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Environmental Protection Technology Series

# Pilot Scale Treatment of Wine Stillage

National Environmental Research Center
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# PILOT SCALE TREATMENT OF WINE STILLAGE

by
E. D. Schroeder

Department of Civil Engineering
University of California
Davis, California 95616

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Project Officer

Max W. Cochrane
Pacific Northwest Environmental Research Laboratory
National Environmental Research Center
Corvallis, Oregon 97330

NATIONAL ENVIRONMENTAL RESEARCH CENTER
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U.S. ENVIRONMENTAL PROTECTION AGENCY
CORVALLIS, OREGON 97330

## ABSTRACT

Pilot and laboratory scale studies were run on aerobic and anaerobic biological treatment of winery stillage over a two year period. The pilot scale studies included work with aerobic lagoons and anaerobic packed towers. Laboratory systems studied were aerobic reactors without recycle and batch fed anaerobic processes. Because suspended solids removal proved to be a key factor in successful biological treatment, centrifugation, detartration, coagulation and flocculation, and combinations of these methods were included in the studies.

Centrifugation proved to be the best method of removing solids prior to biological treatment. Solids removal in combination with an aerobic treatment process can be expected to produce final filtrate chemical oxygen demands of about 700 mg/L and a final filtrate BOD of about 75 mg/L. Anaerobic processes studied did not operate well but produced effluents with chemical oxygen demands of the order of 4000 mg/L.



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# SECTION I

## CONCLUSIONS

1. Biological treatment of California Winery stillage is possible. Aerobic (aerated lagoon) processes are the most feasible systems for treatment and these should be designed as two stage cascades in order to minimize nitrogen requirements.

2. Pretreatment to reduce suspended solids concentrations below 2000 mg/L is necessary for successful biological treatment process operation. Foaming and oxygen transfer problems are uncontrollable at higher concentrations. Centrifugation is the most suitable solids removal method because sludge concentrations produced are of the order of ten percent by weight or greater. Stillage suspended solids concentrations are of the order of two percent by weight and therefore sludge concentrations below ten percent result in an unacceptably large sludge volume.

3. Stillage acidity is not a major process control problem. Because the acidity is due to biodegradable organic acids, the process pH can be controlled by matching the loading rate to the organic removal rate without chemical addition.

4. The maximum organic removal rate was found to be 12.5 grams COD removed/liter-day, and the corresponding loading rate was 14.1 grams COD/liter-day. At higher loading rates, the pH and the organic removal rate decreased rapidly. Effluent quality at the maximum loading rate is approximately 2300 mg/L

COD in the settled supernatant, 650 mg/L COD in the filtrate and 75 mg/L $BOD_5$ in the filtrate.

5. Nutrient addition can be restricted to nitrogen. Very little nitrogen is available in the raw stillage and the stoichiometric requirement is approximately 2 grams N per liter. Nitrogen addition can be minimized by adding it only to the second of two bio-oxidation units operating in series. Results of the 1972 pilot plant studies lead to the conclusion that only about 500 mg/L N must be added at that point. Nitrogen should be added as $NH_4NO_3$ to avoid greatly increasing the sodium (from adding $NaNO_3$) or chloride (from adding $NH_4Cl$) concentrations of the stillage.

6. Foam and fly control in the biological treatment processes is a major problem. A stiff, hard to break foam is generated during aeration which provides an excellent media for flies to deposit eggs. Foaming was considerably less at longer residence times in the laboratory studies (three days or greater), but was a continual problem in the field studies which had a residence time of three days. A method of foam control is necessary if treatment is to be successful.

7. Success of pilot scale anaerobic treatment studies was limited because of poor temperature control. Laboratory study results indicate that effluent quality will be lower than that from aerobic processes. Start up problems were not noted during the second year of the field studies, but the pilot processes never produced effluent filtrate COD concentrations below 3900 mg/L.

# SECTION II

## RECOMMENDATIONS

Wine stillage treatment will be expensive from both capital expenditure and operational cost points of view. For this reason treatment should be considered as an alternative available when land disposal is impossible. If a decision is made to treat stillage at a winery, lack of experience in stillage centrifugation and foam and fly control will present problems. Additional research and development effort will be necessary in these areas.

# SECTION III

## INTRODUCTION

Wastewaters from California wineries include effluents from processing operations, tank cleaning and distillation of beverage brandy and fortifying spirits. Of these discharges, the most difficult treatment problem is associated with the wastewater from the distillation process. This wastewater, normally called stillage or still slops, consists of the nonvolatile material from the bottom plates of continuous stills or the residue remaining in batch stills. Stillage production varies from winery to winery, depending on the quantity of sweet wines and brandy produced, and on the type of still used. Volume of stillage produced per ton of grapes processed is not a useful parameter because not all wineries produce distilled products and those that do vary considerably in the amount of distilling material needed. A medium size operation will produce around 150,000 liters/day (40,000 gallon/day) and a large installation may produce as much as 2,300,000 liters/day (600,000 gallon/day). Nearly 90% of California's winery distillation operations occur from late August to early November. Thus most of the waste is generated during the 45 to 75 day period in which crushing occurs. Virtually all of the California wineries which produce distilled products are located in the San Joaquin Valley, and the problem is, in practical terms, limited to this region.

Currently most wineries dispose of stillage by discharging into municipal sewerage systems, treatment in lagoons or by land disposal through intermittent irrigation. Municipal systems receiving stillage have usually experienced operational problems due to overloading, and

odor problems are often associated with conventionally designed lagoons. Intermittent irrigation has proven to be the most satisfactory method of disposal. Pretreatment is not required, nuisance problems are less than those associated with other treatment methods and the method is particularly ameniable to seasonal operations. Many wineries are located in areas where land is either unsuitable for irrigation or becoming less available. Thus these wineries in particular, and the industry in general, are interested in developing alternative treatment methods.

Fermentation residues are the major source of distilling material during the processing season. The first phase of the fermentation process produces pomace material such as settleable skins, seeds and pulp, and the second fermentation process produces lees, which are yeast and solids coagulated with bentonite and settled. When liquid from pomace or lees materials are used in a distillation process the waste is called pomace or lees stillage, respectively.

Stillage characteristics vary considerably with the source of the distilling material (lees, pomace or wine), the operation of the winery and the type of still used. A general characterization would be that stillage is very high in COD, BOD, suspended solids and acidity however. Typical values reported for pomace stillage are given in Table 1.

Several significant treatment process design and operational problems can be foreseen from waste characteristics discussed up to this point. The high organic concentrations restrict the process choice to anaerobic systems or aerobic systems with a high oxygen transfer

Table 1. POMACE STILLAGE CHARACTERISTICS [1,2,3,4]

| Characteristic | Range Reported |
|---|---|
| pH | 3.5 - 5.0 |
| Acidity, mg/L as | 1,200 - 3,800 |
| Total Solids, mg/L | 13,000 - 30,000 |
| Suspended Solids, mg/L | 14,000 - 18,000 |
| Volatile Solids, mg/L | 10,000 - 27,000 |
| Total $BOD_5$, mg/L | 2,400 - 17,840 |
| Total COD, mg/L | 34,000 - 53,000 |
| Filtrate COD (0.45 Micron), mg/L | 19,000 - 22,000 |
| Total Nitrogen, mg/L as N | 150 - 330 |
| $NH_3$ - N mg/L as N | 2 - 4 |
| Total Phosphorous, mg/L as P | 1,211 - 1,310 |
| Temperature (at the still) | 150°F |

rate (in terms of mass/time), and the extremely high suspended solids concentrations make satisfactory solids removal by conventionally used techniques very difficult. Acidity and pH values associated with stillage force the use of some form of pH control in the treatment process. Nearly all of the nitrogen in the waste is in the organic form and therefore is available for use in a treatment process only as fast as the organic nitrogen containing compounds are broken down. In addition, the total nitrogen in the wastewater falls far short of that needed in aerobic biological treatment process. Finally the seasonal nature of current distillation operations requires that a treatment system has a very short start up time.

## OBJECTIVES

The primary objectives of this project were waste characterization, pilot and laboratory scale investigation of aerobic and anaerobic biological treatment of stillage and development of process design criteria. Preliminary evaluation of treatment costs, alternative treatment methods, such as direct pomace fermentation and effect of grape variety on treatment system operation were also included in the objectives.

## BACKGROUND

Distilling material produced during the crushing season is made up of lees, pomace, stems and leaves and washwater. Pomace includes yeast, pulp, skins and seeds separated from the wine after the first fermentation. This material is usually washed out of the fermentation tank, dewatered and pressed. Wine from the press is recovered and the pressed pomace is mixed with stems, leaves and water in the

disintegrater and scalper. Liquid from this mixture, commonly called pomace distilling material, is then held for distillation. Additional fermentation of residual sugar takes place during storage. Lees material results from precipitation of solids with bentonite at the end of the second fermentation step. In addition to bentonite, lees would be expected to contain yeast and residual solids from the first fermentation. Water is added to lees as they are washed out of fermentation tank. Solids removed during final clarification of the wine before bottling are usually mixed with the lees. These solids may include coagulant aids such as bentonite, gelatine or casein.

Distilling material production resulting from red wines is slightly different from that for white wines. White wine production requires removal of the skins at the crusher along with the stems to prevent coloring during fermentation. This procedure has little, if any, effect on the distilling material characteristics. A schematic of the stillage production process is shown in Figure 1.

Alcohol collected from the distillation process is used to stop fermentation during the production of sweet (dessert wines). Addition of the alcohol takes place during the second fermentation step.

## CURRENT METHODS OF STILLAGE DISPOSAL

Current methods of stillage treatment include lagooning, anaerobic treatment and intermittant irrigation. Anaerobic treatment has been used in South Africa[5,6] with wineries which operate most of the year.

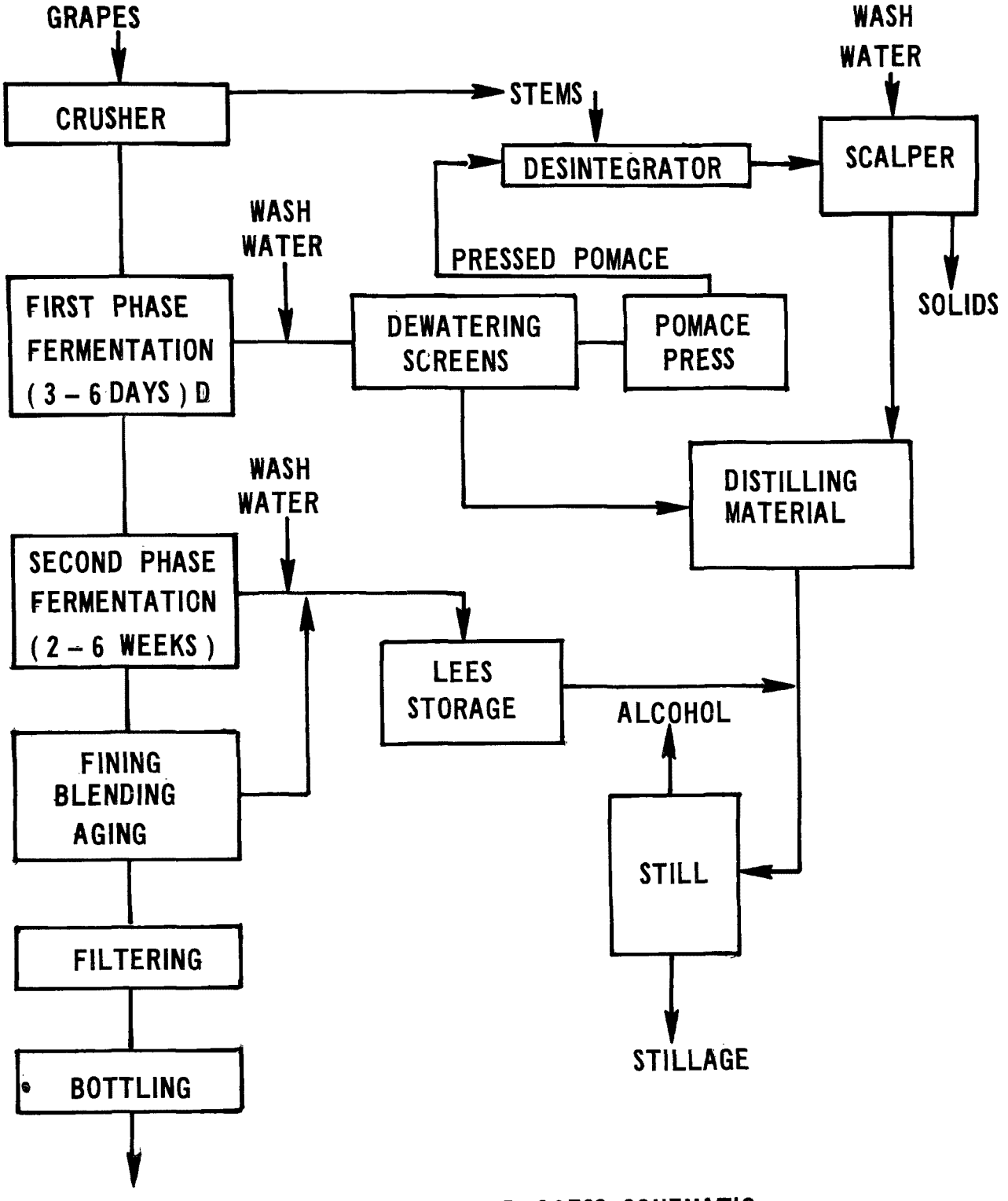


FIGURE 1. WINE PROCESS SCHEMATIC

Systems used are essentially anaerobic contact processes· i.e. the cell and hydraulic residence times are different. Hydraulic residence times are of the order of 7 to 8 days and effluent COD concentrations are of the order of 500 mg/L. No anaerobic processes are being intentionally operated in the California area, although Pearson et. al.[2] and Chadwick and Schroeder[3] reported on laboratory scale anaerobic treatment studies. A major problem with anaerobic processes is the slow start up rates which restrict their use for treating seasonal wastes.

Lagoons are used by a number of wineries in the San Joaquin Valley. Odor complaints have been a general problem associated with these systems. Unfortunately design loadings associated with the production of odors are not known[7].

Intermittant irrigation has proven to be the simplest and least expensive method of stillage disposal[3]. Maximum loading rates are 100,000 gallons per acre per week. Operation is on a batch basis. Furrows are filled and a period is allowed for evaporation and percolation. Dry solids residue is then removed or disced into the soil and the field can be reused. Nuisance problems with this procedure are not great if soil conditions are appropriate and if the disposal area is separated from residential or commercial areas. There has been some concern about potential damage to ground water quality from irrigation[9], but information is not available which would allow evaluation of the problem. York[10] has recently reported on studies of soil core samples in fields when intermittant irrigation has been used. His tentative conclusion is that salt transmission is not a significant problem. Tile drainage would undoubtably be a solution if a threat to ground water is demonstrated in later studies.

Trickling filters[11,12] and activated sludge processes[13,14] have been used to some extent for the treatment of stillage, but with little success. Rates of oxygen demand have invariably been larger than the oxygen transfer capacities of trickling filters used. Filamentous growths have been the major problem in the operation of activated sludge processes. The University of California at Davis has been involved in the study of stillage disposal methods since the revival of the California wine industry in 1933. Much of the work has been by Vaughn and his coworkers[11,15,16]. Studies have included both stillage characterization and methods of stillage treatment.

## CONCEPTUAL APPROACH

Stillage treatment system design and operation must take into consideration the low pH, and low nutrient concentrations and the high acidity, organic content, solids content and temperature of the wastewater. Each of these factors places constraints on the system. For example neutralization with lime results in excessive chemical costs and excessive sludge production due to the low pH and high acidity. The high organic content of stillage results in high cell production rates. In addition, a significant fraction of the material making up the COD can be expected to consist of lignins, cellulose and other difficult to degrade organics. Suspended solids in the stillage make up approximately one half of the COD, but consist primarily of pulp, seeds, skins, stems and yeast cells which pass a 0.24 cm (3/32") dewatering screen and which would be expected to be difficult to bio-oxidize. In addition, the high suspended solids concentrations will decrease potential oxygen transfer rates. Stillage is nitrogen limited, but

the extent of the limitation is difficult to determine. Nearly all of the nitrogen is in the organic state and therefore bio-oxidation must take place before it becomes available for bacterial growth. The extent of nitrogen limitation is therefore dependent on the quantity of nitrogen tied to the nondegradable organics. If all of the organic nitrogen is unavailable nitrogen addition necessary may be as high as 2 grams/liter. Limitations are also imposed by the temperature of stillage. The 66°C temperature reported in Table 1 is immediately following the heat exchanger at the still and the treatment plant. Temperatures in this range would be ideal for anaerobic treatment processes although some additional heat would probably have to be added to maintain thermophillic conditions. Filamentous cultures seem to predominate when high temperatures are (17) maintained in aerobic processes, however.

The constraints on biological waste treatment of stillage imposed by the stillage characteristics can be summarized by saying that a large fraction of the suspended solids must be removed prior to aerobic treatment, pH must be maintained near neutral within the biological processes and, nutrients will have to be added to aerobic treatment processes to satisfy stoichiometric requirements for growth. Temperature control will have to be considered also, but this is not a major problem in full scale systems. Heating of anaerobic processes is standard practice and cooling will occur during solids removal and aeration in aerobic processes.

A major constraint on the process choice is imposed by the seasonal nature of the wastewater production. Product quality control prevents long term storage of the distilling material (19) and thus the problem is inherrently seasonal. Stillage production begins within a few days

of the start of crushing and reaches maximum flow rates within a week. Therefore, the waste treatment process chosen must have a short start up period.

Strength of the waste is also an important consideration. The high soluble COD concentrations, most of which is biodegradable (3,5,6) places constraints on both aerobic and anaerobic processes. Aerobic process design will be limited by the ability to transfer oxygen, while anaerobic systems will be limited by the rate of conversion of organic acids to methane. In either case, the constraints will be represented by minimum hydraulic and mean cell residence time values.

The initial research plan was based on the considerations discussed above. Activated sludge was chosen as the pilot aerobic treatment system and packed bed anaerobic treatment (often called the anaerobic filter) was chosen as the anaerobic process for pilot scale investigation. Determination of acceptable loading rates, possible effluent quality and characteristic operating problems were the immediate objectives of the studies. The choice of the packed bed anaerobic treatment process was based on the need for short start up times. Because the cells have a much longer residence time than the wastewater in this system, cell concentrations become very high. Suspended solids retained in the units with the cells can serve as a partial food source during the off season and it was felt that a satisfactorily short start up period would possibly occur during the second season of operation. Because of the high solids content of the wastewater plugging of the packed bed was a distinct possibility. For this reason a sedimentation tank was placed ahead of the packed bed as well as ahead of the activated sludge processes. Sedimentation tank sizing was based on Chadwick and Schroeder's recommendation of

maximum overflow rates of 2034 liter/square meter/day (50 gallons/ square foot/day) and the maximum expected flow rate.

Initial design of the pilot activated sludge process utilized a cascade reactor concept (Figure 2). The cascade was to be a series of continuous flow stirred tank reactors with the option of settling and cell recycle between each stage. Growth rates would be very high in the first reactor and progressively decrease to the final reactor. Settleability of the culture was expected to improve through the cascade with a highly dispersed culture in the first stage and a progressively more flocculant culture developing as the growth rate decreased. Discussions with Kenneth Dostal of the National Environmental Research Center of the Environmental Protection Agency (Corvallis, Oregon) in March, 1971 resulted in altering the conceptual design to a single large reactor and a two tank cascade operated in parallel.

The program of study involved construction of the pilot plant units during the summer of 1971, and operation of the pilot plant system during the 1971 processing season. Observations and data from the 1971 season was used to set up laboratory studies during fall, winter and summer of 1971-1972. These studies were then used to set operating criteria for the pilot plants during the 1972 processing season.

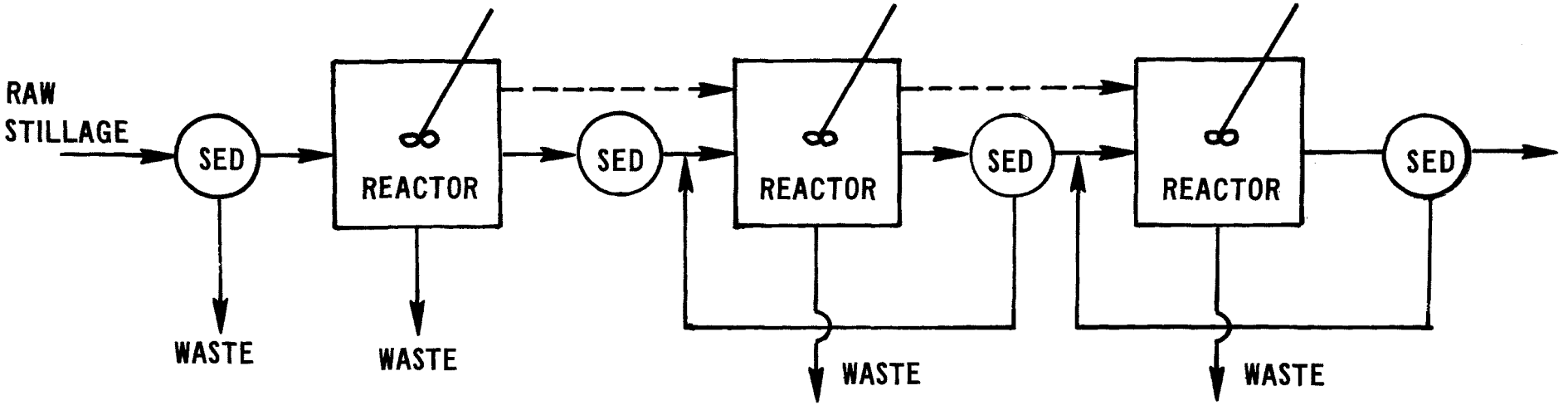


FIGURE 2. CASCADE REACTOR SYSTEM SCHEMATIC

# SECTION IV

# EXPERIMENTAL SYSTEMS AND METHODS

## PILOT PLANTS

Pilot plant studies were carried out at the Gallo Winery in Fresno, California. The winery furnished a concrete pad, electricity and water at the experimental side and also provided a shed for storage of chemicals and equipment. Help with mechanical and electrical problems which occurred from time to time was also furnished by the winery. The concrete pad was directly over the pipe carrying stillage to the fields used for intermittant irrigation and at a point approximately 300 meters from the still. Stillage for the studies was supplied from a tap in the pipe.

