time intervals for BOD5 analyses. Table 11 presents the data collected in this phase of the research work along with associated parameters. Runs were
FIGURE 34

SCHEMATIC REPRESENTATION
OF THE MECHANICAL AERATION SYSTEM
using agitation and compressed air simultaneously, using air in the absence of
mechanical agitation, and using mechanical agitation of the liquid with no air supplied
compressor. The power consumption by the mechanical agitators was measured in
by using a wattmeter.

Results:
The method followed for the calculation of the BOD reaction constants and of the
natt oxygen transfer coefficients is illustrated hereinafter for the first run made, which
run number A-1.
The data gathered showed percentage BOD reductions of 9.1% at the end of 1 hour,
1% after 2 hours, 20.4% after 3 hours, and 34.1% at the end of 4 hours. The BOD
action constants are calculated by rearranging equation 31 to the form

\[ K_{BOD} = \frac{1}{f(1-f)} \]  \hspace{1cm} (32)

which \( f \) is the percentage reduction in BOD divided by 100. Thus, for run A-1,

\[ K_{BOD, i} = \frac{0.091}{(1-0.001)} = 0.1 \text{ hrs}^{-1} \]

\[ K_{BOD, ii} = \frac{0.169}{(1-0.169)^2} = 0.102 \text{ hrs}^{-1} \]

\[ K_{BOD, iii} = \frac{0.204}{(1-0.204)^3} = 0.085 \text{ hrs}^{-1} \]

\[ K_{BOD, iv} = \frac{0.341}{(1-0.341)^4} = 0.13 \text{ hrs}^{-1} \]

The average value of \( K_{BOD} \) for the run is therefore equal to:

\[ \frac{0.1 + 0.102 + 0.085 + 0.13}{4} = 0.104 \text{ hrs}^{-1} \]

The overall oxygen transfer coefficient is calculated using equation 24, and
assuming that a pound of oxygen must be transferred to the liquid for each pound of
<table>
<thead>
<tr>
<th>Run Number</th>
<th>Diameter, Inches</th>
<th>Volume of Wastes, Gallons</th>
<th>Air Rate, LTS./MIN.</th>
<th>Consumption, Watt-Hours</th>
<th>Initial BOD, PPM</th>
<th>Initial Inductation</th>
<th>Duration Hours</th>
</tr>
</thead>
<tbody>
<tr>
<td>A-1</td>
<td>20</td>
<td>30</td>
<td>0</td>
<td>14.7</td>
<td>220</td>
<td>34.1</td>
<td>4</td>
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<td>A-2</td>
<td>20</td>
<td>31</td>
<td>0</td>
<td>13.8</td>
<td>150</td>
<td>50</td>
<td>2.67</td>
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<td>A-3</td>
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<td>31</td>
<td>0</td>
<td>18.1</td>
<td>242</td>
<td>55</td>
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<td>A-7</td>
<td>15</td>
<td>14.3</td>
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<td>46.2</td>
<td>392</td>
<td>27.6</td>
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<td>15</td>
<td>20.6</td>
<td>0</td>
<td>16.4</td>
<td>150</td>
<td>50</td>
<td>1.5</td>
</tr>
<tr>
<td>A-9</td>
<td>15</td>
<td>18</td>
<td>0</td>
<td>51.1</td>
<td>242</td>
<td>63</td>
<td>4</td>
</tr>
<tr>
<td>A-10</td>
<td>15</td>
<td>18</td>
<td>0</td>
<td>44.4</td>
<td>160</td>
<td>66.3</td>
<td>4</td>
</tr>
<tr>
<td>A-11</td>
<td>15</td>
<td>18</td>
<td>6.5</td>
<td>51.1</td>
<td>430</td>
<td>58.1</td>
<td>5.25</td>
</tr>
<tr>
<td>A-12</td>
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<td>9.5</td>
<td>8.0</td>
<td>46.3</td>
<td>150</td>
<td>67</td>
<td>4.0</td>
</tr>
</tbody>
</table>
TABLE 12
(Continued)

<table>
<thead>
<tr>
<th>Case</th>
<th>$K_{BOD}$, HOURS.$^{-1}$</th>
<th>$K_{L,a} \times 10^{-3}$, HRS.$^{-1}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>-1</td>
<td>0.508</td>
<td>4.60</td>
</tr>
<tr>
<td>-0.1</td>
<td>0.644</td>
<td>3.19</td>
</tr>
<tr>
<td>-0.01</td>
<td>0.350</td>
<td>5.75</td>
</tr>
<tr>
<td>-0.05</td>
<td>0.890</td>
<td>3.23</td>
</tr>
<tr>
<td>-0.13</td>
<td>0.656</td>
<td>3.16</td>
</tr>
<tr>
<td>-0.14</td>
<td>0.163</td>
<td>4.73</td>
</tr>
<tr>
<td>-0.15</td>
<td>0.401</td>
<td>3.62</td>
</tr>
<tr>
<td>-0.16</td>
<td>0.229</td>
<td>3.11</td>
</tr>
<tr>
<td>-0.17</td>
<td>0.126</td>
<td>1.57</td>
</tr>
</tbody>
</table>

Figure 26 shows the variation in the oxygen transfer coefficients which occurred as the power supplied to the mechanical agitators was increased. As expected, the coefficients increased with the increases in the speed of agitation which occurred as the power fed to the agitators went up. The data shown in the figure correspond only to those runs in which compressed air was supplied to the oxidation tanks.

A similar trend is observed when the BOD reaction constants are plotted as a function of the power input, although as shown in Figures 27 and 28 the data scatter to a larger degree than the case of the oxygen transfer coefficients.
FIGURE 26
CHANGES IN OXYGEN TRANSFER COEFFICIENT FROM CHANGES IN THE AMOUNT OF POWER APPLIED

NOTE: THE DATA CORRESPOND TO RUNS IN WHICH WATER WAS SUPPLIED AT 30°C.

Power Utilization, Watt-Hr/Gallon of Wastewater
FIGURE 28
CHANGES IN THE ECO REACTION NUMBER P. VARIATIONS IN THE AMOUNT OF POWER SUPPLIED

NOTE: RUNS PERFORMED USING COMPRRESSED AIR INJECTION

Power Utilization, Watt-Hr/Gallon of Wastewater
Conclusions:

Treatment of sugar cane waste waters can be efficiently accomplished by using biological aeration methods. Average BOD reductions in the order of 50% and as high as 70%, were attained in systems which involved blowing compressed air through the waste waters while these were being stirred using variable-speed agitators. These figures are of the same order of magnitude as those observed in practice (9).

In all cases increases in the rates at which the oxidation processes and the transfer of oxygen from the gas phase to the liquid one occurred as the amount of power fed to the mechanical agitators increased.
Carbon Adsorption Treatment:

General Theory:

Although considered to be a tertiary treatment for waste waters, the value of used charcoal as a BOD reducing treatment should not be overlooked. The use of activated carbon to clean up industrial and municipal waste water is not a new born idea. Granular and powdered systems have for sometime been used on a small scale to remove organic contaminants from these streams.

Adsorption is usually explained in terms of surface tension or surface energy per unit area of a given solid. This tension or energy is caused by the molecules in the space layer which are subjected to unbalanced forces. When the surface energy of a carbon is sufficient to overcome the kinetic energy of a molecule in the proximity of the carbon surface, that molecule is adsorbed. This is most commonly known as physical adsorption by "molecular condensation" in the capillaries of the solid. The forces responsible for this "condensation" are called Van der Waals forces. Substances with large molecular weights are most easily adsorbed. There is a rapid formation of an equilibrium interfacial concentration, followed by slow diffusion into the carbon particles. The overall rate of adsorption is therefore controlled by the rate of diffusion of the solute within the capillary pores of the carbon particles. The rate varies inversely with the square of the particle diameter, increases with increasing concentration of solute and with temperature.