Stillage was pumped from the sewer into a hopper bottomed holding tank having a surface area of 1.9 meter$^2$ (20 ft.$^2$) and a volume of 1.9 meter$^3$ (66.7 ft.$^3$). Design residence time in this holding tank was approximately six hours. Previous studies[3] had led to the conclusion that solids removal would not be great in this tank and therefore identical tanks which acted as both holding and sedimentation tanks were placed in front of each group of biological treatment processes.

### Activated Sludge Processes

Two continuous flow, stirred tank activated sludge systems were operated in parallel. One system consisted of a single aeration basin with an attached sedimentation tank and the second system

consisted of two identical aeration basin-sedimentation tank combinations in series (Figure 3). Total aeration tank volume of the two systems was equal. Dimensions of the large aeration basin were 0.915 meter (3 ft.) wide, 1.83 meter (6 ft.) long, and 1.83 meter high. Liquid depth was 1.36 meter (4.45 ft.). The smaller units differed in that they were only 0.915 (3 ft.) in length.

Each aeration basin had an attached sedimentation basin with dimensions of 0.915 meter by 0.61 meter (2 ft.). The units were hopper bottomed and had a total volume of 0.57 meter$^3$ (20 ft.$^3$). Solids were removed at the tank bottom and pumped back into the aeration basin. Each sedimentation tank was divided into two sections by a partition which allowed operation at two different overflow rates for any given flow rate.

Design hydraulic residence time of the activated sludge systems was 12 hours. This corresponds to a flow rate of 190 liters/hour. Design overflow rate in the holding-sedimentation tank was therefore 2400 liters/meter$^2$-day (59 gallons/ft.$^2$-day) and the design overflow rate in the secondary clarifiers was either 8170 liters/meter$^2$-day (200 gallons/ft.$^2$-day) or 16340 liters/meter$^2$-day (400 gallons/ft.$^2$-day).

Mixing and aeration of the activated sludge units were provided by 0.61 meter (2 ft.) diameter flat bladed turbines turning at 70 rpm. The large aeration basin had two turbines and the small aeration basins had one. Power for the turbines was provided by 1490 watt (2 hp) Baldor motors (Baldor Manufacturing Company, Fort Smith, Arkansas) operating at 1725 rpm.

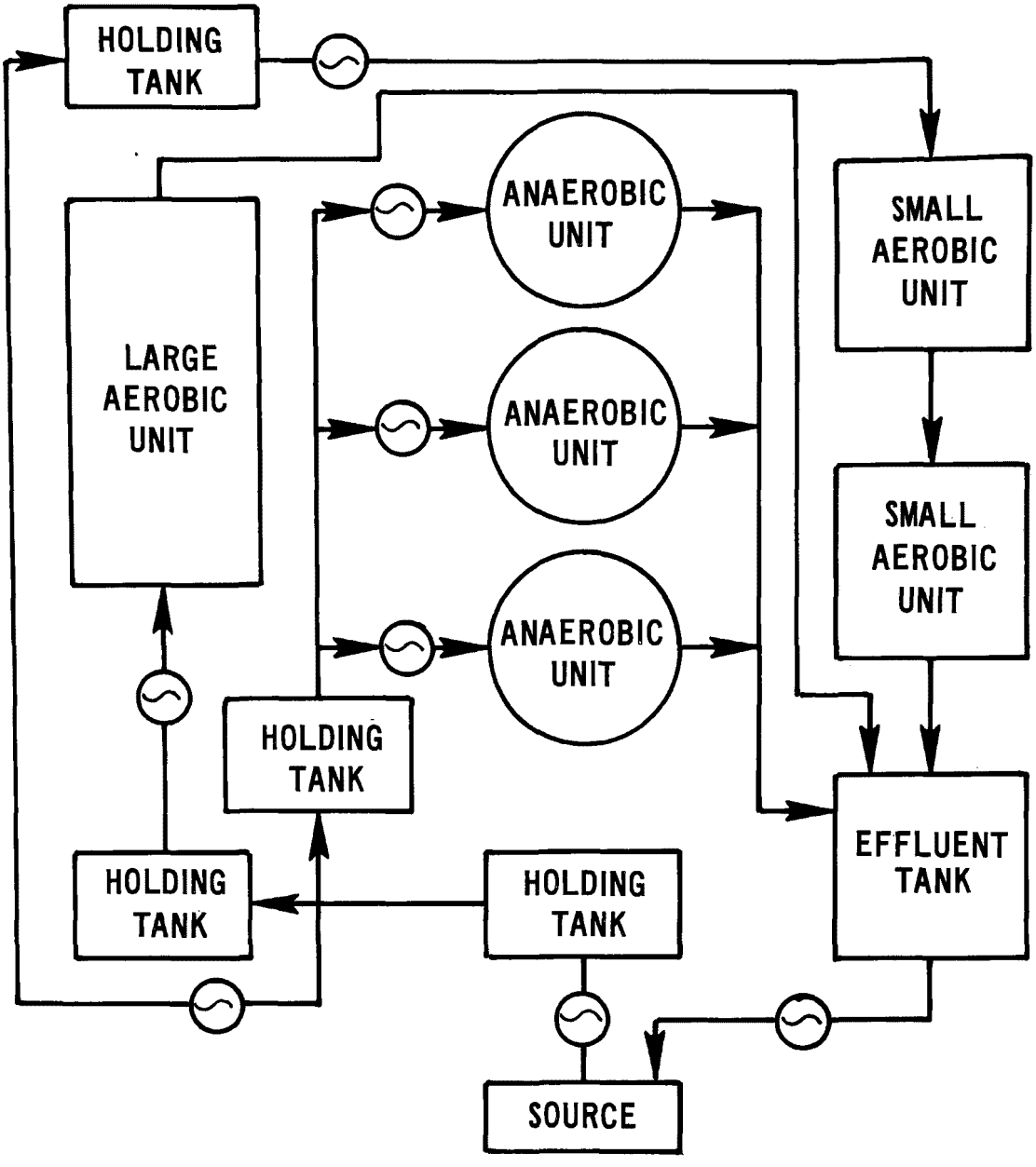


FIGURE 3 PILOT SYSTEM SCHEMATIC

A sketch of the activated sludge units is given in Figure 4.

## Anaerobic Packed Beds

The anaerobic packed beds were designed on the basis of a nominal maximum loading of 9.6 grams COD/liter/day (0.6 lb. COD/ft.$^3$/day) and an influent COD concentration of 20,000 mg/L (taken from Chadwick and Schroeder's (3) settled stillage COD values). Three 0.915 meter (3 ft.) diameter and 2.44 meter (8 ft.) deep steel tanks were constructed to house the anaerobic processes. An influent distribution section was provided in the lower 0.46 meter (18 inches) of the tank and a 0.46 meter freeboard was provided at the top. The 1.83 meter section remaining was packed with 5 cm (2 inch) Douglas Fir bark chips. Packing was contained by expanded metal grates placed at the top and bottom of the units. Sampling ports were placed at 0.3 meter (1 ft.) intervals in the packed volume as shown in Figure 5. For the anaerobic packed apparent (unpacked) volume the flow rate corresponding to the maximum loading rate was 240 liters/hr. (63.6 gallons/hr.) and the apparent hydraulic residence time was 5 hrs. Three identical units were constructed and operated in parallel. Design flow rates were 240 liters/meter, 120 liters/hour and 60 liters/hour.

## Pumps and Flow Control

Pumps used were Jabsco model B3-M6 (Jabsco Pump Company, Costa Mesa, California) rated at 38 liters/minute (10 gpm) at zero head. Flow rate control was provided by use of cam timers. For example, to maintain an average flow of 4540 liters/day for the activated sludge systems timers with a five minute cycle were chosen and operated in

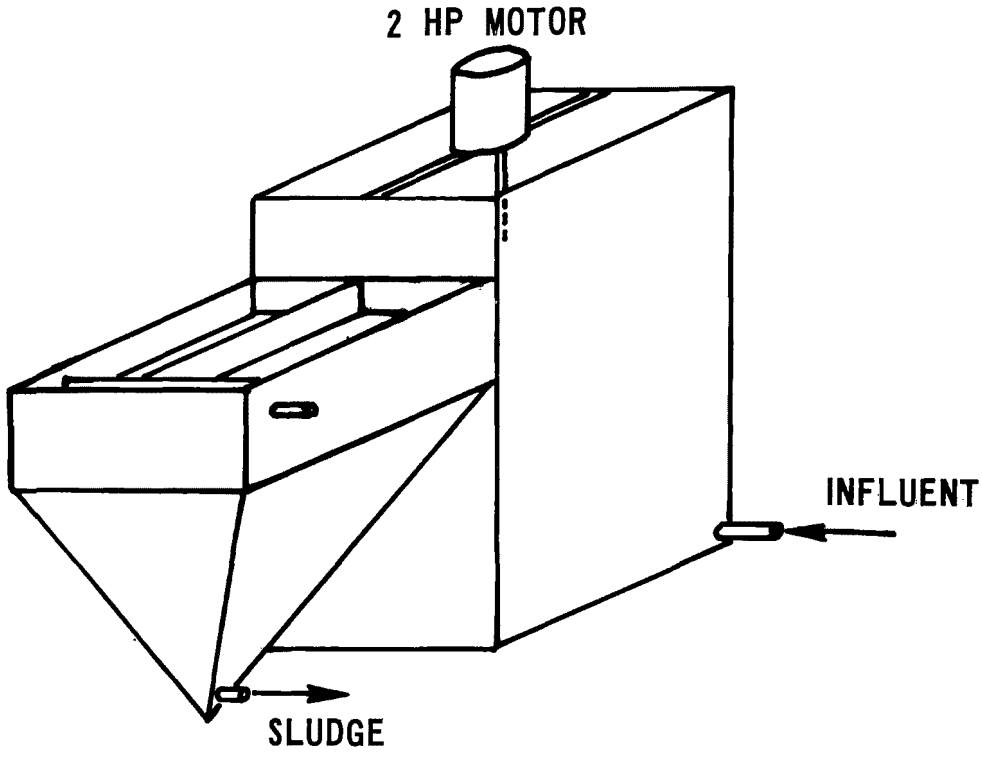


FIGURE 4. AEROBIC TREATMENT UNIT

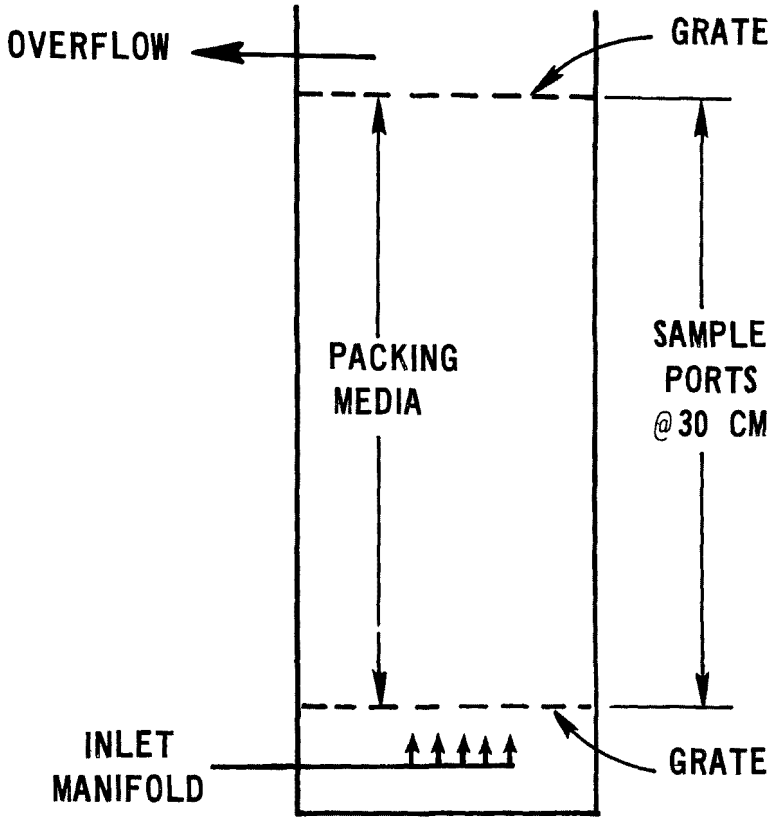


FIGURE 5. ANAEROBIC PACKED BED REACTOR

the on position for 25 seconds per cycle. Design flow rates reported here are based on pump characteristic curves, measured head, and the timing cycles used. Other flow rates reported were measured on a volumetric basis over several cam timer cycles.

## Pomace Fermenters

Pomace fermentation is conventionally carried out after mixing of the pomace with stems in the disintegrator and washing with water (see Figure 1). The pomace is partially fermented prior to this operation, but residual sugars and sugars washed off of the stems are converted to alcohol at this point. Alcohol concentration is low (1 to 4%) but economic recovery is possible. Because most of the potential alcohol is in the pomace, the possibility exists for fermenting the pomace without the stems and wastewater and greatly decreasing the wastewater flow rate. Temperature control is the primary concern because the material being fermented would be pressed pomace which is similar to a filter cake. The purpose of the experiments in this portion of the study was to determine the amount of heat generated during fermentation with the objective of developing a proposed method of temperature control.

Pilot scale pomace fermenters were designed and constructed during the second year of the project. Because the primary problem was expected to be temperature increases within the fermenting pomace, the units were designed to minimize heat exchange and allow semicontinuous temperature monitoring at four points. Three identical fermenters were constructed. Each cylindrical plexiglass unit had an inside diameter of 28 cm, an insulation space between the inner and outer walls of 1.8 cm, and a length of 61 cm.

Thermometers were inserted three points along the length of the cylinder. The thermometer position could be adjusted radially allowing development of complete temperature profiles.

## LABORATORY SYSTEMS

Laboratory scale work included aerobic biological treatment, anaerobic biological treatment, and solids removal studies. Stillage used in all of the laboratory studies was obtained at the Gallo Winery in Fresno, transported and stored on the Davis Campus at -30°C. The stillage used in the solids removal studies was dewatered by flash evaporation at the winery and reconstituted after thawing, prior to use. Tap water was used in reconstituting the stillage and the dilution factor used was such that the filtrate COD was 18000 mg/L. Changes in stillage characteristics during storage were not detected.

### Activated Sludge

Laboratory aerobic biological treatment systems consisted of two 3.5 liter plexiglass stirred reactors in series without cell recycles. Diffused air was the source of oxygen and mixing energy. Stillage fed to the units was the supernatant liquor from thawed 20 liter aliquots which had been allowed to settle for 24 hours. A variable speed Masterflex tubing pump, model V-13 (Cole-Parmer Co., Chicago) controlled by a cam timer was used to feed the stillage to the system. Temperature was maintained at 23°C by housing the entire experimental system in a controlled temperature room.

The pH of stillage fed to the aerobic units was not adjusted, but nitrogen (3.6 g/L $NH_4Cl$) was added. Hydraulic, and therefore mean

cell residence times used, ranged from 1.1 days to 4.7 days in each unit. Thus, total residence time in the two reactor series varied between 2.2 and 9.4 days. System pH, COD and mixed liquor suspended solids concentration was monitored until steady state conditions were achieved at each residence time studied. These conditions were maintained for one to two weeks of operation.

## Anaerobic Treatment

The laboratory anaerobic treatment studies were conducted using 1.5 liter continuously mixed batch reactors. Operating temperature of the units was 57°C (135°F). This temperature was chosen on the basis of stillage being available at a temperature of 66°C at the still. Operation was on a daily fill and draw basis. Three residence times, 15 days, 30 days and 60 days were used. Settled stillage was used in these experiments which had a COD value of 15,500 mg/L. Gas production in the anaerobic systems was measured by liquid displacement.

## Solids Removal Studies

Solids concentration and removal methods considered for pomace stillage were: a) coagulation with polyelectrolytes, flocculation and sedimentation, b) centrifugation, c) centrifugation with polyelectrolyte addition, and d) detartration with polyelectrolyte addition, flocculation and sedimentation. Plain sedimentation and dissolved air flotation were considered by Chadwick[3] and were not included in these studies. All of the solids removal studies were conducted at room temperature (approximately 21°C).

Coagulation -

Coagulants used were limited to polyelectrolytes because of the low pH and high acidity of the stillage. Use of cationic, anionic and nonionic polymers was investigated. Polymers used are listed in Table 2.

TABLE 2
POLYELECTROLYTES USED IN COAGULATION EXPERIMENTS

| CATIONIC | ANIONIC | NONIONIC |
|---|---|---|
| Purifloc C-31 | Purifloc A-23 | Purifloc N-12 |
| Purifloc C-41 | Amoco Anionic | Amoco Nonionic |
| Amoco Cationic | Separan MC 200 | Separan MGL |
| Nalco 610 | | Nalco 607 |
| Nalco 610-HD | Bentonite | Nalco 634 |
| | Nalco 671 | |
| | Nalco 676 | |

Coagulation tests were run using Standard Jar Test Apparatus and methods[19] except for pH adjustment. Polyelectrolyte concentrations used were in the range of 10 to 200 mg/L. In each case an appropriate quantity of polyelectrolyte was added to 500 ml of reconstituted stillage. The mixture was then stirred at 100 rpm for 10 minutes and flocculated for 2 minutes at 20 rpm. After flocculation the mixture was poured into a 500 ml graduated cylinder and allowed to settle. Clear water interface height and supernatant liquor COD were measured as functions of time.

Centrifugation -

Variables considered in the centrifugation studies included rotational speed and time of centrifugation. All experiments were conducted using 75 ml samples and an IEC model UV centrifuge. Rotational speeds and run times used were 1200, 1800, 2400 and 3000 rpm, and 1, 2, 4, 8 and 15 minutes, respectively. Cake moisture (in %), supernatant liquor COD and supernatant liquor suspended solids concentration were recorded as functions rotational speed and run time.

Comparison of centrifuges is made by using an index termed relative centrifugal force (RCF)[22]:

$$RCF = 0.00001117\ r\ N^2 \qquad (1)$$

where:

r = radius in cm

N = speed of rotations in RPM.

Proper test data includes the size of tubes used, time of centrifugation, and RCF values at the tip of the tube and free surface of the liquid. The usefulness of RCF is that when data is to be compared or developed using different apparatus the depth of liquid at a given angular velocity which will correlate with previous results can be determined by measuring the radius to the tip of the tube of the centrifuge. The liquid depth will be given by:

$$\text{Liquid Depth} = \left(\frac{\text{Reference RCF at tip} - \text{Reference RCF at surface}}{\text{RCF at Tip}}\right)\left(\frac{\text{Tip}}{\text{Radius}}\right) \qquad (2)$$

Speeds used in these studies corresponded to RCF values of 259 x g

to 313 x g at 1200 rpm, 584 x g to 705 x g at 1800 rpm, 1040 x g to 1262 x g at 2400 rpm, and 1620 x g to 1956 x g at 3000 rpm.

Several centrifugation experiments were run using 8 mg/L of Amoco cationic polyelectrolyte as an additive. Run time was varied between one and fifteen minutes, but rotational speed was held constant at 2400 rpm. Run time was calculated from the point that the desired speed was attained. Thus, acceleration and deceleration time was not included.

Detartration with Polyelectrolyte Addition -

The pH of California brand stillage is approximately 3.5, and tartrate is present almost entirely as potassium bitartrate (cream of tartar). Soluble salts are precipitated as calcium tartrate when calcium hydroxide added as lime is used to neutralize the stillage in the presence of soluble calcium ion (as calcium chloride). If calcium chloride is not present only about one half of the bitartrate will be removed. The other half will remain in solution as the very soluble potassium tartrate. The equations below illustrate the necessity for the addition of soluble calcium salt:

$$2KHC_4H_4O_6 + Ca(OH)_2 \rightleftarrows CaC_4H_4O_6 + K_2C_4H_4O_6 + 2H_2O \quad (3)$$

$$K_2C_4H_4O_6 + CaCl_2 \rightleftarrows CaC_4H_4O_6 + 2KCl \quad (4)$$

Tartrate recovery is complicated by the fact that extraneous materials are also precipitated. This results in contamination of the product. Where tartrate is to be a saleable product, the process pH should not be above 4.5 (approximately the iso-electric point of calcium tartrate).

If tartrate recovery is not important, precipitation of the extraneous materials may be advantageous in COD reduction.

Equal weights of calcium chloride and calcium hydroxide (varying from 0.25 grams to 2.25 grams) were added to 500 ml samples of reconstituted stillage. In each case, the mixture was mixed at 100 rpm for 30 minutes and flocculated for five minutes at 20 rpm. Mixing time was long because the calcium chloride and calcium hydroxide did not readily dissolve. Filtered COD and pH values were measured.