The degree of adsorption and the resulting equilibrium relationships have been correlated according to the empirical relationship of Freundlich (10) and the theoretically derived Langmuir (10) relationship. The Freundlich isotherm is expressed as:
\[
\frac{X}{M} = K \cdot C^{1/n}
\]

\(X\) = weight of substance adsorbed (solute)
\(M\) = weight of adsorbent
\(C\) = concentration of solute remaining in solution
\(K\) and \(n\) are constants which vary with temperature, adsorbent, and the substance adsorbed.

The Langmuir equation is based on an equilibrium between condensation and evaporation of adsorbed molecules, considering a mono-molecular adsorption layer:

\[
\frac{X}{M} = \frac{aC}{1+aC}
\]

where \(X\), \(C\), and \(M\) hold the same identity as in the Freundich equation, and
\(a\) = constant which increases with increasing molecular weight
\(C\) = amount adsorbed to form a monolayer on the surface of the carbon

The adsorptive capacity of a carbon adsorbent is directly related to the total surface area of the adsorbent. In order to minimize activated charcoal usage, it is important to develop maximum accessible surface area per unit volume while achieving high contaminants removal at the least possible cost.

Granular carbon pioneered its way into the waste water treatment few years ago, while powdered carbon was side-lined because of the handling problems it created. Recently, however, modern technology has moved ahead to overcome these problems and powdered carbon is rapidly gaining acceptance. What powdered carbon offers over granular is lower cost - about 9-15 \(\$\)/lb. vs. 30 \(\$\)/lb.

Apart from cost considerations, one of the more interesting proposed concepts based on powdered carbon is the upgrading of secondary effluent quality through biological
and, without a large capital outlay. Recently, Du Pont has developed a system
with the purpose in mind. (7). They have registered it with the name PACT (Powdered
Activated Carbon Treatment). Du Pont claims that the addition of powdered activated
carbon to the aerator of a secondary waste treatment plant can produce the effect of
primary treatment. This is possible because the carbon particles not only adsorb organics
that might poison microorganisms but also act as growth sites for these. In a clarifier, the microorganism/carbon particles tend to flocculate and settle
as they can do either alone. This results in a more dense and compact sludge; less
wastewater and fewer suspended solids, thus increasing the hydraulic capacity of the
secondary treatment system. Some results are tabulated below.

**TABLE 14**

**PERFORMANCE COMPARISON BETWEEN TWO WASTE TREATMENT METHODS**

<table>
<thead>
<tr>
<th></th>
<th>Activated Sludge Process</th>
<th>Du Pont PACT Process</th>
</tr>
</thead>
<tbody>
<tr>
<td>Detention Time, Hrs.</td>
<td>7.3</td>
<td>7.3</td>
</tr>
<tr>
<td>Carbon Dosage, G/L</td>
<td>0</td>
<td>0.4</td>
</tr>
<tr>
<td>% BOD Removal</td>
<td>79</td>
<td>96</td>
</tr>
<tr>
<td>Filtered Samples</td>
<td>56</td>
<td>86</td>
</tr>
<tr>
<td>Effluent Color, APHA Units</td>
<td>400</td>
<td>30</td>
</tr>
</tbody>
</table>
Experimental Procedures Followed:

The investigation on the activated charcoal treatment was originally intended for a liquid carbon-packed column, but the nature of the problems faced on this type of operation suggested the adoption of the batch process. The charcoal used was Nuchar 20-N, a finely divided activated carbon that was directly responsible for extreme pressure drops across the packed column thus preventing successful operation.

The waste water for the activated charcoal investigation was obtained from the main sugar mill only. It should be noted at this time, that this sugar mill produces two independent waste water streams: one is the so-called process waste water consisting of steam condenser water, cooling water and service water, and the other one is the washing water. The latter was not suited for carbon adsorption due to its high concentration of suspended solids as evidenced by table 1 (9).