Polyelectrolyte coagulation of the detartrated stillage was also studied. Purifloc A-23 anionic polyelectrolyte added to the detartrated stillage (pH 11) in concentrations of 1, 2, 4, 10, 14, 20, 26, 30, 40, 50 and 70 mg/L, mixed at 100 rpm for ten minutes and flocculated for two minutes at 20 rpm. The mixture was then poured into a graduated cylinder (500 ml) and interface height and supernatant liquor COD were measured as functions of time.

Removal of Solids from Supernatant Liquor -

Settled or centrifuged stillage still contains high suspended solids concentrations. Removal of these materials by coagulation with polyelectrolytes, flocculation and sedimentation, dissolved air flotation and detartration with polyelectrolyte addition was studied. The supernatant liquor used was obtained by centrifuging 150 ml samples of reconstituted stillage for two minutes at 2400 rpm. Supernatant liquor suspended solids concentration was of the order of 5800 mg/L. Variables considered in the coagulation of stillage supernatant were cationic polyelectrolyte and bentonite concentrations. Nalco 610 polyelectrolyte was used in concentrations of 5, 15, 38,

60 and 100 mg/L. Bentonite concentration used were 10 and 20 mg/L. In each case, the coagulants were added to 200 mg/L of stillage supernatant, mixed at 100 rpm for ten minutes and flocculated for 2 minutes at 20 rpm. The mixture was then poured into a 250 ml graduated cylinder and settling rate and supernatant liquor COD were measured as functions of time.

Dissolved air flotation was studied using a range of coagulant concentrations, cell pressures and recycle ratios. The coagulants, Amoco nonionic and Amoco cationic polyelectrolytes were used in concentrations of 1, 3 and 7 mg/L. Cell pressures used were $2.1 \times 10^6$, $2.8 \times 10^6$, $3.5 \times 10^6$ and $4.1 \times 10^6$ dynes/cm$^2$. The recycle fluids were process supernatant and tap water and ratios of 3.3 and 5 were used.

Detartration of stillage supernatant with polyelectrolyte additive was studied using the same range of calcium chloride and calcium hydroxide concentrations used in the stillage detartration studies (500 to 4500 mg/L). In each case, the mixture was mixed for 20 minutes at 20 rpm. Filtered COD and pH values were measured. A polyelectrolyte (Purifloc A-23) was then added to the detartrated supernatant liquor (which had a pH value of 11). The mixture was stirred at 100 rpm for five minutes and flocculated at 20 rpm for one minute and poured into a 250 ml graduated cylinder. Supernatant liquor COD and suspended solids were measured after settling.

## FIELD SAMPLING PROCEDURES

Samples were taken from the sedimentation influent, activated sludge mixed liquor and sampling ports of the anaerobic packed beds.

Settling rate pH and temperature were measured immediately. During 1971 samples to be analyzed for chemical oxygen demand and suspended solids were packed in ice and immediately transported to Davis for analysis. During the 1972 operating season suspended solids measurements were made in the enology laboratory of California State University, Fresno. Chemical oxygen demand samples were frozen and analyzed at a more convenient time. This procedure was found to be acceptable by Chadwick and Schroeder[3].

Stillage used in the laboratory studies was put into clean, 55 gallon drums, transported to Davis and stored at -55°C until needed. The laboratory solids removal studies made use of stillage which had been dewatered at the winery, stored at -30°C on the Davis Campus and reconstituted in small batches by thawing and diluting with five parts tap water to one part stillage concentrate. A reconstituted stillage filtrate chemical oxygen demand value between 18000 and 20,000 was used as a guide in determining the dilution ratio.

## ANALYTICAL METHODS

Temperature was determined with a thermometer in both field and laboratory studies. Hydrogen ion concentration (pH) was measured in the field by a portable pH meter, and in the laboratory using a conventional laboratory instrument (Leeds and Northrup). Dissolved oxygen measurements were made with a YSI model 54 portable dissolved oxygen meter.

Suspended solids were measured using 0.45 micron pore diameter, silver membrane filters (selas Flotronics - Springhouse, Pennsylvania) and a conventional membrane filter apparatus. Except for the choice of

filters, the method used followed that of the EPA chemical analysis manual[18].

Chemical oxygen demand was measured according to procedures given in Standard Methods[19].

# SECTION V

# EXPERIMENTAL STUDIES AND RESULTS

## 1971 PILOT PLANT STUDIES

Work on the project between July 30, when the University received notice that the grant would be awarded, and December 31, 1971 was entirely related to the Fresno operation. Prior to July 30, treatment system plans (Figures 3, 4, 5) were drawn up and equipment could not be made until a project account was set up. Because of the later than expected starting date unit construction in the shops was considerably delayed. Two of the five sedimentation-holding tanks planned and all three of the anaerobic treatment units were completed early in September and moved to Fresno on September 10th. The still had been put into operation less than a week prior to the move and thus the anaerobic treatment processes were in operation for essentially the entire season. Although completion of the aerobic units was slow the controlling factor was the arrival of the gear motors. Five, two horsepower turbines were ordered in August from a company which stated they were in stock. After repeated conversations two motors arrived during the last week in September and the supplier indicated the other three would not be available until November. The order was then switched to a second supplier, but the last three gear motors did not arrive until the final week of October. For this reason only the large aerobic treatment unit was put into operation.

Because a number of questions needed to be answered before the 1972 operating season a quantity of stillage was stored for use in the

laboratory at Davis. Most of stillage stored was collected by the staff of the Gallo Winery and kept under refrigeration until transfer to Davis was convenient (see Section IV for conditions of storage).

One of the primary reasons a two year project period was proposed was the assumption that operational problems could arise. Because of the short time period in which the still is in operation any delays are serious. Four problems were encountered in 1971 at Fresno: pH control, flow rate control, temperature control, and removal of suspended solids. The most severe problem was flow rate control. At no time during the fall was a steady controlled flow obtained, and therefore the treatment units could not be left unattended. Whenever the operator left the site for more than a few minutes he was forced to shut the entire operation down. Initially the only control device was a valve between the stillage sewer (a cast iron force main) and sedimentation tank. Flow was intermittent, even with the valve wide open. Because of the intermittent flow and the fact that pipe pressure seemed to vary a pump with a throttling valve was then tried but proved unsatisfactory also. A major result of the flow problems was that design flow rates were never reached.

Temperature control was planned for the project and heaters, cooling coils and control devices were purchased. Unfortunately the heaters for the anaerobic treatment unit (which was to operate at 50°C) did not arrive until November, far too late to install them in the units. Stillage temperature at the pilot site varied widely also, and because of the flow control problem the sedimentation tank was allowed to cool overnight, every night. Anaerobic process temperatures dropped below 70°F on occasion. Heating coils were used in the sedimentation tanks

in November and this improved the situation, raising the temperatures in the anaerobic units into the low 80's, but not enough to generate satisfactory fermentation rates.

Control of pH was expected to be a problem, stillage acidities reported in the literature are in the range of 1200 mg/L to 3000 mg/L (Table 1), and therefore considerable difficulty in neutralization might be expected. Stillage acidity is due to organic acids which can be oxidized, and therefore if the mass input rate does not exceed the oxidation rate pH problems should not result. Because of the low operating temperatures the anaerobic units were not able to operate at design rates and pH control, using sodium hydroxide and ammonium hydroxide (the nitrogen source), was initiated. The program was successful and the sedimentation tank pH was maintained above 6.0 for most of October and November.

Suspended solids removal is a basic part of the waste treatment process. Because of the nature of the suspended solids problem with stillage (high concentration and high fraction with near colloidal size) biological breakdown was considered a possible method of removal. Good solids removal is achieved by gravity sedimentation (up to 90%) if settling velocities of the order of 1.8 to 2.1 meters/day (six to seven feet per day) are used. Conventional (eg. municipal) treatment processes utilize a settling velocity range of 32 to 49 meters/day (105 to 160 ft/day). Sedimentation velocities of stillage are low because of the high concentration of solids (settling rates are inversely related to solids concentration), and it should be remembered that even when 90 percent of the solids have been removed, the supernatant liquid has a suspended solids concentration on the order of 1700 mg/L. On a relative volume basis the thickened sludge is

about forty to fifty percent of the original volume, thus gravity settling is not a suitable process.

Biological activity of the treatment processes can be estimated from the chemical oxygen demand data reported in Tables 3 and 4. Table 3 contains COD data on samples filtered through a 0.45 μ filter. The anaerobic treatment processes were operated as upflow units, thus the difference between bottom and top filtrate COD readings is equivalent to filtrate biochemical oxygen demand removed or converted because the only method of conversion available was biological. As was stated above, fermentation rates in the anaerobic processes were very slow, but fermentation was occurring as shown in Table 3. Tanks 1, 2 and 3 correspond to flow rates of 60, 120 and 240 liters per hour, respectively.

Organic reductions in the aerobic process were much more satisfactory, particularly because there was no acclimation of the culture to the stillage. This latter fact is important because of the seasonal nature of the waste. As was stated earlier, $NH_4OH$ was added to the reactors as a source of nitrogen. The difference between the inlet tank filtrate COD concentration and the mixed liquor or effluent filtrate COD concentration (they should be approximately the same) is a measure of BOD reduction. Previous studies[3] have indicated that the minimum attainable COD concentration is approximately 1500 mg/L.

Table 4 contains COD data on unfiltered samples. Because process installation was after the grape harvest began, no attempt to leach out soluble organics from the wood chips was made. COD concentrations in the anaerobic processes are extremely high because solids accumulate in the systems. This feature was designed into the processes on the

TABLE 3

COD ANALYSIS OF FRESNO SAMPLES (Filtered Samples - 0.45 μ) mg/L

| | 10/25/71 | 10/29/71 | 10/30/71 | 10/31/71 | 11/1/71 | 11/5/71 |
|---|---|---|---|---|---|---|
| Tank 1 Bot.* | 17,476 | 18,505 | 18,620 | 17,065 | 16,012 | 16,376 |
| Tank 1 Top | 12,122 | 14,876 | 16,028 | 16,531 | 15,245 | 16,473 |
| Tank 2 Bot.* | 17,161 | 17,028 | 17,206 | 19,392 | 16,683 | 16,570 |
| Tank 2 Top | 14,642 | 18,674 | 16,145 | 17,318 | 14,957 | |
| Tank 3 Bot.* | 15,114 | 18,701 | 18,620 | 21,331 | | |
| Tank 3 Top | 15,272 | 16,301 | 16,028 | 15,901 | 15,533 | 16,473 |
| A.S. Effluent | | 4,985 | 4,478 | 5,825 | | 7,073 |
| Mixed Liquor | | 4,669 | 3,889 | 5,825 | 4,698 | 7,461 |
| Inlet Tank | | 15,193 | 15,438 | 14,012 | | 15,795 |
| Raw Waste | 14,721 | | 16,263 | 18,657 | 16,971 | 16,473 |

* Tanks 1, 2 and 3 correspond to inlet flow rates of 60, 120 and 240 liters/hr. respectively.

TABLE 3 Cont.

COD ANALYSIS OF FRESNO SAMPLES (Filtered Samples - 0.45 μ) mg/L

| | 11/6/71 | 11/7/71 | 11/10/71 | 11/11/71 | 11/15/71 |
|---|---|---|---|---|---|
| Tank 1 Bot. | 16,695 | | 18,093 | 17,066 | 18,601 |
| Tank 1 Top | 14,419 | 14,824 | 17,193 | 16,056 | 15,435 |
| Tank 2 Bot. | 15,936 | 16,823 | 17,193 | 16,460 | 16,721 |
| Tank 2 Top | 14,703 | 15,813 | 16,693 | 15,854 | 17,018 |
| Tank 3 Bot. | 16,695 | 17,631 | 17,893 | 17,873 | 19,491 |
| Tank 3 Top | | 16,722 | 16,993 | 17,167 | 15,534 |
| A.S. Effluent | 6,166 | 6,220 | 5,598 | 6,665 | 4,452 |
| Mixed Liquor | 6,166 | 6,523 | 6,097 | 6,867 | 4,452 |
| Inlet Tank | 13,755 | 13,995 | 16,194 | 17,066 | 13,159 |
| Raw Waste | 15,557 | 15,409 | 15,594 | 14,844 | |

TABLE 4

COD ANALYSIS OF FRESNO SAMPLES (Unfiltered Samples) mg/L

| | 10/25/71 | 10/29/71 | 10/30/71 | 10/31/71 | 11/1/71 | 11/5/71 |
|---|---|---|---|---|---|---|
| Tank 1 Bot.* | | | | 56,043 | 67,407 | 48,723 |
| Tank 1 Top | | 33,377 | 51,198 | 35,669 | 41,773 | 34,191 |
| Tank 2 Bot.* | | 48,274 | | 74,768 | 83,502 | 45,685 |
| Tank 2 Top | 29,700 | | 35,339 | | 44,417 | |
| Tank 3 Bot.* | 36,748 | | 58,299 | 65,272 | 62,171 | |
| Tank 3 Top | 15,360 | 34,164 | 35,458 | 34,472 | 40,304 | 33,711 |
| A.S. Effluent | | 12,359 | 14,864 | 13,875 | 17,568 | 14,439 |
| Mixed Liquor | | 14,170 | 15,575 | 16,776 | 18,703 | 16,932 |
| Inlet Tank | | 34,401 | 31,197 | | 30,904 | 35,150 |
| Raw Waste | | | 33,351 | 38,334 | 30,806 | 28,534 |

* Tanks 1, 2 and 3 correspond to flow rates of 60, 120 and 240 liters/hr.

TABLE 4 Cont.

COD ANALYSIS OF FRESNO SAMPLES (Unfiltered Samples) mg/L

| | 11/6/71 | 11/7/71 | 11/10/71 | 11/11/71 | 11/15/71 |
|---|---|---|---|---|---|
| Tank 1 Bot. | 60,947 | 82,598 | 77,712 | 65,113 | 90,666 |
| Tank 1 Top | 32,617 | 24,737 | 36,413 | 42,674 | 23,091 |
| Tank 2 Bot. | 64,594 | 77,091 | 73,312 | 64,114 | 63,545 |
| Tank 2 Top | 38,721 | 34,613 | 41,462 | 44,390 | 47,980 |
| Tank 3 Bot. | | 68,737 | 60,714 | 71,112 | 63,545 |
| Tank 3 Top | | 28,285 | 33,081 | 49,036 | 30,788 |
| A.S. Effluent | 17,985 | 14,670 | 16,621 | 15,712 | 15,794 |
| Mixed Liquor | 17,500 | 14,574 | 18,136 | 20,660 | 13,395 |
| Inlet Tank | 34,458 | 16,499 | 30,759 | | 12,595 |
| Raw Waste | 17,500 | 16,683 | 32,980 | 28,941 | |

assumption that solids degradation would occur during the off season. If a significant degradation does occur a solution to the solids degradation problem using nominal amounts of land will be available.

Data presented in Table 4 for the aerobic process is of interest because the difference between inlet tank COD and effluent COD is greater than that for the filtrate. Thus some breakdown and oxidation of suspended solids occurred. Inlet tank COD can be seen to be about fifty percent in the filtrate and fifty percent in the suspended solids. The data in Tables 3 and 4 indicates that approximately one third of the suspended solids were removed.

## 1972 LABORATORY STUDIES

Aerobic and anaerobic biological treatment studies were run on a laboratory scale during the winter of 1972. Experimental systems and procedures were described in Section IV. Settled stillage supernatant liquor was used as a feed for both aerobic and anaerobic systems. Stillage settling rate varies from sample to sample, but values are uniformly low. Results of a typical settling test on raw stillage are shown in Figure 6. Settling rates measured in the field varied from about 0.18 L/cm$^2$-day (45 gal/ft$^2$-day) down to zero (no clear-water-solids interface formed and very little solids accumulation on the cylinder bottom after a 24 hour period). Supernatant liquor used in the laboratory studies was obtained by filling 20 liter carboys with thawed stillage and allowing the material to settle for 24 hours.

Supernatant liquor COD and suspended solids concentrations were approximately 19,000 mg/L and 12000 mg/L, respectively. Influent

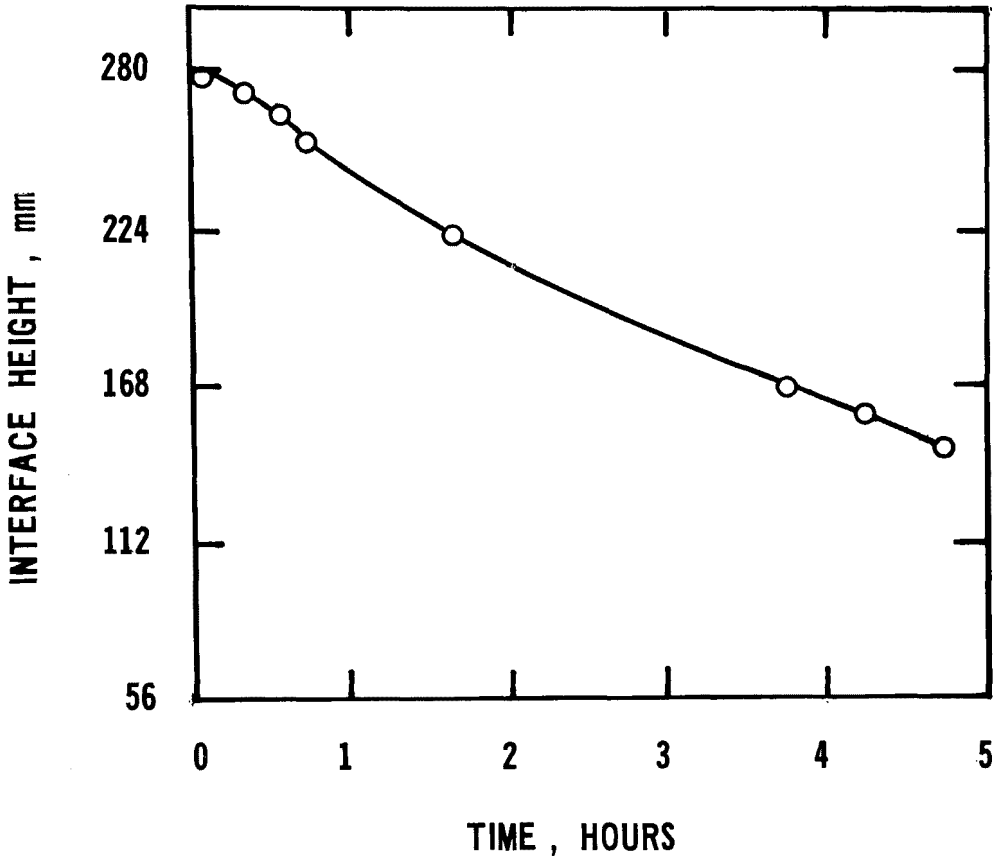


FIGURE 6. TYPICAL RAW STILLAGE SETTLING CURVE

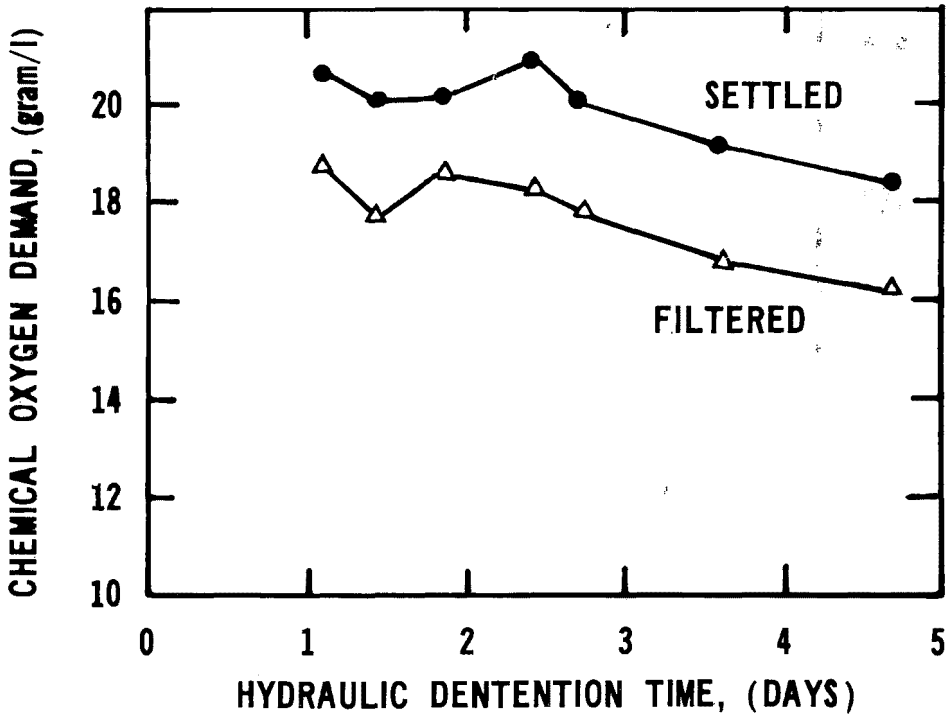


FIGURE 7. STILLAGE FEED CONCENTRATION vs. HYDRAULIC DENTENTION TIME FOR LABORATORY REACTOR

characteristics were constant for each experiment (i.e. each residence time, but varied somewhat throughout the study).

Aerobic Treatment Studies

Aerobic process influent COD concentrations (to the first reactor in the series) are shown in Figure 7 as a function of residence time. The processes were monitored at a given detention time until steady state conditions were attained. This normally took between one and two weeks. Steady state data reported here was taken over a fourteen day period in each case. Under the conditions imposed on the treatment units pH was an excellent indicator of process performance. The low pH and high acidity of stillage is due, primarily, to organic acids. As long as the organic acid conversion rate equals the input rate pH remains near neutral. When input rate exceeds the conversion rate effluent COD concentrations increase and pH values decrease because of the uncoverted organic acids present.