The equipment used consisted of a mixing vessel, a mechanical stirrer with electric motor, one-100 ml. Pyrex beaker, glass stirrer, and a vacuum filtering assembly composed of a vacuum pump, 500 ml. filter flask and a 25 ml. Gooch crucible with filter paper.

The charcoal concentration to be used on a particular test was established arbitrarily. The charcoal was carefully weighted and one liter of waste water was measured at room temperature. A sample of the untreated waste water was prepared to be analyzed for BOD₅ and other related tests.

After pouring the weighted charcoal and the waste water in the mixing vessel, the mechanical stirrer was activated to achieve complete mixing in the least possible time. Samples were extracted from the mixing vessel at definite time intervals and
to remove the charcoal. Afterwards, the samples were prepared for the BODS and all pertinent data logged in. It is interesting that the highest charcoal fraction that could be effectively mixed was 200 gm/liter. At this high concentration, it took approximately one hour to wet the carbon mass. This long residence definitely introduced some degree of error in the BOD reduction calculations. It was necessary to say, higher concentrations of carbon were not attempted.

Conclusions and Findings:

The performance data on this activated carbon study appear on Table 13, page 78. Percent BOD reductions are given for the filtered samples at the end of a 24-hour run.

Though batch laboratory adsorption studies provide useful information regarding adsorption of adsorption to the removal of waste constituents, continuous carbon columns provide the most practical application of this process in waste water treatment. The reason are self-explanatory:

1. A subsequent carbon separation step is not required.
2. Higher removals of odor, taste, or BOD can be accomplished.
3. Greater flexibility of operation can be attained.
4. Operating costs are lower than those for a batch process.

This preliminary research on the adsorption process as applied to the treatment of waste waters from sugar mills, revealed the following findings:

1. The adsorption of organic contaminants from these wastes by powdered activated charcoal (Nuchar - 1,000), agrees with the usual theoretical and empirical correlations published in the literature as indicated by figure 29 and 30.
<table>
<thead>
<tr>
<th>Carbon</th>
<th>Concentration</th>
<th>Initial</th>
<th>Temperature</th>
<th>BOD</th>
<th>% BOD</th>
<th>Agitation</th>
<th>Process</th>
<th>Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>:</td>
<td>:</td>
<td>23</td>
<td>120</td>
<td>87.5</td>
<td>continuous</td>
<td>batch</td>
<td></td>
</tr>
<tr>
<td>100</td>
<td>:</td>
<td>:</td>
<td>21</td>
<td>135</td>
<td>33.4</td>
<td>continuous</td>
<td>batch</td>
<td></td>
</tr>
<tr>
<td>15</td>
<td>:</td>
<td>:</td>
<td>23</td>
<td>50</td>
<td>33.0</td>
<td>continuous</td>
<td>batch</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>:</td>
<td>:</td>
<td>23</td>
<td>89</td>
<td>56.2</td>
<td>continuous</td>
<td>batch</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>:</td>
<td>:</td>
<td>22.5</td>
<td>145</td>
<td>43.3</td>
<td>continuous</td>
<td>batch</td>
<td></td>
</tr>
</tbody>
</table>
5. Evaluation or determination of the ultimate or asymptotic saturation value
   for Nuclait = 1,000 activated carbon was not possible due to wide variations
   in waste water composition from one day to the other. Further research aimed
   in this direction is hereby suggested.

3. Values of the constant 'n' in the Freundlich equation are normally greater than
   unity as seen from figures 29 and 30. This can be demonstrated theoretically
   if the existence of an adsorptivity limit (ultimate saturation value) is assumed.
   The proof follows:
   a) Assume
      \[ \lim \frac{x}{m} = C_u = \lim K C^{1/n} \]  
      (35)
      applying derivative:
      \[ \frac{d}{dC} \frac{x}{m} = K \left( \frac{1}{n} \right) C^{1/n-1} \]  
      (36)
      \[ \lim \frac{d}{dC} \frac{x}{m} = \left( \frac{K}{n} \right) \lim C_u^{1/n-1} = 0 \]  
      (37)
      therefore, \( C_u^{1/n-1} \) can only be zero if and only if \( n \) is greater than
      unity.