Total reactor suspended solids concentrations are given in Table 5. Effluent quality, as measured by COD concentration was not improved by using the series reactor system. Operation was stable in the first reactor system at residence times down to 1.4 days, although sludge settling deteriorated below values of 1.87 days. Settled effluent COD values were virtually the same for both units. Dissolved oxygen concentrations reflected the relative activity of the two units, as was mentioned previously pH was not controlled, but instead was used as a measure of overloading. Values of pH, dissolved oxygen and effluent COD concentrations are reported for the first reactor in Table 6 and Figure 8, and for the second reactor in Table 7 and Figure 9. Biochemical oxygen demand ($BOD_5$) of the filtered effluent

was approximately 75 mg/L.

Removal rates, cell growth rates and cell yield are important parameters in process design. Normally the rates are believed to be linear functions of the cell mass concentration and this allows the use of specific or unit rates (rate per unit mass of cells). Because the suspended solids concentration of the settled stillage was high (∿1200 mg/L) the mixed liquor suspended solids concentration (MLSS) could not be assumed to represent the cell mass concentration, and thus could not be directly used in calculating unit rates. An estimate of the cell mass concentration was made by subtracting the stillage suspended solids concentration from the MLSS concentration.

Stillage solids include grape pulp, bits of stems and leaves and yeast cell residues, all of which have been broken up during the distilling process. Thus the material remaining in nonsoluble form can be assumed difficult to degrade. Subtraction of the settled stillage SS from the MLSS concentration and using this estimated cell mass concentration to calculate the unit removal rate results in the information presented in Figure 8. Unit growth rate in a well mixed unit without cell recycle is equal to the inverse of the hydraulic residence time. The ratio of the two rates (growth rate/removal rate) is the maximum cell yield, and as noted on Figure 10, the value is 0.37 grams cells produced per gram of COD removed.

Net cell yield is not a constant due to increasing cell maintenance energy requirements with increasing cell age. Estimation of the maintenance energy requirement is difficult with the data presented here because of the high growth rates used in the study and the method of determination of cell mass concentration. Extension of the

TABLE 5

MIXED LIQUOR SUSPENDED SOLIDS CONCENTRATIONS, mg/L

| θ; days | Reactor 1 | Reactor 2 |
|---|---|---|
| 1.09 | 5600 | 7400 |
| 1.41 | 8400 | 6800 |
| 1.87 | 8700 | 6700 |
| 2.42 | 9200 | 6900 |
| 2.75 | 8700 | 6900 |
| 3.58 | 7100 | 5900 |
| 4.67 | 7200 | 6100 |

TABLE 6

OPERATIONAL VARIABLES FOR FIRST LABORATORY REACTOR IN SERIES

| θ, days | DO mg/L | pH | Effluent COD, mg/L Settled | Filtered |
|---|---|---|---|---|
| 1.09 | 1.9 | 5.1 | 12,079 | 7,009 |
| 1.41 | 3.1 | 6.5 | 2,264 | 636 |
| 1.87 | 3.2 | 6.3 | 1,484 | 592 |
| 2.42 | No Data | 6.3 | 3,303 | 763 |
| 2.78 | 3.7 | 6.1 | 2,380 | 643 |
| 3.58 | 5.2 | 6.2 | 1,484 | 537 |
| 4.67 | 6.6 | 6.6 | - | 666 |

TABLE 7

OPERATIONAL VARIABLES FOR SECOND LABORATORY REACTOR /w SERIES

| θ, days | DO mg/L | pH | Effluent COD, mg/L Settled | Filtered |
|---|---|---|---|---|
| 1.09 | 1.9 | 7.3 | 3,149 | 1,031 |
| 1.41 | 6.7 | 6.4 | 1,287 | 664 |
| 1.87 | 6.4 | 6.2 | 1,552 | 773 |
| 2.42 | - | 6.1 | 2,839 | 663 |
| 2.75 | 7.6 | 5.9 | 2,149 | 608 |
| 3.58 | 7.7 | 5.7 | 1,450 | 554 |
| 4.67 | 7.7 | 6.1 | No Settling | 724 |

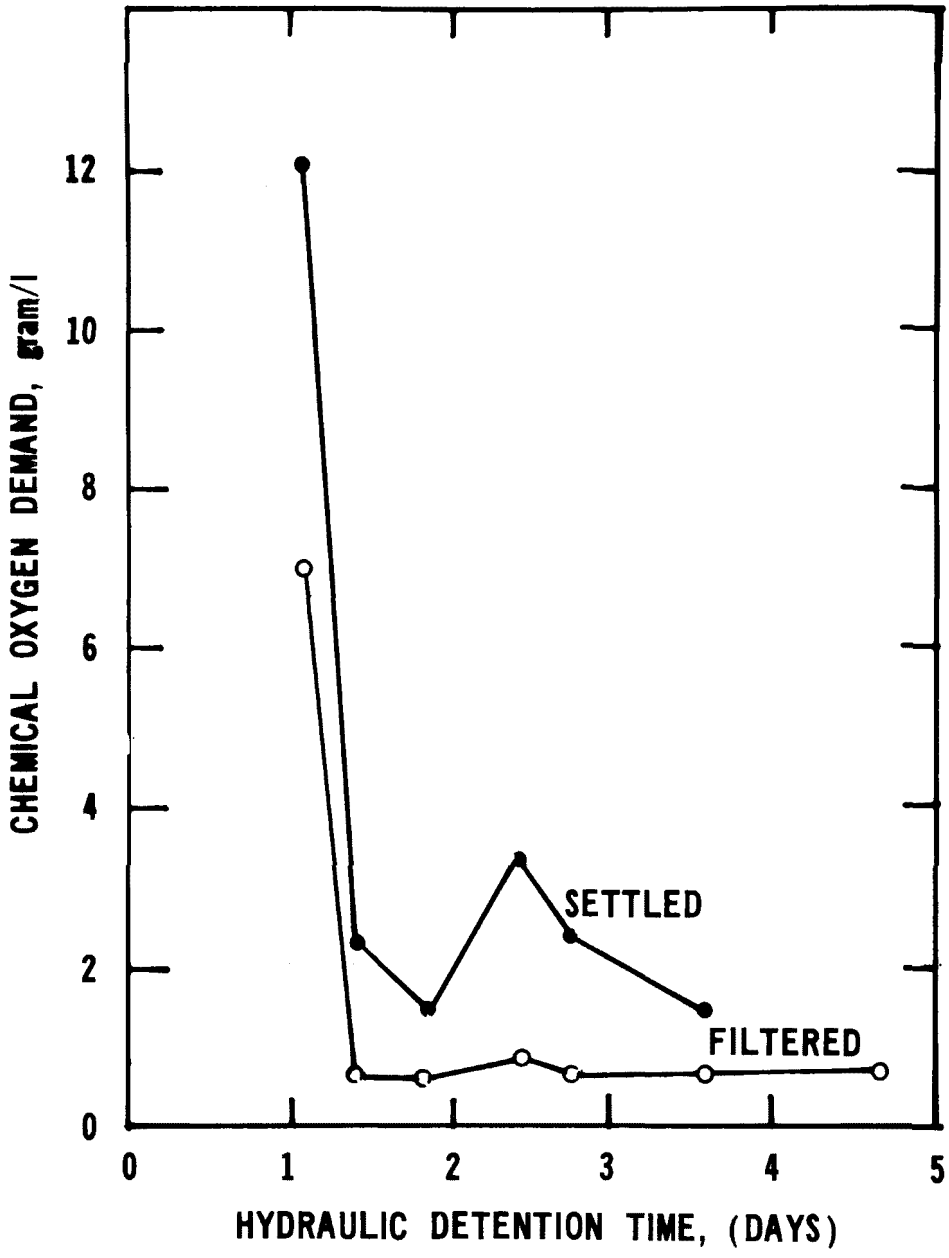


FIGURE 8. EFFLUENT COD CONC. vs. HYDRAULIC DETENTION TIME FOR LABORATORY REACTOR 1

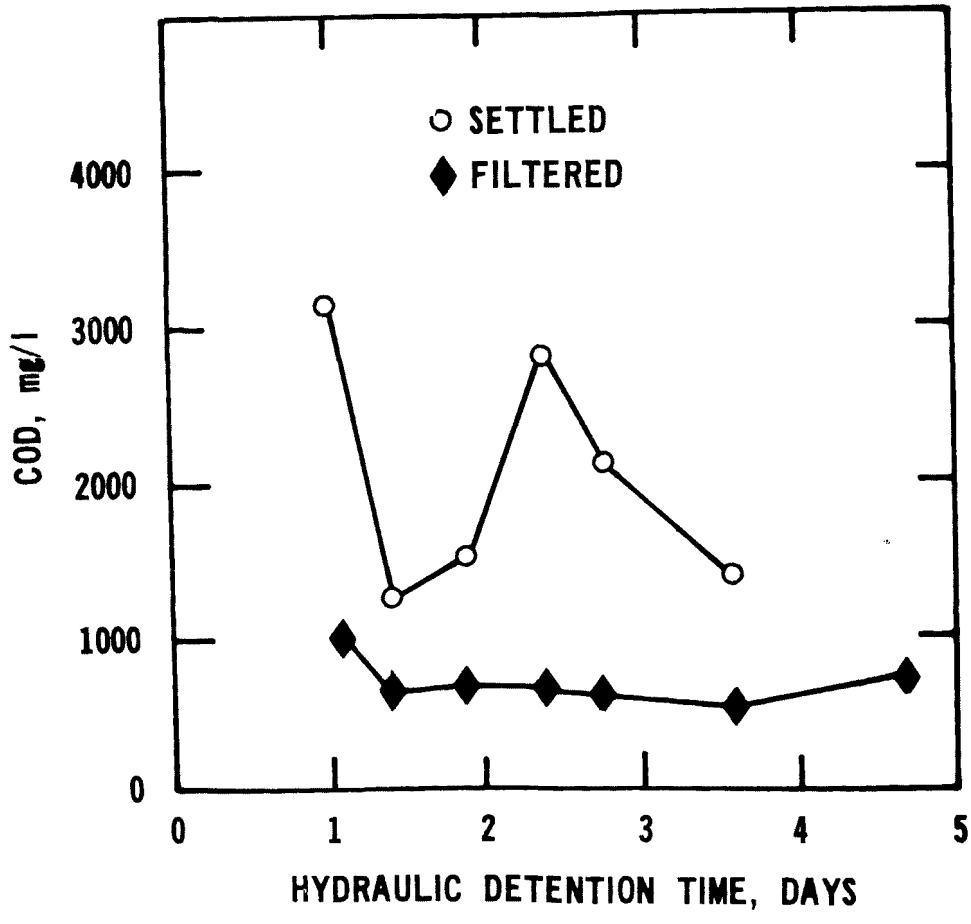


FIGURE 9. EFFLUENT COD CONC. vs. HYDRAULIC DETENTION TIME FOR REACTOR 2 IN SERIES

curve in Figure 10 to the y axis gives a value of 0.01 $day^{-1}$ for the maintenance energy coefficient, $k_d$, in the equation below:

$$\mu = - (Y^* r_{ox} + k_d) = \frac{1}{\theta} \quad (5)$$

where $\mu$ is the unit growth rate and $r_{ox}$ is the unit removal rate. Note that $r_{ox}$ is inherently negative.

Anaerobic Treatment Studies

Methods and procedures used in the laboratory anaerobic treatment studies were described in Section IV. As was noted the feed COD concentration to these systems was 15,500 mg/L.

Results of the anaerobic experiments were not promising. The 15 day residence time unit failed completely, producing very little gas and having an effluent COD of about 14,000 mg/L. At a 30 day residence time COD concentrations were reduced to an average value over a two month period of 5100 mg/L with a gas production of 500 ml/day and a pH of 6.9. The gas production corresponds to 15 $ft.^3$ of gas per pound of COD removed. Operation at a residence time of 60 days did not improve COD conversion measurably, and of course gas production rate decreased proportionately (to approximately 250 ml/day).

1972 PILOT PLANT STUDIES

Unfortunately 1972 was a poor year for grapes in the San Joaquin Valley. The harvest season was very short and stillage production extended only from the last week of August to the second week of

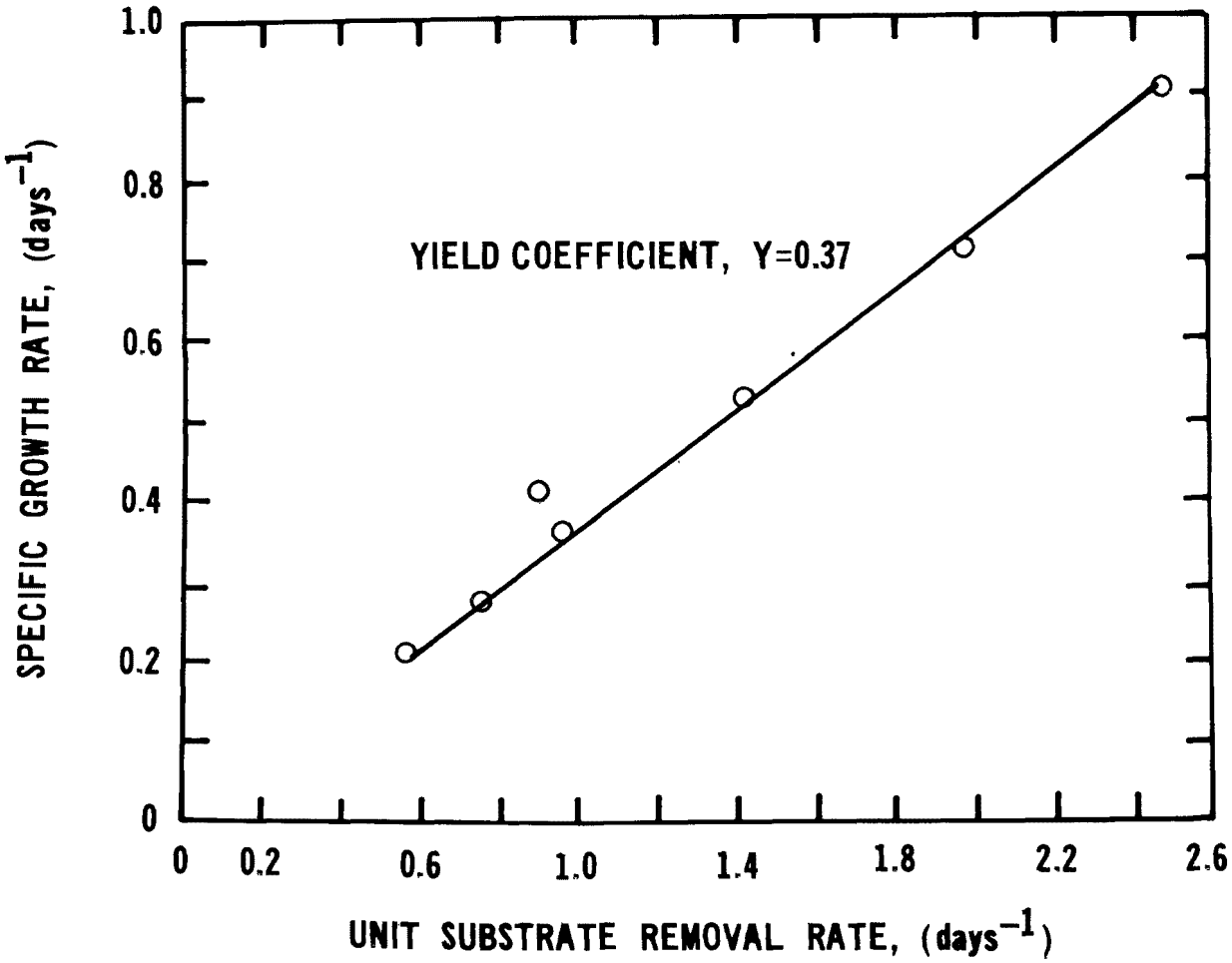


FIGURE 10. SPECIFIC GROWTH RATE VERSUS UNIT SUBSTRATE REMOVAL RATE

October. The pilot plant systems were set up as shown in Figure 5 but operational problems with the small aerobic units (primarily electrical problems with the motors) prevented extensive operation of the entire system.

Loading rates used during the 1972 field studies were chosen on the basis of the laboratory results. Aerobic processes were operated without recycle at a residence time of 3 days. Because the 1971 anaerobic studies run at very short hydraulic residence times were unsuccessful the 1972 studies were run at 3 day residence times.

Nitrogen was not added to the stillage in 1972. Stoichiometric quantities needed would be of the order of 1000 mg/L. Assuming nitrogen present in the stillage is available for synthesis approximately 250 mg/L would still have to be added. This corresponds to 3230 mg/L of ammonium chloride or 2130 mg/L of ammonium nitrate per liter of stillage. For a winery as large as Gallo-Fresno, this would mean adding over 4000 kilograms (9000/lbs.) per day. Because of the large nitrogen requirement it was decided to determine the extent of treatment possible without nitrogen addition. This resulted in a COD/N ratio of approximately 60:1.

A number of operating problems associated with the characteristics of the stillage also occurred. These problems together with the extremely short 1972 operating season hampered data collection.

Two problems of significance were difficulties in pumping stillage and foaming resulting from agitation in the aerobic processes. Pumping difficulties resulted from the impeller sizes used. Clogging by stems and debris was difficult to control, but would not be a

problem with different pumps. Foaming was a far more significant problem and resulted in considerable problems, both for the pilot plant operation and for the winery. Foam layers built up to the point that overflow of the aeration tanks occurred on occasion. In addition the attached secondary clarifier units (which were not used, but through which the flow passed by using a bottom drawoff) developed a layer of thick stable foam which often overflowed the tank also. The foam restricted oxygen transfer by the surface aerators with resulting odor problems, and proved to be an ideal breeding area for flys. Screens were placed over all of the units except the aeration tanks and insecticides were applied. Gallo provided advice and help on the problem, but control was never completely satisfactory. Because of the proximity of the experimental area to the winery there was considerable concern that the fly problem would cause action by the county health officer.

The anaerobic treatment units did not function well during the 1972 season but were improved over 1971. Installation of heaters into the anaerobic processes was impossible without complete draining and media removal. Because of the difficulty of this process, it was decided to place the heaters in the sedimentation tanks. Lack of temperature control was again the primary factor in the poor results, and thus data reported here is qualitative in nature. During the 1971 season which extended from September 5th to November 15th virtually no anaerobic treatment took place as has been noted although the units proved to be excellent sedimentation tanks. During the 1972 season, COD removals were greatly improved. Effluent filtrate COD values as low as 3900 mg/L and ranging up to 7500 mg/L were obtained with influent COD values ranging from 16,000 to 19,000 mg/L. Control of pH was maintained in the pilot units by cutting off

influent flow whenever pH values dropped below 6.5.

Startup of the anaerobic units during the 1972 season was done without adding new cells. The units were left unattended from November 15, 1971 until early August, 1972 when water was added to make up for evaporation. Because of the large quantities of organic solids in the tanks at the end of the 1971 season it was felt that an improved culture would develop during the nonoperational period. Evidently a culture did develop because there were no start up problems and COD conversion was much improved as noted previously.

Aerobic treatment results must be evaluated in light of the lack of available nitrogen. Effluent filtrate COD values ranged from 1460 to 7320 mg/L with an average value of 4600 mg/L. Settled effluent COD values were about 1800 mg/L greater than filtrate COD values. Suspended solids concentrations in the aeration tank varied with the settleability of the incoming stillage. Values ranged from 7500 mg/L to 14,000 mg/L. Data for 1972 correlated very well with that obtained in 1971.

Significant start up problems did not occur either year, and pH control was not a problem as long as the system was not organically overloaded. The major operational problems were stillage solids during periods when stillage settleability was poor, and foaming. Both solids and foaming retard oxygen transfer and thus decrease process efficiency and can result in anaerobic conditions.

## POMACE FERMENTATION

Pomace fermentation is conventionally carried out after mixing with

stems in the disintegrator and washing with water. The pomace is partially fermented prior to this operation, but residual sugars and sugars washed off of the stems are converted to alcohol at this point. Alcohol concentration is low (1 to 4%) but economic recovery is possible. Because most of the potential alcohol is in the pomace the possibility exists for fermenting the pomace only (leaving out the stems and washwater), and greatly decreasing the wastewater flow rate.

Temperature control is the primary concern because the material being fermented would be the pressed pomace (Figure 1) which is similar to a filter cake. The purpose of the experiments in this study was to determine the amount of heat generated during fermentation and propose a method of dealing with the problem. Temperature rise can be controlled by controlled dilution with water providing mixing and cooling (a tumbler type device) or by blowing inert gases through the pomace (eg. water saturated $N_2$ or $CO_2$). Because of the operational problems encountered at Fresno during the 1972 season there was not time enough to run the experiments at the pilot plant site. Material was stored and experiments were then conducted at Davis after the processing season. Unfortunately these experiments failed and there was no way to repeat them. Useable data was not obtained from these experiments.

## COAGULATION STUDIES

Coagulants used were limited to polyelectrolytes because of the low pH and high acidity of the stillage. Although a generally negative charge on the particles was believed to exist, anionic and nonionic polymers were investigated. These included Purifloc A-23, Amoco

Anionic, Separan MG 200, Separan MGL, Purifloc N-12, Nalco 607, Nalco 634, Nalco 671, Nalco 676, Bentonite, and Amoco Nonionic Coagulation was not induced in any of the experiments with the polyelectrolytes.

Cationic polymers used included Purifloc C-31, Purifloc C-41, Amoco Cationic, Nalco 610 and Nalco 610-HD. Jar tests with stillage were run using concentrations ranging from 10 to 200 mg/L of each polyelectrolyte. Coagulation occurred with all of the materials, but the best results were obtained with Purifloc C-41 and Amoco Cationic. Settling tests were then run on a 1/2, 2/3 by weight mixture of Purifloc C-41 and Amoco Cationic at concentration values of 7, 15, 24, 36 and 60 mg/L. Results are shown in Figures 11 and 12. Data points were omitted in Figure 11 because of the number of curves and the fact that scatter was nonexistant (i.e. curves were drawn from point to point).

## CENTRIFUGATION

Results of the raw stillage centrifugation studies are shown in Figures 13, 14 and 15. Satisfactory sludge concentration was obtained even at the lowest speed (see Figure 15) where the cake moisture content and suspended solids removal were both approximately 88% after five minutes. Cake moisture content decreases with time because the cake becomes more compressed. Thus although removal of suspended solids increases with time, the sludge volume does not necessarily increase. For example, when suspended solids removal goes from 86% at 2 minutes to 93% at 15 minutes for the 120 rpm curve, the cake moisture decreases from 88% to 87.1% and the corresponding sludge volume (based on an initial suspended solids

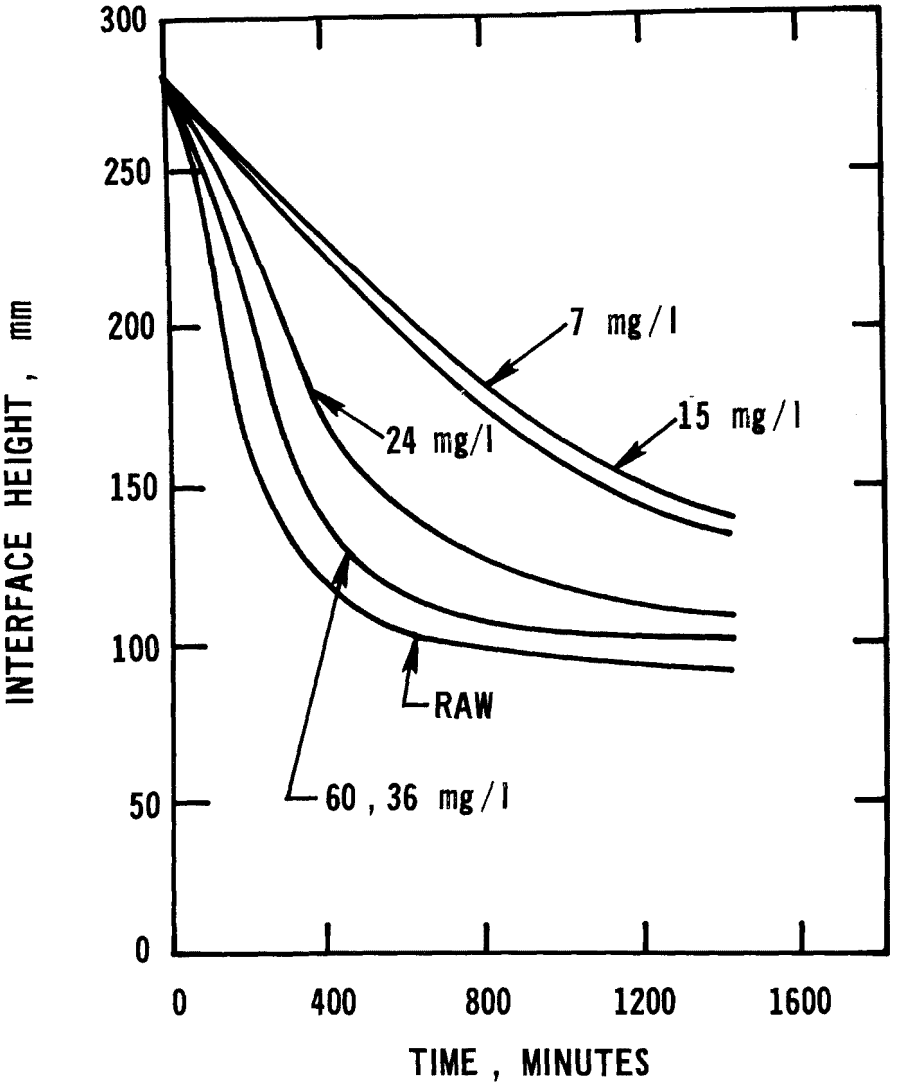


FIGURE 11. SETTLING CURVES FOR PURIFLOC C-41 AMOCO CATIONIC MIXTURE.

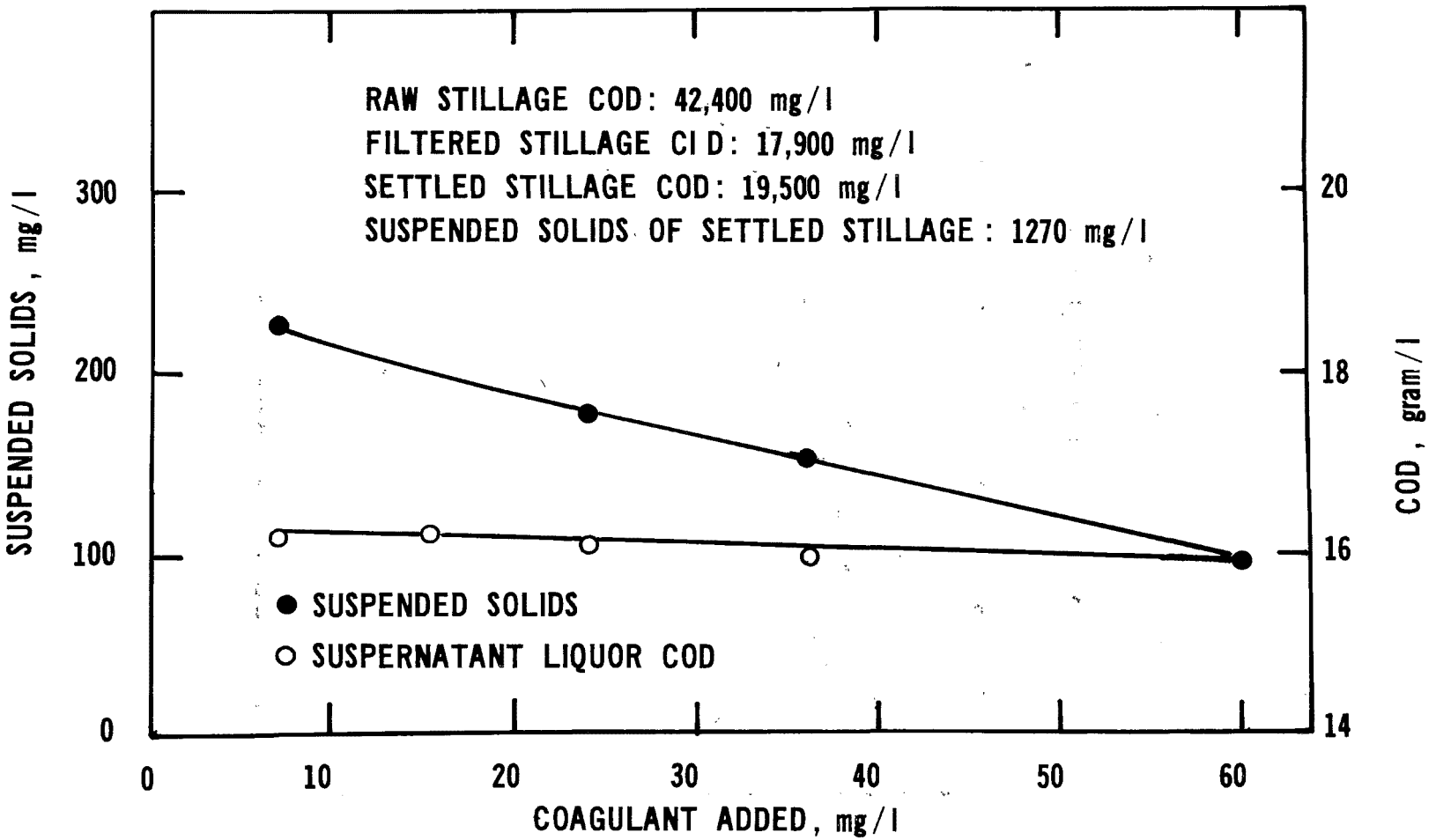


FIGURE 12. EFFECT OF COAGULANT ADDITION ON SUPERNATANT LIQUOR

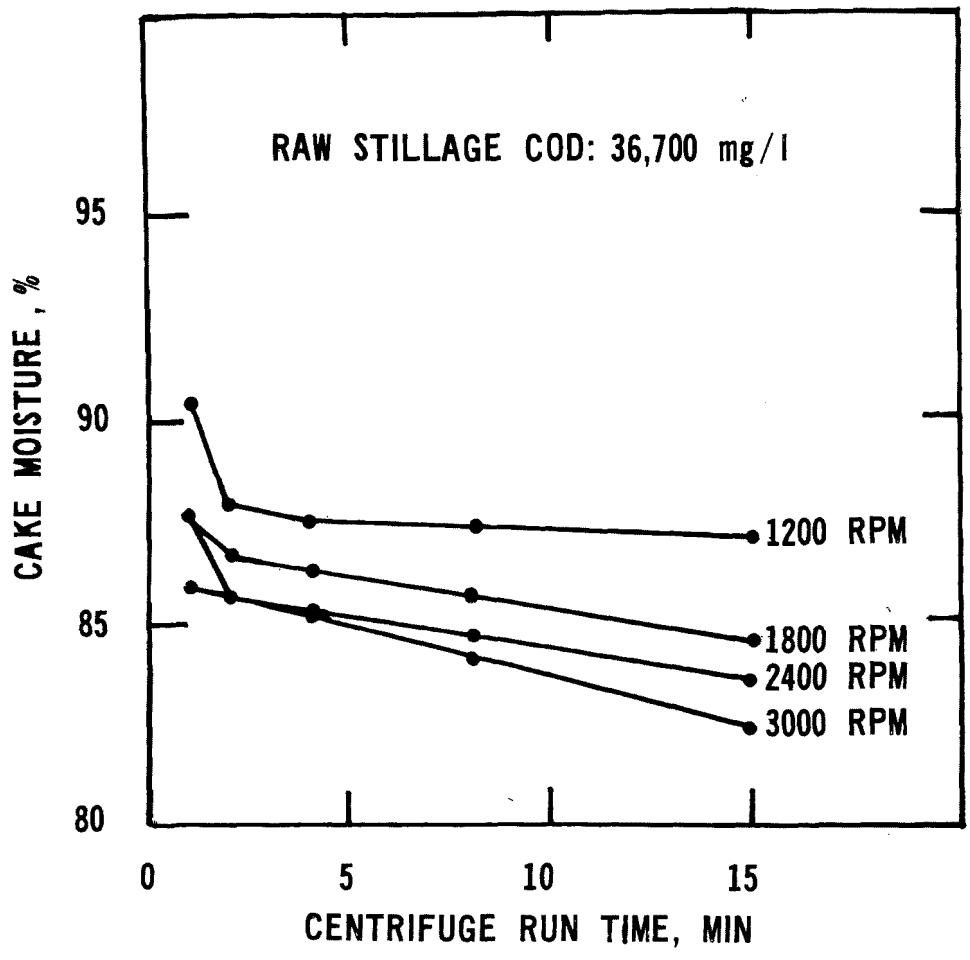


FIGURE 13. CAKE MOISTURE vs. CENTRIFUGE RUN TIME

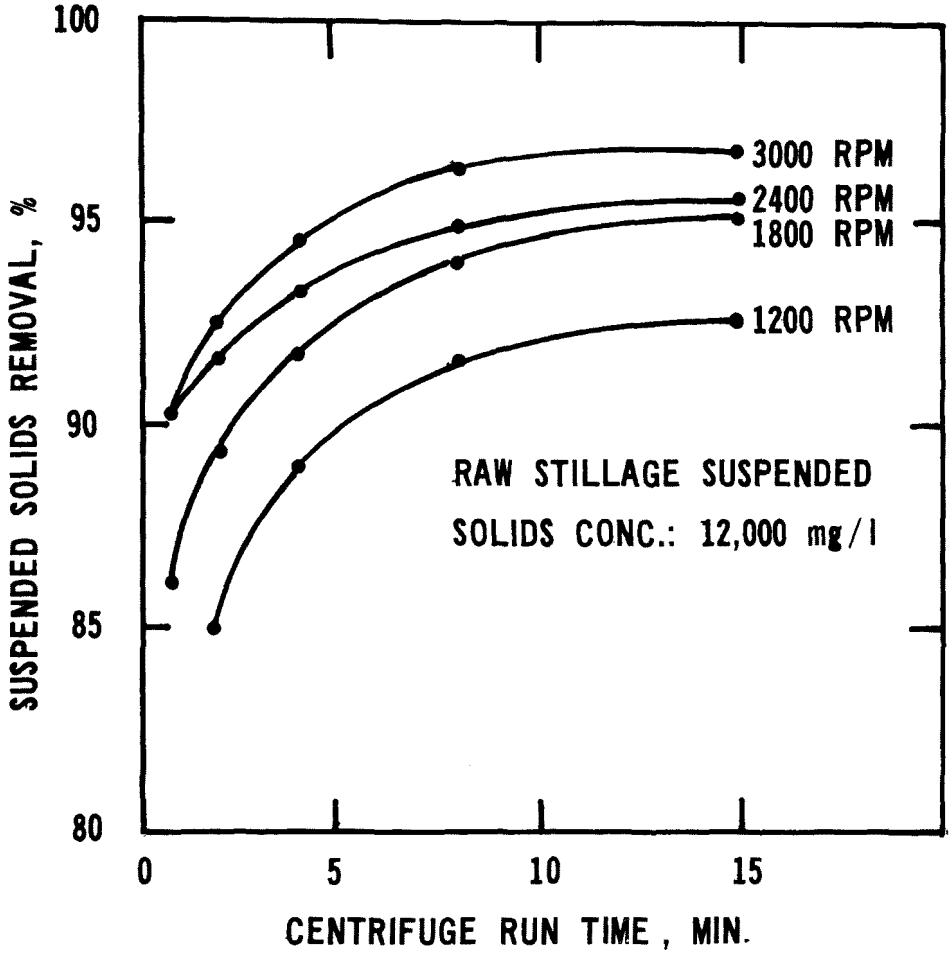


FIGURE 14. SUSPENDED SOLIDS REMOVAL VERSUS CENTRIFUGE RUN TIME

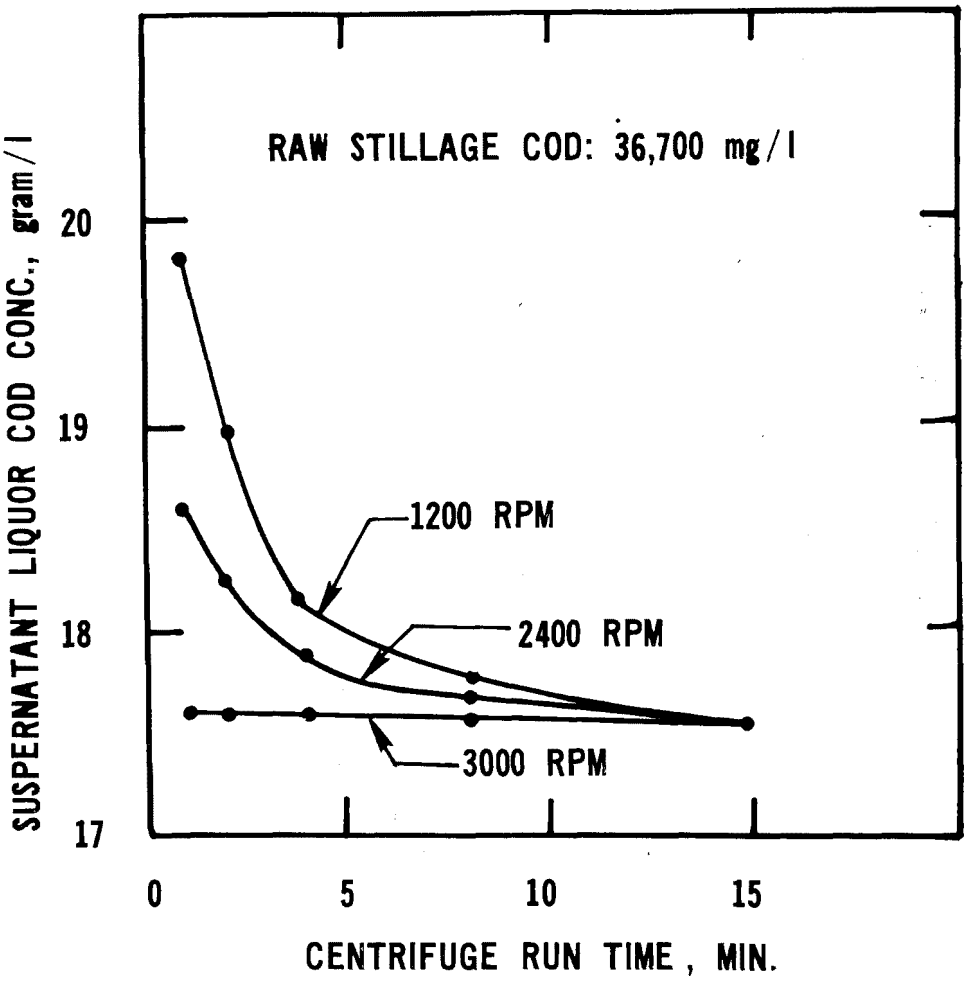


FIGURE 15. SUPERNATANT LIQUOR COD VERSUS CENTRIFUGE RUN TIME

concentration of 11,952 mg/L) increases from 75.2 to 75.7 mg per liter of waste. At 3000 rpm, the sludge volume after one minute run time is 66.4 ml/L of waste. This decreases to 54.9 ml/L of waste after 15 minutes.

Chemical oxygen demand values also decreased sharply after centrifugation. Rotational speed was held constant at 2400 rpm (corresponding to a RCF value of 1040 x g to 1262 x g). Run times used were 1, 3, 6, 10 and 15 minutes. Amoco cationic polyelectrolyte was used at one concentration, 8 mg/L. Cake moisture (in %), COD, and suspended solids concentration were measured as functions of time. Results are shown in Figures 16, 17, 18.

Results of the experiments on detartration of the raw stillage followed by polyelectrolyte addition are shown in Figures 19 and 20. Figure 19 shows the relationship between the quantity of calcium hydroxide and calcium chloride added and the resulting supernatant liquor pH and COD. The curves in Figure 20 are the result of adding an anionic polyelectrolyte (Purifloc A-23) to the detartrated stillage at a pH of 11. Polyelectrolyte was added in concentrations of 1, 2, 4, 10, 14, 20, 26, 30, 40, 50 and 70 mg/L, but only four curves are shown in Figure 20 because many of the curves fell on top of one another. It should be noted also that the ordinate in Figure 20 does not go to zero.