4. Conclusions:
   The charcoal adsorption process can be therefore used to reduce the pollutional
   charge from sugar mill waste waters as well as for partially eliminating tastes, color,
   and toxic substances with a high degree of efficiency. The highly significant
   impact of the economic considerations involved, suggests to use it as a tertiary treat-
   ment or as an effluent quality upgrading in secondary treatment facilities.
FIGURE 30

MODIFIED FREUNDLICH ADSORPTION ISOTHERM FOR SUGAR MILL WASTEWATER ON ACTIVATED CHARCOAL

Residual BOD Concentration, mg/l
General Conclusions:

The experimental work reported in this study was undertaken to determine the processability of conventional waste treatment processes to the waste waters generated by the sugar cane industries in Puerto Rico. In total five different processes were considered, three of these involved biological treatment of the wastes, the operation of the other two being based on physical and chemical principles. The processes evaluated were anaerobic digestion, activated sludge treatment, trickling filtration, oxidation using mechanical aeration, and adsorption in activated charcoal. The first four methods are secondary treatment processes, while adsorption in activated charcoal is a tertiary treatment one which in industrial applications would have use in treating the effluents from secondary processes.

The applicability of these methods to the treatment of the waste waters from the sugar cane industry was determined using laboratory units which were actual prototypes of those used in industrial operations. Waste waters brought in from various sugar mills located in the western part of Puerto Rico were used as raw materials, the experimental work requiring two consecutive cane grinding seasons for its completion.

Waste waters used for cane washing purposes were found to carry a significantly higher pollutional load than process waste waters properly.

Cane sugar waste waters were found to be amenable to treatment by anaerobic digestion procedures. The rate at which organic matter present in the wastes under-went biological decomposition was found to increase sharply with time during the early stages of the process. It then declined to reach a nearly constant value at the end of 7 days of digestion. Gas production had also decreased to nearly zero at this time. Reduc-
in dissolved organic matter of the order of 50% were obtained, this in agreement
 reported efficiencies of the anaerobic digestion process when applied to other types
 of waste waters.

As digestion time increased the acidity of the waste waters went up, the concent-
ration of suspended solids went down, and the amount of gas generated increased. These
observations are in agreement with the basic theories underlying the anaerobic digestion
processes. These establish the mechanism for the formation of acids as one in which
biological decomposition of organic compounds initiates and is followed by the
transformation of these acids into gases through subsequent bacterial action. A gasifi-
ation constant of 0.4 days⁻¹ was determined to characterize the rate at which gasifi-
ation occurred.

Activated sludge treatment of cane sugar waste waters resulted in efficiency values
for the removal of dissolved organic matter as high as 80%. When no recirculation of
sludge was done the BOD reductions attained were considerably smaller than those corre-
sponding to the use of recycle. This conforms to theoretical expectations arising from
the principles underlying the activated sludge process, according to which the addition
of the active biological floc to the fresh waste waters being fed to the unit enhances the
rate at which the biochemical oxidation proceeds. The data obtained on the efficiency
of the process as a function of detention time and recirculation ratio were correlated by
the equation

\[ L_t = 240 r^{1.02} \]  

(19)

In this expression \( r \) is the BOD removal constant, defined by the ratio of change in BOD
to detention time, and \( L_t \) is the total organic loading, defined in turn as a function of
change in BOD and of the recirculation ratio. The above equation permits calculating
efficiency of the process, expressed in terms of per cent BOD removal, as a function
treatment time and recirculation ratio.