## REMOVAL OF SOLIDS FROM SUPERNATANT LIQUOR

Solids concentration and removal methods for pomace stillage supernatant were: a) coagulation with polyelectrolytes, flocculation and sedimentation, b) dissolved air flotation with and without

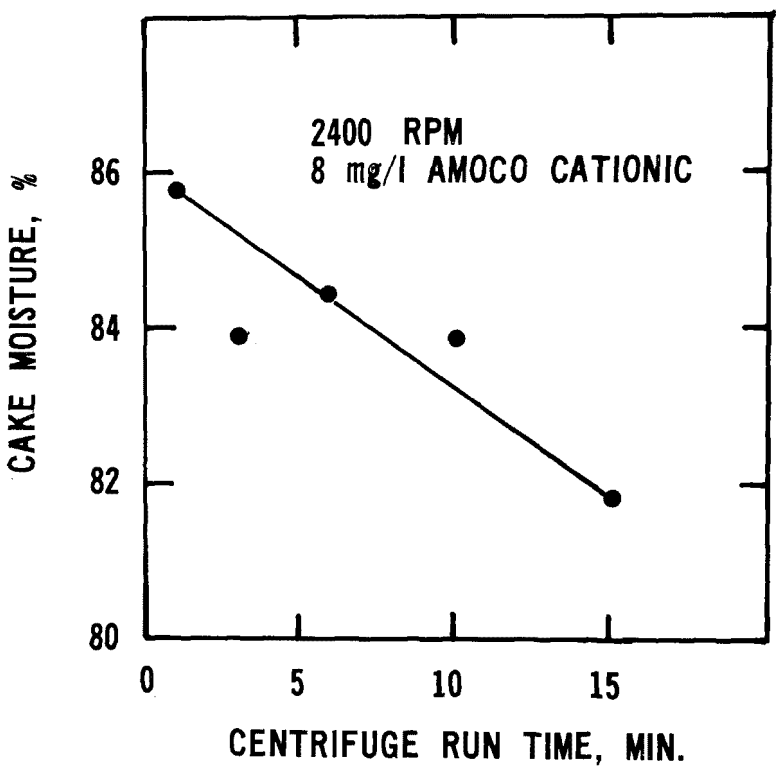


FIGURE 16. CAKE MOISTURE vs. CENTRIFUGE RUN TIME

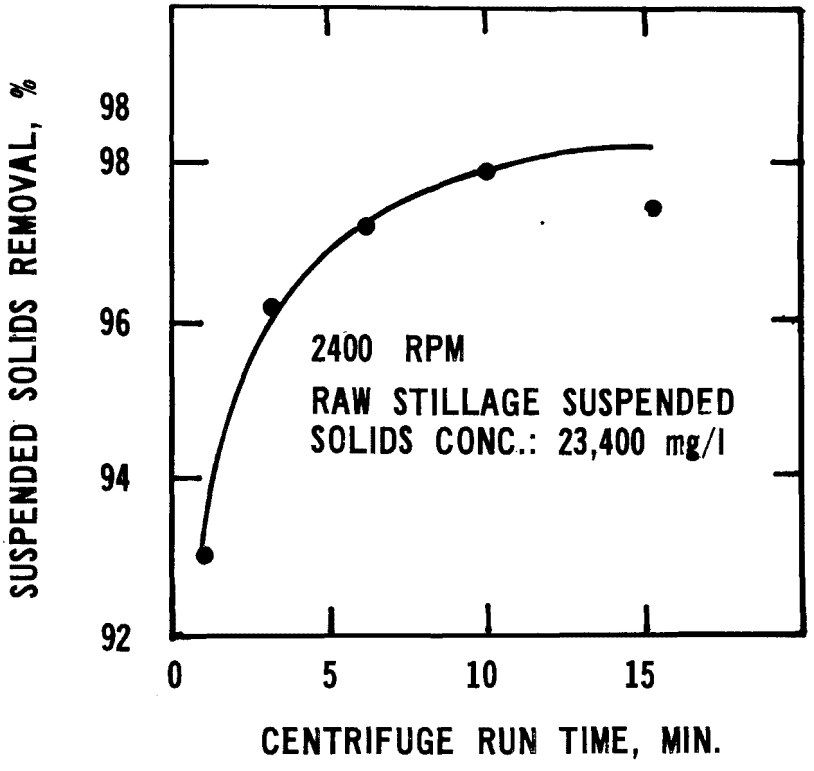


FIGURE 17. SUSPENDED SOLIDS REMOVAL VERSUS CENTRIFUGE RUN TIME

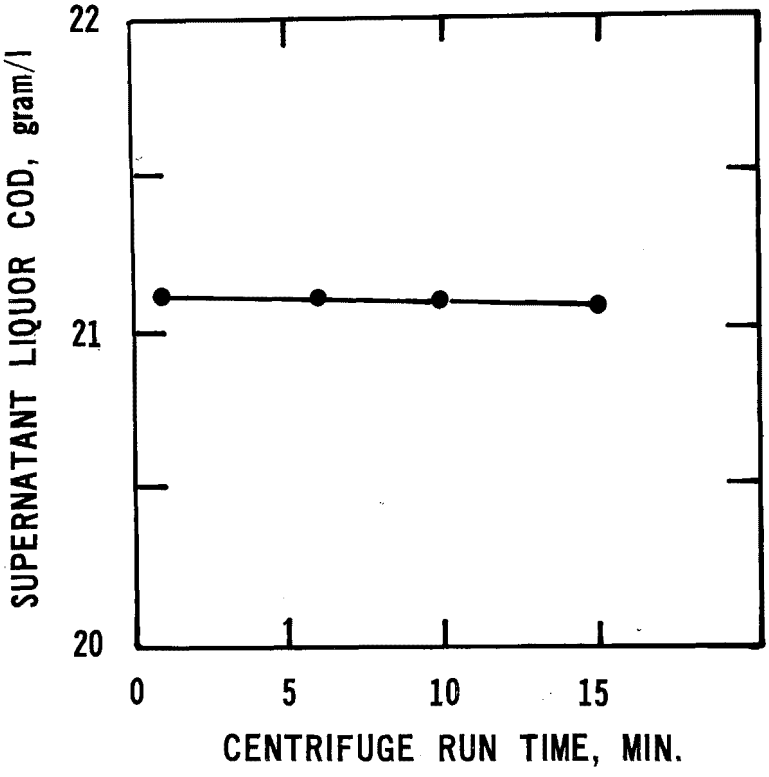


FIGURE 18. SUPERNATANT LIQUOR COD VERSUS CENTRIFUGE RUN TIME

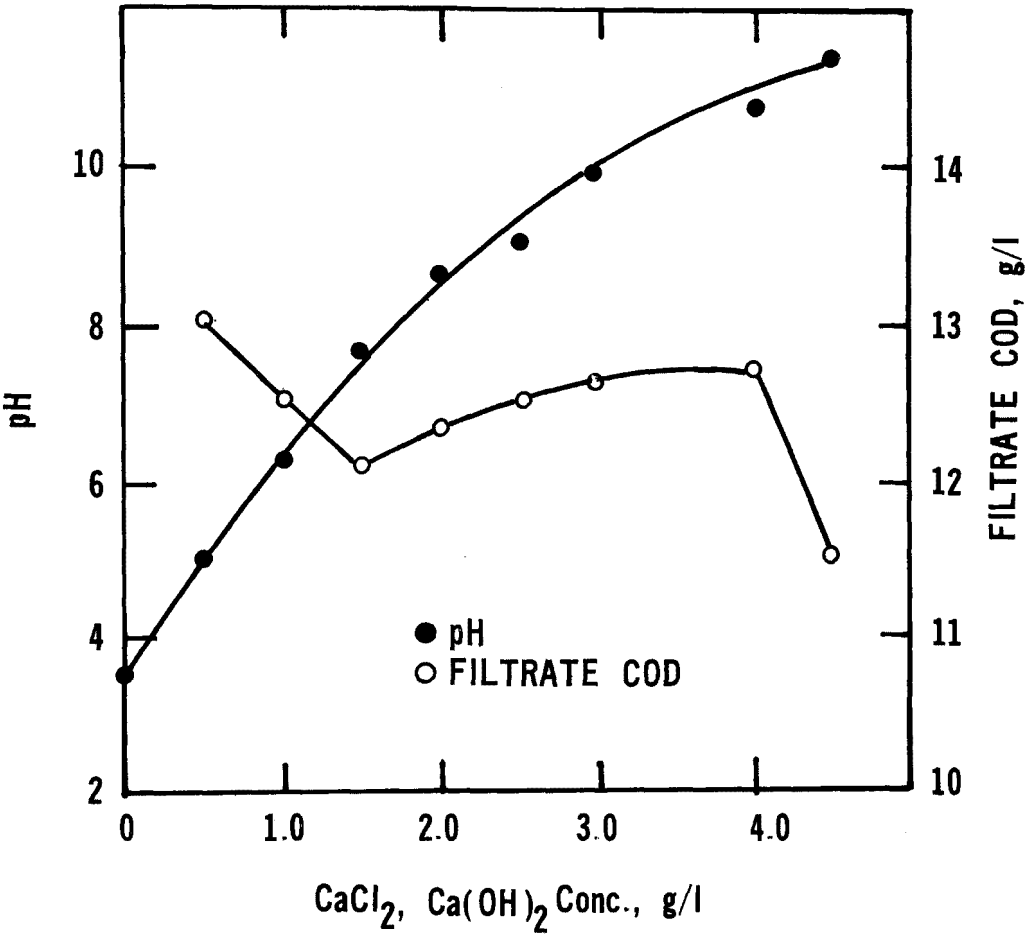


FIGURE 19. SUPERNATANT LIQUOR pH AND FILTRATE COD vs. $CaCl_2$ and $Ca(OH)_2$

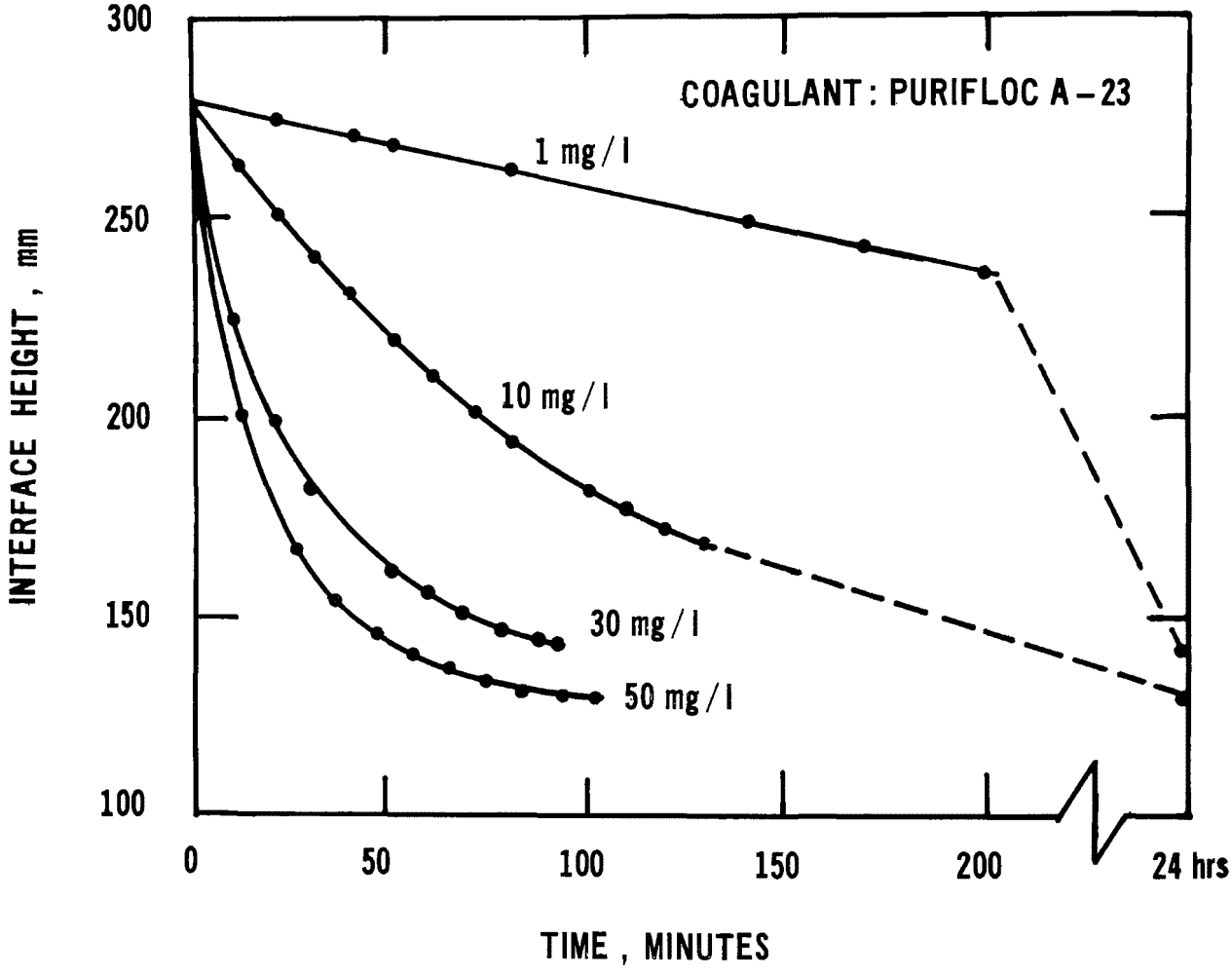


FIGURE 20. SETTLING OF DETARTRATED RAW STILLAGE

polyelectrolyte addition, and c) detartration with polyelectrolyte addition. The supernatant was obtained by running 150 ml samples of reconstituted raw stillage through the IEC centrifuge for two minutes at 2400 rpm. Supernatant liquor suspended solids concentration was of the order of 5800 mg/L.

## Coagulation

Variables considered in the coagulation of the stillage supernatant included cationic polyelectrolyte and bentonite concentrations. Nalco 610 polyelectrolyte was used at concentrations of 5, 15, 35, 60, and 100 mg/L. Bentonite concentrations were 10 and 20 mg/L.

In each case the coagulants were added to 200 ml of stillage supernatant, mixed at 100 rpm for ten minutes and flocculated for two minutes at 20 rpm. The mixture was then poured into a 250 ml graduated cylinder. Settling properties and supernatant COD were measured as function of time. Settling times were less than five minutes in all cases, and an interface did not form (i.e. settling was not hindered). Results are shown in Figure 21.

## Dissolved Air Flotation

Variables considered in the dissolved air flotation studies included coagulant, air concentrations, recycle fluid, pressure and recycle ratios. Cell pressures and recycle ratios used were $2.07 \times 10^6$, $2.76 \times 10^6$, $3.45 \times 10^6$, $4.14 \times 10^6$ dynes/cm$^2$ (30, 40, 50, and 60 psig) and 3.33 and 5.0, respectively. Water and recycled supernatant were used as the recycle fluid. Amoco nonionic and Amoco cationic polyelectrolytes were used in 1, 3, and 7 mg/L concentrations. Runs

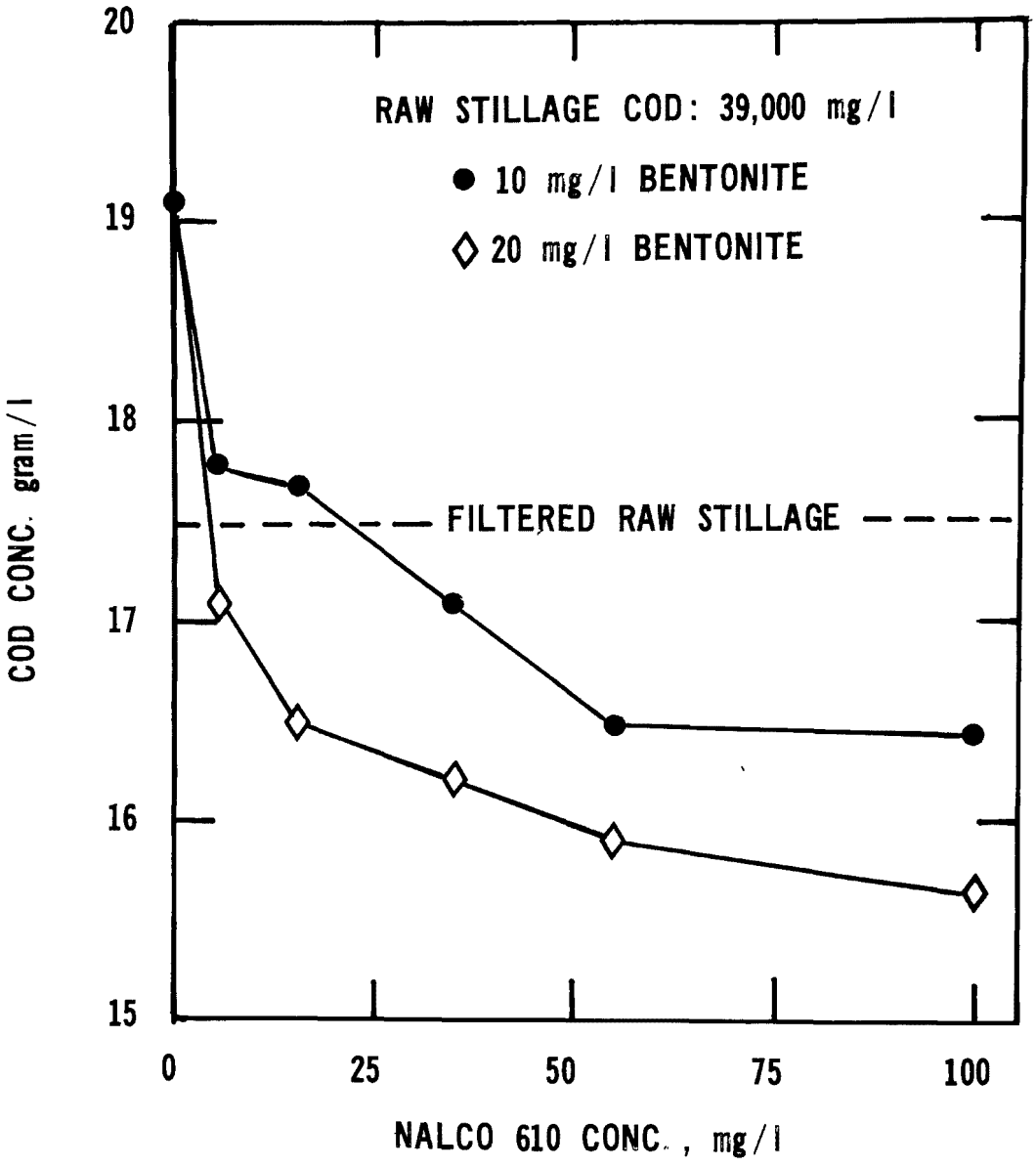


FIGURE 21. SUPERNATANT COD vs. COAGULANT CONC.

on the stillage supernatant without aids were also run. Satisfactory solids separation did not occur in any of the experiments.

Detartration With Polyelectrolyte Addition

Tartrate in stillage is in a soluble form as was noted in Section IV. Thus, detartration of the stillage supernatant liquor rather than raw stillage has the advantage of less contamination of the precipitate. Quantities of calcium chloride and calcium hydroxide necessary would be expected to be similar, as is shown in Figure 22.

Addition of an anionic polyelectrolyte (Purifloc A-23) improved both suspended solids and COD removal as shown in Figure 23.

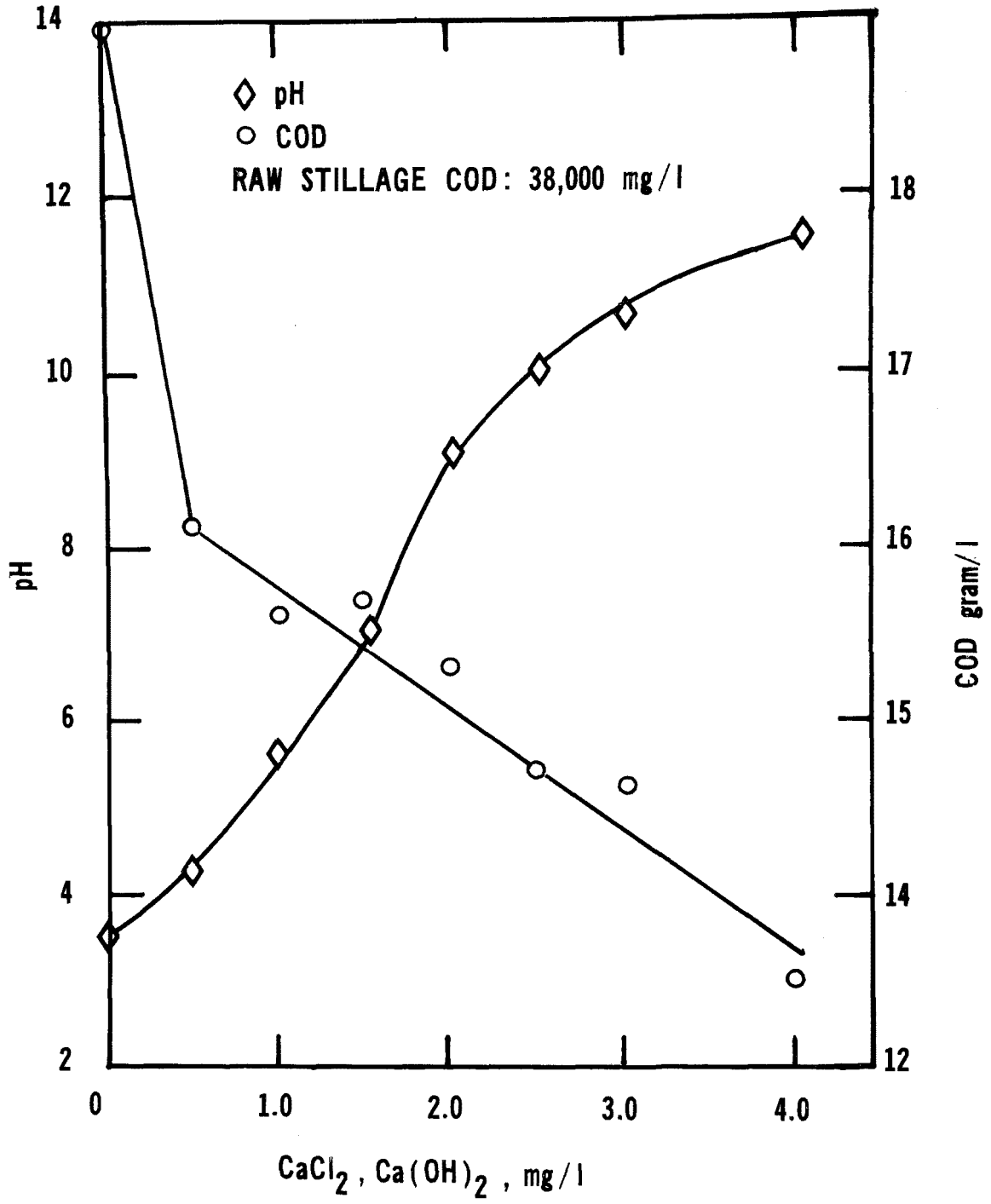


FIGURE 22. pH AND COD VERSUS $CaCl_2$ and $Ca(OH)_2$ CONC.