Trickling filtration was found to be an effective means of treating waste waters
in the sugar cane industry. Reductions in dissolved organic matter as high as 75 per
cent were reached using recycle ratios of 1.0. This performance, when combined with
additional reductions which would result from the use of a settler following the trick-
ker, make the results obtained equivalent to the efficiencies usually reached in
plants by this mode of treatment.

The performance data obtained on the trickling filters were correlated by the
relations,

\[
\frac{L_e}{L_0} = C_R \left( \frac{D}{Q} \right)^{S_R} \tag{6}
\]

in which the proportion \( \frac{L_e}{L_0} \) represents the fraction of the total dissolved organic matter
which is not removed by the treatment process, \( D \) is the depth of the filter, \( Q \) is the
hydraulic loading on the unit, and \( C_R \) and \( S_R \) are functions of the recycle ratios used.
The equation allows the calculation of the efficiency of a trickling filter used to treat
sugar cane waste waters from a knowledge of the variables of operation being used.

Blowing compressed air through sugar cane waste waters while these are being
agitated results in the oxidation of the dissolved organic matter contained in them.
Average BOD reductions in the order of 50%, and as high as 73%, were obtained by
using this mechanical aeration method of treatment. Increases in both the BOD removal
constant and in the rate at which mass transfer occurred from the gas phase to the liquid
...as the amount of power delivered to the liquid per unit volume went up. The...}
parameters of operation such as air flow rate, liquid depth and liquid volume, the
lower this probably being the irregularities in flow regimes introduced by end and wall
which aerate from the tanks used.

Activated charcoal can be used with a high degree of efficiency to reduce the
content of dissolved organic matter in waste waters from the sugar cane industry. Average
reductions in the order of 50 per cent were easily achieved, this in spite of the fact
that suspended solids were not entirely removed from the wastes at the moment they came
in contact with the surface of the carbon. The data obtained agree remarkably well
with those of the adsorption isotherms plots resulting from Freundlich's equation.
Treatment with activated charcoal is envisioned as a process of application in the treat-
ment of these waste waters once they have been submitted to an adequate secondary
...treatment process.

In general, the pollutional load of waste waters from the sugar cane industry may
therefore be significantly reduced by treatment using the processes of anaerobic digestion,
activated sludge, trickling filtration, mechanical aeration and adsorption in activated
charcoal. Puerto Rico's environment need not be contaminated further from this source if
its sugar industry can withstand the economic impact involved in making its operations
clean ones.
REFERENCES


7. Browning, J. E., Chemical Engineering, 81, 36 (Feb., 1972).


APPENDIX

Tables, 2, 3 and 4

Figures 31, 32, and 33

Figures 20, 21, 22, 23, 24, 35 and 36

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<th>TABLES:</th>
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<td>92</td>
</tr>
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<th>FIGURES:</th>
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<td>94</td>
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<td>95</td>
</tr>
<tr>
<td>Figure 33: Trickling Filter Set-Up</td>
<td>96</td>
</tr>
<tr>
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<td>97</td>
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<td>Figure 23: Effect of Operating Time on BOD Removal for Activated Sludge Tests - Series IV</td>
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<td>------</td>
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<td>3.4: Effect of Operating Time on BOD Removal for Activated Sludge Tests - Series V</td>
<td>101</td>
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<tr>
<td>3.6: Detention Time Effect on Amount of Organics Adsorbed from Sugar Mill Waste Waters Using Activated Charcoal</td>
<td>103</td>
</tr>
</tbody>
</table>
### TABLE 2

**EFFECT OF ANAEROBIC DIGESTION TIME ON THE GENERATION OF GASES FROM SUGAR CANE WASTE WATERS**

<table>
<thead>
<tr>
<th>Days</th>
<th>Amount of Gas Produced from five gallons of wastes, mls.</th>
<th>Percent of Total</th>
<th>Rate of Generation, mls. Per Day Per Gallon</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>1</td>
<td>45</td>
<td>8.3</td>
<td>9</td>
</tr>
<tr>
<td>2</td>
<td>188</td>
<td>34.8</td>
<td>26.6</td>
</tr>
<tr>
<td>3</td>
<td>500</td>
<td>92.8</td>
<td>31.2</td>
</tr>
<tr>
<td>4</td>
<td>523</td>
<td>96.5</td>
<td>4.6</td>
</tr>
<tr>
<td>5</td>
<td>535</td>
<td>99.0</td>
<td>2.4</td>
</tr>
<tr>
<td>7</td>
<td>540</td>
<td>100</td>
<td>1.0</td>
</tr>
</tbody>
</table>