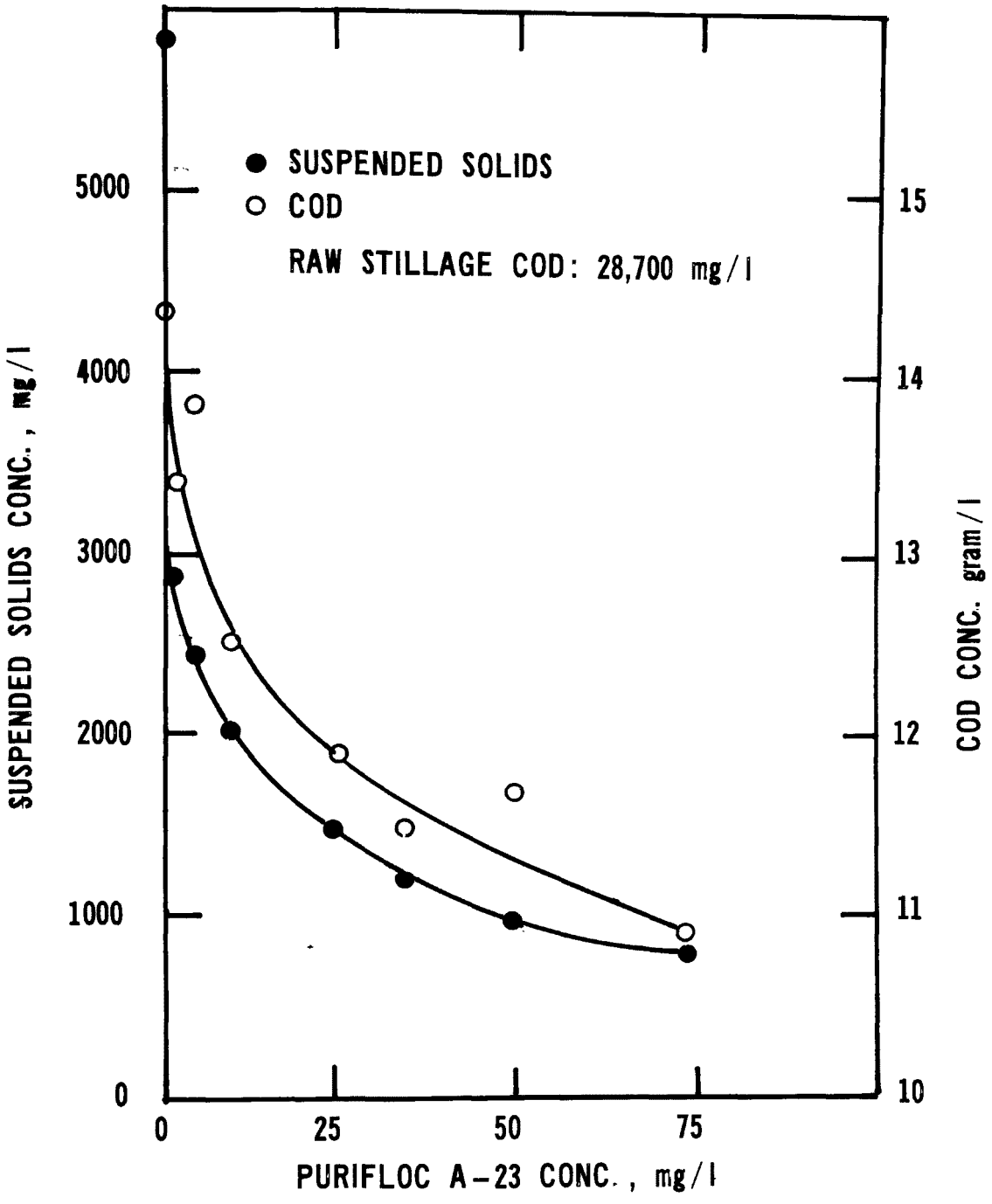


FIGURE 23. SUSPENDED SOLIDS AND COD CONC. OF DETARTRATED SETTLED SUPERNATANT LIQUOR

# SECTION VI

# DISCUSSION

Biological treatment of California winery stillage is possible, either anaerobically or aerobically. In either case additional treatment will be necessary. Aerobic treatment will require that a significant fraction (of the order of 90%) of the suspended solids be removed prior to aeration. Suspended solids removal may not be necessary prior to anaerobic treatment, but the high effluent organic concentrations (COD concentrations of the order of 5000 mg/L) associated with anaerobic treatment will force the use of an additional, probably aerobic, treatment process.

Because of the seasonal nature of the waste aerobic processes seem more desirable than anaerobic processes. Aerobic process "start up" time is short and the results of the pilot and laboratory studies lead to the conclusion that a successful treatment system can be designed and constructed. Aerobic treatment of settled stillage can produce effluents with filtrate COD and $BOD_5$ values of the order of 700 mg/L and 75 mg/L, respectively. Settling rates of the activated sludge are very low, even after further aeration in secondary units, and the quantity of nonsettleable material is relatively high. Thus, while aerobic treatment does an excellent job of converting organic material, the residual effluents COD and suspended solids concentrations are still unsatisfactory. Finally, the quantities of nitrogen which must be added are extremely high. Ammonium chloride should not be used because of the high quantity of chlorides which would be added to the effluent. Less ammonium nitrate is needed on a pound per gallon basis, but ammonium nitrate is about twice as

expensive as ammonium chloride. Phosphorous is available in excess and will not need to be added.

Successful aerobic biological treatment will be dependent upon pretreatment for solids removal. Successful solids removal will involve concentration from about two percent to ten percent. This is necessary to minimize the solids volume to reasonable proportions. Several methods of treating the solids are possible including anaerobic digestion. Selection of a method of suspended solids disposal was beyond the scope of this project.

Based upon the laboratory and pilot plant studies results, a system of aerated lagoons is recommended as the best method of biological treatment of winery stillage.

Following solids removal, aerated lagoons designed for a three to three and a half day residence time should be used to remove most of the organic material. In order to reduce the nitrogen requirement nitrogen should not be added to these aerated lagoons, but instead should be added to the effluent as it flows into a second set of aerated lagoons. Based on the 1972 pilot studies the organic concentration will be about 20 to 25 percent of that in the settled raw stillage, allowing a correspondingly lower addition of nitrogen. Effluent from the second set of aerated lagoons should be allowed to settle in holding ponds with a minimum of one day residence time. Settled solids can collect on the bottom and degrade during the nonoperating months as in conventional stabilization ponds. A schematic of the proposed process is shown in Figure 24. Effluent from the final ponds should be suitable for irrigation or possibly for discharge into municiple sewers.

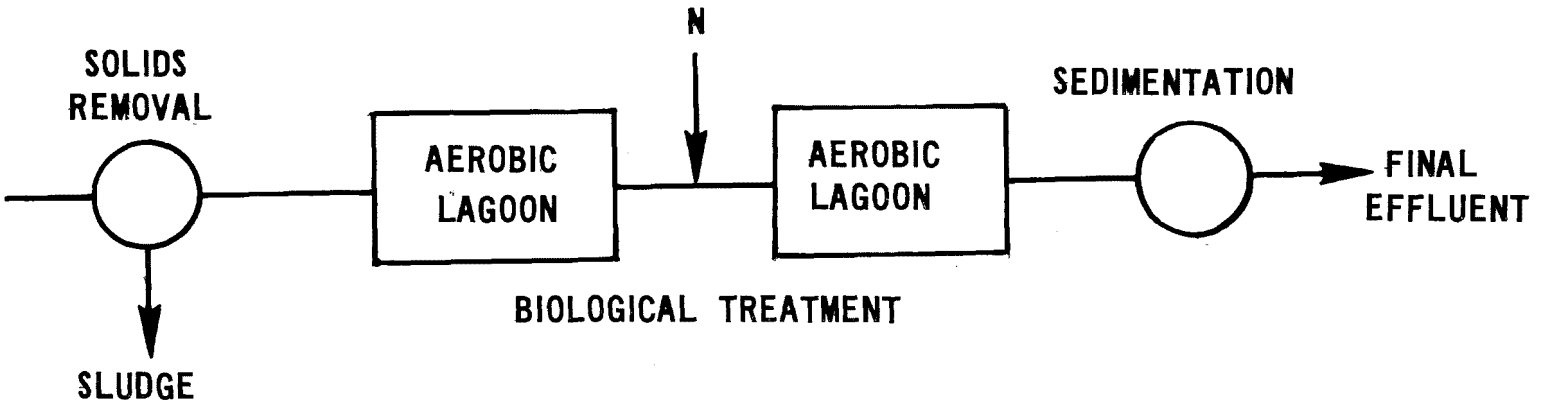


FIGURE 24. PROPOSED TREATMENT SYSTEM

Coagulation, flocculation and sedimentation of stillage appear to be an unsatisfactory solids removal method because of the sludge volume produced. In all of the experiments sludge volume exceeded one third of the original liquid volume and therefore use of this process would simply create two disposal problems from the initial one. The fact that raw stillage had better settling properties than the coagulated stillage is probably due to the increase in particle interaction with flocculation. A point should be made that supernatant suspended solids concentrations are much lower in the coagulated stillage than in the untreated stillage (Figure 12). Chemical oxygen demand values are also less but this is probably primarily a result of the improved solids removal.

Centrifugation proved to be the best method of removing solids from the stillage. Sludge volume was satisfactory (approximately 10%) and removals can be achieved at feasible speeds and run times. Operating a continuous flow centrifuge under conditions to match the batch data at five minutes and 1800 rpm would produce a product of approximately 1000 mg/L suspended solids and 18,000 mg/L COD. Reardon[4] found that stillage of this strength was treatable in aerated lagoons. Cake moisture under these conditions is approximately 8%, and the corresponding sludge volume would be 9% of the original volume. It should be noted that cake moisture content is an average value and thus tends to decrease as lighter, less compactable solids settle out at longer run times.

The effect of run time on cake moisture content is even more noticeable when coagulants are used. Improved suspended solids removal occurs, increasing the total amount of solids in the cake and the cake volume. Cake moisture content decreases more sharply than

with the untreated stillage in this case, as would be expected.

Detartration is of interest if potassium bitartrate is to be recovered. Results of the detartration experiments on raw stillage lead to the conclusion that the process is not suitable. Three problems are associated with this procedure, final separation of the tartrate, the very low settling rates which develop and the large sludge volume that would result, even when high polyelectrolyte concentrations are used.

Studies on solids removal from supernatant liquor were undertaken to determine what quality of effluent can be achieved with respect to solids removal. Pretreatment by centrifugation was chosen because this appeared to be the most effective treatment process for the raw stillage. Coagulation with a polyelectrolyte and bentonite worked extremely well on this less concentrated material and the batch settling time was less than five minutes in all cases.

Detartration of the supernatant liquor is much more straight forward than in the case of the raw stillage, particularly with respect to solids removal. As in the case of coagulation of the supernatant liquor settling rates were high (batch times of the order of two minutes or less and no interface was formed).

Foaming problems will be less than those experienced in the pilot studies in larger ponds. Good solids removal will also remove much of the light pulpy material which gave the pilot study foam the properties most difficult to deal with.

Biological treatment of winery stillage is an alternative to intermittant

irrigation. Prior to any changes in the present method of disposal, intermittant irrigation should be studied further. The possible use of tile drainage to collect the wastewater should be considered, together with study of the quantity and quality of water actually moving through the soil. If nuisance problems exist some effort should be made to develop a systematic method of application and of nuisance control.

Additional study of anaerobic treatment is not recommended. The major advantages of anaerobic treatment are the utilization of the high temperature effluent from the still, the small amount of nitrogen addition necessary (not established in these studies but probably of the order of 10% of that stoichiometrically needed for aerobic processes) and the methane gas production. Major disadvantages include the long start up times, and the requirement of additional treatment of the effluent. The start up problem is extremely important and the fact that the aerated lagoon systems proposed appear satisfactory leads the conclusion that aerobic processes are preferable.

## ESTIMATED CAPITAL COST

The proposed treatment process would include solids removal by centrifugation, biological treatment without nitrogen addition in an aerated pond with a three day residence time followed by biological treatment in an aerated pond with nitrogen addition and a three day residence time and finally a clarification pond to remove and store solids, as shown in Figure 24.

The centrifugation step will be a major cost item. A continuous cake discharge, solid bowl, scroll type centrifuge ranges in cost from

approximately $25,000 for a machine capable of handling 55 liters/min. (14.5 gpm) to approximately $100,000 for a unit capable of handling 475 liters/min. (125 gpm). A very large winery (eg. Gallo-Fresno) therefore would need about four large centrifuges with a capital outlay of approximately $400,000.

Basin cost for a $2.3 \times 10^6$ liter/day ($6 \times 10^8$ gpd) plant assuming four identical aeration basins, each six feet deep, and having a total volume of 13,655 cubic meters (17,800 cubic yards) and two settling basins, also six feet deep and having a total volume of 2300 cubic meters (3000 cubic yards) is approximately $57,000 or $25/1000 liters/day ($24/3785 gpd). Costs are based on $2.10 per cubic meter for excavation and $2.63 per cubic meter for basin walls.

Floating aerators would be the best choice for the system described. Considering the expected cell yield and the average settled stillage COD values the expected oxygen demand is approximately 1136 kilograms per hour (2500 lb/hr) for a $2.3 \times 10^6$ liter/day flow rate. Aerators up to 112 kilowatts (150 hp) in size are available and considering the high power to volume which will be achieved oxygen transfer rates of 1.22 gram/watt-hr (2 lb/hr-hr) can be expected. Approximately nine aerators at an installed cost of about $30,000 each would be required.

Thus the total cost of centrifuges, aerators and basins for a 2,300,000 liter per day treatment plant would be approximately $730,000.

Piping, pumps and laboratory space and equipment must be added to this total. Operation and maintenance are not included either. Figure 25 presents estimated costs for the three major capital items as a function of flow rate. Consideration of the total cost of treatment,

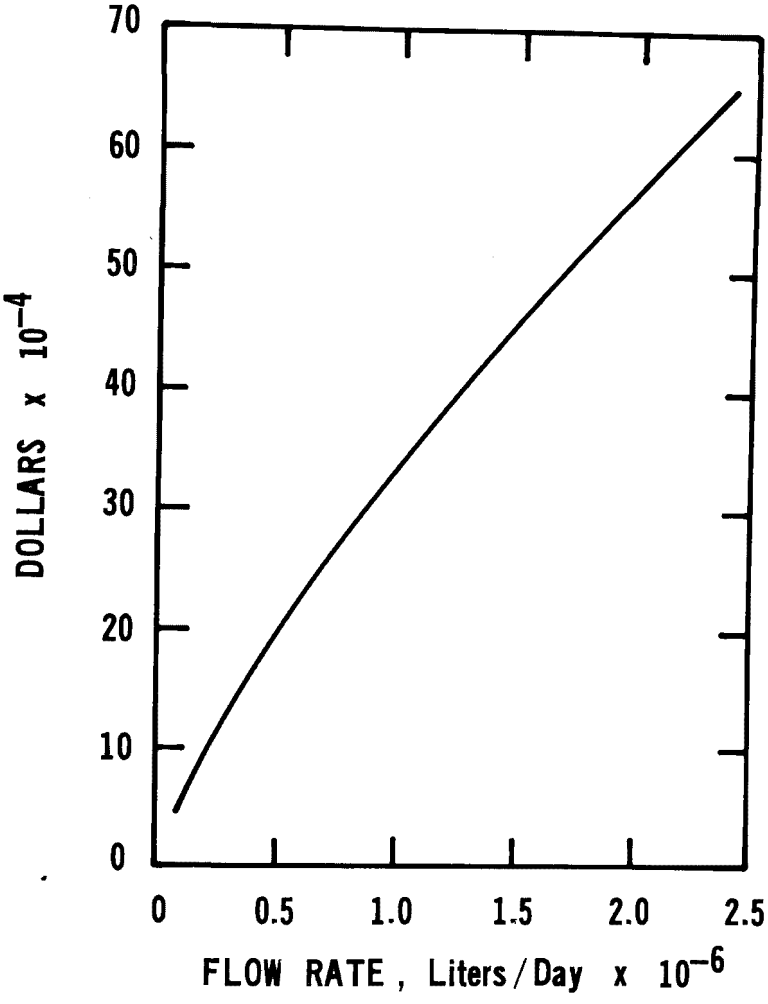


FIGURE 25. ESTIMATED TOTAL COST OF CENTRIFUGES AERATORS AND BASINS.

the seasonal nature of winery operations and fact that treated effluents will probably be used for irrgation wherever possible leads to the conclusion that biological treatment is a less satisfactory process than direct land disposal by intermittant irrigation. The primary concern with respect to intermittant irrigation is the possible contamination of the soil and groundwater with salts. Biological treatment of stillage is feasible and will be suitable in cases where suitable land is not available for irrigation. Properly designed processes should produce a product suitable for use as irrigation water or for discharge into a municipal sewer.

## SECTION VII

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# SECTION IX

# GLOSSARY

## ABBREVIATIONS

| | |
|---|---|
| BOD | Biochemical Oxygen Demand |
| cm | centimeter |
| COD | Chemical Oxygen Demand |
| m | meter |
| mg/L | milligram per liter |
| mm | millimeter |
| min | minute |
| MLSS | Mixed liquor suspended solids |
| RCF | Relative centrifugal force |
| RPM | Revolutions per minute |

## SYMBOLS

| | |
|---|---|
| g | gravitational constant $ML^{-1}t^{-2}$ |
| $k_d$ | Maintenance energy coefficient, $t^{-2}$ |
| N | Rotational Speed, $t^{-1}$ |
| pH | Negative logrithm of the hydrogen ion concentration |
| r | Centrifuge radius, L |
| $r_{ox}$ | Specific organic removal rate, $t^{-1}$ |
| SS | Suspended solids concentration, $ML^{-3}$ |
| Y | Cell yield |
| $\mu$ | Specific growth rate, $t^{-1}$ |

# SECTION XI

# APPENDICES


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TABLE A-1. 1971 PILOT PLANT INFLUENT COD DATA

| DATE | COD, mg/L | | | |
|---|---|---|---|---|
| | Raw Waste | | Inlet Holding Tank | |
| | Unfiltered | Filtered | Unfiltered | Filtered |
| 10/28 | | 14,721 | | |
| 10/29 | | | 34,401 | 15,193 |
| 10/30 | 33,351 | 16,263 | 31,197 | 15,438 |
| 10/31 | 38,334 | 18,657 | | 14,012 |
| 11/1 | 30,806 | 16,971 | 30,904 | |
| 11/5 | 28,534 | 16,473 | 35,150 | 15,795 |
| 11/6 | 17,500 | 15,557 | 34,458 | 13,755 |
| 11/7 | 16,683 | 15,409 | 16,499 | 13,995 |
| 11/10 | 32,980 | 15,594 | 30,759 | 16,194 |
| 11/11 | 28,941 | 14,844 | | 17,066 |
| 11/15 | | | 12,598 | 13,159 |

TABLE A-2. 1971 ACTIVATED SLUDGE PILOT PLANT

| DATE | COD, mg/L | | | |
|---|---|---|---|---|
| | Mixed Liquor | | Effluent | |
| | Unfiltered | Filtered | Unfiltered | Filtered |
| 10/25 | | | | |
| 10/29 | 14,170 | 4,669 | 12,359 | 4,985 |
| 10/30 | 15,575 | 3,889 | 14,864 | 4,478 |
| 10/31 | 16,776 | 5,825 | 13,875 | 5,825 |
| 11/1 | 18,703 | 4,698 | 17,568 | |
| 11/5 | 16,932 | 7,461 | 14,439 | 7,073 |
| 11/6 | 17,500 | 6,166 | 17,985 | 6,166 |
| 11/7 | 14,574 | 6,523 | 14,670 | 6,220 |
| 11/10 | 18,136 | 6,097 | 16,621 | 5,598 |
| 11/11 | 20,660 | 6,867 | 15,712 | 6,665 |
| 11/15 | 13,395 | 4,452 | 15,794 | 4,452 |

TABLE A-3 1971 UNFILTERED ANAEROBIC PACKED BED COD DATA

| DATE | COD, mg/L | | | | | |
|---|---|---|---|---|---|---|
| | TANK 1 | | TANK 2 | | TANK 3 | |
| | Bottom | Top | Bottom | Top | Bottom | Top |
| 10/25 | | | | 29,700 | 36,748 | 15,360 |
| 10/29 | | 33,377 | 48,274 | | | 34,164 |
| 10/30 | | 51,198 | | 35,339 | 58,229 | 35,458 |
| 10/31 | 56,043 | 35,669 | 74,768 | | 65,272 | 34,472 |
| 11/1 | 67,407 | 41,773 | 83,502 | 44,417 | 62,171 | 40,304 |
| 11/5 | 48,723 | 34,191 | 45,688 | | | 33,711 |
| 11/6 | 60,947 | 32,617 | 64,594 | 38,721 | | |
| 11/7 | 82,598 | 24,737 | 77,091 | 34,613 | 68,737 | 28,288 |
| 11/10 | 77,712 | 36,413 | 73,312 | 41,462 | 60,714 | 33,081 |
| 11/11 | 65,113 | 42,674 | 64,114 | 44,390 | 71,112 | 49,036 |
| 11/15 | 90,666 | 23,091 | 63,548 | 47,980 | 63,548 | 30,788 |

TABLE A-4. 1971 ANAEROBIC PACKED BED FILTRATE COD DATA

| DATE | COD, mg/L | | | | | |
|---|---|---|---|---|---|---|
| | TANK 1 | | TANK 2 | | TANK 3 | |
| | Bottom | Top | Bottom | Top | Bottom | Top |
| 10/25 | 17,476 | 12,122 | 17,161 | 14,642 | 15,114 | 15,272 |
| 10/29 | 18,505 | 14,876 | 17,028 | 18,674 | 18,701 | 16,301 |
| 10/30 | 18,620 | 16,028 | 17,206 | 16,145 | 18,620 | 16,028 |
| 10/31 | 17,065 | 16,531 | 19,392 | 17,318 | 21,331 | 15,901 |
| 11/1 | 16,012 | 15,245 | 16,683 | 14,957 | | 15,533 |
| 11/5 | 16,376 | 16,473 | 16,570 | | | 16,473 |
| 11/6 | 16,695 | 14,419 | 15,936 | 14,703 | 16,695 | |
| 11/7 | | 14,824 | 16,823 | 15,813 | 17,631 | 16,722 |
| 11/10 | 18,093 | 17,193 | 17,193 | 16,693 | 17,893 | 16,993 |
| 11/11 | 17,066 | 16,056 | 16,460 | 15,854 | 17,873 | 17,167 |
| 11/15 | 18,601 | 15,435 | 16,721 | 17,018 | 19,491 | 15,534 |