*Note: The data correspond to the digestion of waste waters which had been used for washing cane.*
TABLE 3

EFFECT OF ANAEROBIC DIGESTION TIME ON THE ACIDITY AND THE CONCENTRATION OF SUSPENDED SOLIDS IN CANE SUGAR WASTE WATERS

<table>
<thead>
<tr>
<th>Time of Digestion, days</th>
<th>Waste Waters from Cane Washing Operation</th>
<th>Waste Waters from the Manufacturing Process</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Total Acidity (increase) : Suspended Solids (decrease)</td>
<td>Total Acidity (increase) : Suspended Solids (decrease)</td>
</tr>
<tr>
<td>0</td>
<td>0 : 0</td>
<td>0 : 0</td>
</tr>
<tr>
<td>0.5</td>
<td>6.3% : 0</td>
<td>0 : 0</td>
</tr>
<tr>
<td>3</td>
<td>56.5% : 22%</td>
<td>52.1% : 5.5%</td>
</tr>
<tr>
<td>5</td>
<td>114.2% : 36%</td>
<td>44.8% : 10.9%</td>
</tr>
<tr>
<td>7</td>
<td>1114% : 81%</td>
<td>43.5% : 12%</td>
</tr>
</tbody>
</table>

Note: The final total acidities were in the order of 450 for the cane washing wastes and 290 for the process wastes, expressed as milligrams of calcium carbonate per liter.
TABLE 4

AFFECT OF ANAEROBIC DIGESTION TIME ON THE REDUCTION IN BIOCHEMICAL OXYGEN DEMAND OF WASTE WATERS FROM CANE WASHING OPERATIONS

<table>
<thead>
<tr>
<th>Time of Digestion</th>
<th>Per Cent Reduction in Biochemical Oxygen Demand</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>2 hrs.</td>
<td>1 %</td>
</tr>
<tr>
<td>4 hrs.</td>
<td>26%</td>
</tr>
<tr>
<td>6 hrs.</td>
<td>39%</td>
</tr>
<tr>
<td>12 hrs.</td>
<td>48%</td>
</tr>
<tr>
<td>24 hrs.</td>
<td>49%</td>
</tr>
<tr>
<td>2 days</td>
<td>48%</td>
</tr>
<tr>
<td>4 days</td>
<td>51%</td>
</tr>
<tr>
<td>5 days</td>
<td>54%</td>
</tr>
<tr>
<td>6 days</td>
<td>47%</td>
</tr>
<tr>
<td>7 days</td>
<td>49%</td>
</tr>
</tbody>
</table>
FIGURE 31
TRICKLING FILTER RECYCLING SYSTEM

The pumps used for feeding and recycling and the effluent piping to the multi-chamber settler are shown in this photograph.
This picture shows the 55-gal. combined feed drum on top of the plastic distributor as part of the Trickling Filter.
This photograph shows part of the equipment used in the Trickling Filter study. Includes: Fresh feed drum and rotameter on the left hand side and filter and receiving drum to the right.
EFFECT OF OPERATING TIME ON BOD REMOVAL FOR ACTIVATED SLUDGE TESTS, SHEETS III

% BOD REMOVAL

OPERATING TIME, HRS.
FIGURE 35

DETECTION TIME EFFECT UPON BOD REDUCTION
OF SUGAR MILL WASTEWATER ON ACTIVATED CHARCOAL

CONTACT TIME, HOURS