TABLE A-5. 1971 TEMPERATURE AND pH DATA FOR AEROBIC AND ANAEROBIC SYSTEMS

| Date | Inlet tank | | Anaerobic No. 1 | | | | Anaerobic No. 2 | | | | Anaerobic No. 3 | | | | Aerobic | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | pH | P°C | pH | | T°C | | pH | | T°C | | pH | | T°C | | pH | T°C |
| | | | Bot | Top | Bot | Top | Bot | Top | Bot | Top | Bot | Top | Bot | Top | | |
| 10/27 | 7.2 | 27 | 6.2 | | 24 | | 6.2 | | 19 | | 6.2 | | 19 | | 7.7 | 21 |
| 10/28 | 6.4 | 29 | 6.2 | | 24 | | 6.3 | | 22 | | 6.2 | | 21 | | 6.9 | 12 |
| 10/29 | 6.9 | 33 | 6.3 | | 24 | | 6.3 | | 23 | | 6.3 | | 21 | | 7.2 | 16 |
| 10/30 | 6.8 | 38 | 6.3 | | 24 | | 6.3 | | 20 | | 6.3 | | 22 | | 7.6 | 21 |
| 10/31 | 6.3 | 42 | 6.2 | 6.0 | 26 | 26 | 6.2 | 6.1 | 23 | 23 | 6.2 | 6.0 | 23 | 24 | 7.5 | 21 |
| 11/1 | 6.3 | 44 | 6.3 | | 28 | | 6.5 | | 25 | | 6.2 | | 23 | | 7.4 | 24 |
| 11/4 | 6.3 | 36 | 6.2 | | 24 | | 6.5 | | 25 | | 6.3 | | 25 | | 7.4 | 21 |
| 11/5 | 6.3 | 42 | 6.4 | | 29 | | 6.5 | | 24 | | 6.3 | | 27 | | 7.5 | 24 |
| 11/6 | 6.5 | 37 | 6.3 | | 28 | | 6.3 | | 24 | | 6.3 | | 27 | | 7.4 | 22 |
| 11/7 | 6.7 | 38 | 5.8 | | 28 | | 6.0 | | 25 | | 5.8 | | 27 | | 7.0 | 19 |
| 11/9 | 5.9 | 30 | 6.4 | | 25 | | 6.1 | | 22 | | 6.0 | | 22 | | 7.3 | 22 |
| 11/10 | 5.9 | 41 | 6.0 | 6.1 | 26 | 29 | 6.2 | 6.1 | 24 | 29 | 6.0 | 6.1 | 25 | 29 | 7.3 | 27 |
| 11/11 | 5.9 | 39 | 5.9 | | 21 | | 5.9 | | 24 | | 6.0 | | 19 | | 7.1 | 22 |
| 11/14 | 5.9 | 24 | 6.3 | | 14 | | 6.2 | | 13 | | 6.2 | | 13 | | 7.4 | 13 |
| 11/15 | 6.4 | 31 | 6.2 | | 19 | | 6.3 | | 17 | | 6.2 | | 22 | | 7.3 | 17 |

TABLE B-1. 1972 AEROBIC PILOT PLANT DATA

| Date | Mixed Liquor pH | Influent | | | Effluent | | |
|---|---|---|---|---|---|---|---|
| | | Susp. Solids mg/L | COD mg/L | Filtrate COD mg/L | Susp. Solids mg/L | COD mg/L | Filtrate COD mg/L |
| 9/22 | | | 77,000 | 19,700 | | | >4,100 |
| 9/16 | 7.1 | | | | | | |
| 9/18 | 7.1 | | | | | | |
| 9/19 | | | 74,600 | 17,100 | | 22,400 | 6,700 |
| 9/20 | 7.2 | | | | | | |
| 9/21 | 6.4 | | | | | | |
| 9/24 | | 17,800 | 22,600 | 14,800 | 9,200 | 12,600 | 7,320 |
| 9/25 | 7.2 | | | | | | |
| 9/27 | 7.2 | | | | 3,840 | 21,200 | 3,000 |
| 9/28 | | | | | 5,700 | 21,600 | 6,800 |
| 9/29 | 7.3 | | | | 7,400 | 9,250 | 3,200 |
| 9/30 | | | | | 3,800 | 13,500 | 1,500 |
| 10/1 | 7.4 | | | | | | |
| 10/2 | 7.6 | | | | 3,560 | 30,800 | 3,600 |
| 10/5 | | 6,500 | 7,300 | 7,250 | | | |
| 10/8 | 7.7 | | | | | | |
| 10/10 | 7.5 | | | | | | |

TABLE B-2. SEPTEMBER 24, 1972 ANAEROBIC PACKED BED NUMBER 1 DATA

| Sample Port | COD mg/L | Filtrate COD mg/L | Susp. Solids mg/L | pH |
|---|---|---|---|---|
| 1 | 133,000 | 12,800 | 18,500 | 5.5 |
| 4 | 7,500 | 700 | 5,500 | |
| 6 | 9,300 | 7,500 | 7,300 | 5.9 |

TABLE B-3. SEPTEMBER 24, 1972 ANAEROBIC PACKED BED NUMBER 2 DATA

| Sample Port | COD mg/L | Filtrate COD mg/L | Susp. Solids mg/L | pH |
|---|---|---|---|---|
| 1 | 54,800 | 13,200 | 21,400 | 5.9 |
| 4 | 8,900 | | 7,100 | |
| 6 | 27,300 | 3,900 | 4,100 | |

TABLE B-4. SEPTEMBER 12, 1972 ANAEROBIC PACKED BED NUMBER 3 DATA

| Sample Port | COD mg/L | Filtrate COD mg/L | pH |
|---|---|---|---|
| 1 | 66,500 | 28,700 | 6.0 |
| 4 | 40,000 | 28,500 | |
| 6 | 12,400 | 6,500 | 6.0 |

TABLE B-5. SEPTEMBER 24, 1972 ANAEROBIC PACKED BED NUMBER 3 DATA

| Sample Port | COD mg/L | Filtrate COD mg/L | Susp. Solids mg/L | pH |
|---|---|---|---|---|
| 4 | 25,000 | 7,000 | 10,200 | 5.8 |
| 6 | 11,100 | 3,900 | 5,500 | 5.9 |

TABLE B-6. 1972 RAW STILLAGE SETTLING DATA*

| September 17 | | September 19 | | September 20 | |
|---|---|---|---|---|---|
| Time min. | Interface ht., cm | Time min. | Interface ht., cm | Time min. | Interface ht., cm |
| 0 | 280 | 0 | 360 | 0 | 280 |
| 7 | 274 | 13 | 360 | 98 | 266 |
| 16 | 232 | 22 | 349 | 124 | 258 |
| 18 | 224 | 30 | 342 | 138 | 253 |
| 19 | 210 | 40 | 331 | 144 | 252 |
| 22 | 199 | 70 | 297 | 196 | 235 |
| 23 | 193 | 84 | 284 | 261 | 212 |
| 25 | 182 | 95 | 234 | 274 | 207 |
| 27 | 175 | 105 | 218 | 415 | 171 |
| 29 | 168 | 115 | 214 | 467 | 162 |
| 31 | 162 | 404 | 178 | 514 | 154 |
| 35 | 153 | 1440 | 173 | 559 | 151 |
| 38 | 144 | | | 596 | 148 |
| 49 | 123 | | | | |
| 58 | 109 | | | | |
| 77 | 90 | | | | |
| 87 | 83 | | | | |
| 107 | 73 | | | | |
| 123 | 67 | | | | |
| 174 | 56 | | | | |
| 1440 | 39 | | | | |

* Samples run on September, 21 and October 12 did not settle.

TABLE B-7. 1972 ACTIVATED SLUDGE
MIXED LIQUOR SETTLING DATA

| September 20 | | September 21 | | September 23 | |
|---|---|---|---|---|---|
| Time min. | Interface cm | Time min. | Interface cm | Time min. | Interface cm |
| 0 | 280 | 0 | 280 | 0 | 280 |
| 149 | 95 | 5 | 274 | 10 | 277 |
| 201 | 87 | 22 | 272 | 27 | 273 |
| 248 | 53 | 35 | 263 | 45 | 266 |
| 293 | 50 | 45 | 255 | 52 | 262 |
| 330 | 48 | 51 | 252 | 75 | 251 |
| 1440 | 39 | 103 | 221 | 90 | 244 |
| | | 226 | 168 | 123 | 227 |
| | | 255 | 157 | 150 | 207 |
| | | 288 | 146 | 200 | 179 |
| | | 353 | 95 | 227 | 168 |
| | | 1440 | 50 | 268 | 151 |
| | | | | 1440 | 56 |

TABLE B-7. (cont.) 1972 ACTIVATED SLUDGE
MIXED LIQUOR SETTLING DATA

| September 24 | | September 26 | | September 28 | |
|---|---|---|---|---|---|
| Time min. | Interface cm | Time min. | Interface cm | Time min. | Interface cm |
| 0 | 280 | 0 | 280 | 0 | 280 |
| 15 | 275 | 30 | 277 | 12 | 90 |
| 30 | 272 | 45 | 274 | 65 | 70 |
| 75 | 255 | 70 | 266 | 120 | 84 |
| 105 | 244 | 90 | 253 | 1440 | 50 |
| 148 | 227 | 110 | 244 | | |
| 295 | 202 | 131 | 232 | | |
| 335 | 182 | 166 | 202 | | |
| 370 | 165 | 211 | 174 | | |
| 390 | 146 | 326 | 154 | | |
| 711 | 87 | 571 | 112 | | |
| 1440 | 59 | | | | |

TABLE B-7. (cont.) 1972 ACTIVATED SLUDGE
MIXED LIQUOR SETTLING DATA

| September 30 | | October 2 | | October 3 | |
|---|---|---|---|---|---|
| Time min. | Interface cm | Time min. | Interface cm | Time min. | Interface cm |
| 0 | 280 | 0 | 280 | 0 | 280 |
| 5 | 62 | 30 | 272 | 50 | 274 |
| 10 | 45 | 60 | 255 | 275 | 244 |
| 115 | 39 | 75 | 252 | 350 | 235 |
| 275 | 34 | 155 | 218 | 560 | 202 |
| | | 180 | 207 | 1440 | 84 |
| | | 250 | 188 | | |
| | | 320 | 176 | | |
| | | 450 | 129 | | |
| | | 1440 | 84 | | |

TABLE B-7. (cont.) 1972 ACTIVATED SLUDGE
MIXED LIQUOR SETTLING DATA

| October 10 | | October 11 | | October 12 | |
|---|---|---|---|---|---|
| Time min. | Interface cm | Time min. | Interface cm | Time min. | Interface cm |
| 0 | 280 | 0 | 280 | 0 | 280 |
| 25 | 272 | 20 | 266 | 20 | 274 |
| 75 | 252 | 40 | 249 | 80 | 249 |
| 110 | 238 | 85 | 221 | 140 | 227 |
| 175 | 212 | 145 | 193 | 320 | 179 |
| 200 | 202 | 205 | 174 | 580 | 140 |
| 410 | 140 | 240 | 165 | 1440 | 90 |
| 590 | 118 | 460 | 134 | | |
| 1440 | 84 | 900 | 109 | | |
| | | 1440 | 84 | | |

TABLE C-1. LABORATORY REACTOR 1 AT $\theta_c$ = 4.67 DAYS

| Date | Feed COD mg/L | Filtered Feed COD mg/L | Mixed Liquor Filtrate COD mg/L | Flow Rate mL/day | D.O. mg/L | pH | Suspended Solids mg/L |
|---|---|---|---|---|---|---|---|
| 6/13 | | | | 755 | | | |
| 6/15 | | | | | 6.5 | | |
| 6/16 | | | | 792 | | | |
| 6/17 | | | | | | | 7,820 |
| 6/18 | 18,318 | 16,963 | 794 | | | | |
| 6/20 | 18,047 | 14,472 | 573 | 665 | | 6.2 | 7,490 |
| 6/22 | | | 696 | 778 | | | 7,225 |
| 6/23 | 18,675 | 16,531 | 620 | | | | 6,960 |
| 6/26 | | | 713 | | | | 6,775 |
| 6/27 | 18,328 | 16,363 | 601 | 778 | 6.7 | 6.3 | |

TABLE C-2. LABOTATORY REACTOR 2 AT $\theta_c$ = 4.67 DAYS

| Date | Mixed Liquor Filtrate COD mg/L | D.O. mg/L | pH | Suspended Solids mg/L |
|---|---|---|---|---|
| 6/15 | 890 | 7.5 | 6.2 | |
| 6/17 | | | | 6,080 |
| 6/18 | 794 | | | |
| 6/20 | 716 | | 6.1 | 5,748 |
| 6/22 | 838 | | | 6,340 |
| 6/23 | 761 | | | 6,095 |
| 6/26 | 792 | | | 6,160 |
| 6/27 | 435 | 7.8 | 6.0 | |

TABLE C-3. LABORATORY REACTOR 1 AT $\theta_c$ = 3.58 DAYS

| Date | Feed COD mg/L | Filtered Feed COD mg/L | Mixed Liquor Filtrate COD mg/L | Flow Rate mL/day | D.O. mg/L | pH | Suspended Solids mg/L |
|---|---|---|---|---|---|---|---|
| 7/1 | 17,621 | 15,636 | 2,892 | 955 | | | |
| 7/2 | | | | | 6.0 | 6.4 | |
| 7/3 | 19,365 | 16,598 | 544 | | | | |
| 7/4 | 19,003 | 16,916 | 515 | 920 | 5.5 | 6.3 | 6,740 |
| 7/5 | 19,739 | 17,384 | 503 | 1,030 | | | |
| 7/6 | 19,396 | 17,186 | 566 | | | 6.1 | 7,000 |
| 7/7 | 19,739 | 16,977 | 558 | 1,010 | 4.0 | 5.8 | 7,468 |

TABLE C-4. LABORATORY REACTOR 2 AT $\theta_c$ = 3.58 DAYS

| Date | Mixed Liquor Filtrate COD mg/L | D.O. mg/L | pH | Suspended Solids mg/L |
|---|---|---|---|---|
| 7/1 | 608 | | | |
| 7/2 | | 7.5 | 6.1 | |
| 7/3 | 632 | | | |
| 7/4 | 612 | 7.7 | 6.0 | 5,743 |
| 7/5 | 492 | | | |
| 7/6 | 544 | | 5.9 | 5,775 |
| 7/7 | 471 | 8.0 | 5.6 | 6,240 |

TABLE C-5. LABORATORY REACTOR 1 AT $\theta_c$ = 2.75 DAYS

| Date | Feed COD mg/L | Filtered Feed COD mg/L | Mixed Liquor Filtrate COD mg/L | Flow Rate mL/day | D.O. mg/L | pH | Suspended Solids mg/L |
|---|---|---|---|---|---|---|---|
| 7/9 | | | | 1,210 | | | |
| 7/10 | 20,335 | 17,656 | 648 | 1,190 | 1.3 | 6.1 | 8,503 |
| 7/11 | 19,641 | 17,388 | 643 | 1,320 | 5.0 | 6.1 | |
| 7/12 | 19,641 | 17,143 | 615 | | 2.0 | 6.1 | 8,918 |
| 7/13 | 20,448 | 18,162 | 651 | 1,230 | | | 8,930 |
| 7/14 | 19,965 | 17,806 | 638 | 1,420 | | | 8,785 |
| 7/16 | 19,904 | 17,960 | 665 | 1,250 | | | |
| 7/18 | | | | 1,280 | | 6.0 | 8,535 |
| 7/19 | | | | | 6.5 | | |

TABLE C-6. LABORATORY REACTOR 2 AT $\theta_c$ = 2.75 DAYS

| Date | Mixed Liquor Filtrate COD mg/L | D.O. mg/L | pH | Suspended Solids mg/L |
|---|---|---|---|---|
| 7/10 | 702 | 8.0 | 5.9 | 6,745 |
| 7/11 | 626 | 7.5 | 5.9 | |
| 7/12 | 588 | 7.4 | 5.9 | 6,733 |
| 7/13 | 570 | | | 6,878 |
| 7/14 | 534 | | | 6,985 |
| 7/16 | 606 | | | |
| 7/18 | | | 5.9 | 7,315 |
| 7/19 | | 7.5 | | |

TABLE C-7. LABORATORY REACTOR 1 AT $\theta_c$ = 2.42 DAYS

| Date | Feed COD mg/L | Filtered Feed COD mg/L | Mixed Liquor Filtrate COD mg/L | Flow Rate mL/day | D.O. mg/L | pH | Suspended Solids mg/L |
|---|---|---|---|---|---|---|---|
| 8/7 | 22,238 | 19,078 | 1,293 | | | 6.2 | 10,002 |
| 8/8 | 22,274 | 19,661 | 724 | 1,480 | | | 9,082 |
| 8/9 | | | | 1,440 | | | |
| 8/10 | 20,354 | 18,289 | 647 | | Meter Broken | | 9,292 |
| 8/12 | 20,013 | 18,075 | 696 | 1,440 | | 6.6 | 8,655 |
| 8/14 | 19,930 | 17,825 | 645 | 1,460 | | 6.2 | 8,930 |
| 8/16 | 20,207 | 16,959 | 574 | 1,410 | | 6.2 | |
| 8/17 | | | | | | | 9,193 |

TABLE C-8. LABORATORY REACTOR 2 AT $\theta_c$ = 2.42 DAYS

| Date | Mixed Liquor Filtrate COD mg/L | D.O. mg/L | pH | Suspended Solids mg/L |
|---|---|---|---|---|
| 8/7 | 862 | | 6.1 | 6,508 |
| 8/8 | 360 | | | 6,907 |
| 8/9 | | Meter Broken | | |
| 8/10 | 670 | | | 6,758 |
| 8/12 | 720 | | 6.1 | 6,813 |
| 8/14 | 750 | | 6.1 | 7,550 |
| 8/16 | 615 | | 6.1 | |
| 8/17 | | | | 6,723 |

TABLE C-9. LABORATORY REACTOR 1 AT $\theta_c$ = 1.87 DAYS

| Date | Feed COD mg/L | Filtered Feed COD mg/L | Mixed Liquor Filtrate COD mg/L | Flow mL/day | D.O. mg/L | pH | Suspended Solids mg/L |
|---|---|---|---|---|---|---|---|
| 8/20 | | | | 1,730 | | 6.0 | |
| 8/21 | | | | 1,590 | | 6.1 | |
| 8/22 | 14,922 | 13,707 | 525 | | | | |
| 8/23 | 23,777 | 21,752 | 623 | 2,040 | | 6.5 | 7,980 |
| 8/24 | 20,236 | 19,710 | 628 | | | | 9,478 |
| 8/25 | | | | 2,020 | 3.5 | 6.4 | |
| 8/26 | | | | 1,870 | | | |
| 8/28 | 19,004 | 17,500 | 593 | 1,900 | 4.0 | 6.7 | 8,685 |
| 8/29 | 18,679 | 16,755 | 565 | 1,940 | 2.2 | | 8,685 |
| 8/30 | | | | 1,870 | 3.0 | 6.3 | 8,515 |

TABLE C-10. LABORATORY REACTOR 2 AT $\theta_c$ = 1.87 DAYS

| Date | Mixed Liquor Filtrate COD mg/L | D.O. mg/L | pH | Suspended Solids mg/L |
|---|---|---|---|---|
| 8/20 | | | 6.2 | |
| 8/21 | | | 6.2 | |
| 8/22 | 789 | | | |
| 8/23 | 788 | | 6.1 | 5,535 |
| 8/24 | 746 | | | 6,390 |
| 8/25 | | 5.7 | 6.3 | |
| 8/26 | | | | |
| 8/28 | 745 | 6.3 | 6.3 | 7,057 |
| 8/29 | 798 | 7.0 | | 7,148 |
| 8/30 | | 6.5 | 6.2 | 7,328 |

TABLE C-11. LABORATORY REACTOR 1 AT $\theta_c$ = 1.41 DAYS

| Date | Feed COD mg/L | Filtered Feed COD mg/L | Mixed Liquor Filtrate COD mg/L | Flow Rate mL/day | D.O. mg/L | pH | Suspended Solids mg/L |
|---|---|---|---|---|---|---|---|
| 8/31 | 19,382 | 16,952 | 623 | 2,470 | | 6.4 | |
| 9/2 | | | | 2,520 | 3.5 | 6.6 | |
| 9/3 | | | | | | | 8,690 |
| 9/4 | 19,314 | 16,875 | 589 | | | | |
| 9/5 | | 16,021 | 516 | | | | 6,945 |
| 9/6 | 20,027 | 18,282 | 629 | 2,470 | 3.5 | 6.5 | 7,960 |
| 9/7 | 21,454 | 19,248 | 649 | | 2.3 | 6.3 | 8,585 |
| 9/8 | 19,868 | 18,774 | 811 | | | | |
| 9/10 | | | | | | 6.6 | 9,400 |

TABLE C-12. LABORATORY REACTOR 2 AT $\theta_c$ = 1.41 DAYS

| Date | Mixed Liquor Filtrate COD mg/L | D.O. mg/L | pH | Suspended Solids mg/L |
|---|---|---|---|---|
| 8/31 | 706 | | 6.2 | |
| 9/2 | | 6.5 | 6.3 | |
| 9/3 | | | | 7,713 |
| 9/4 | 589 | | | |
| 9/5 | 575 | | | 6,580 |
| 9/6 | 640 | 6.5 | 6.5 | 6,455 |
| 9/7 | 684 | 7.0 | 6.3 | 6,542 |
| 9/8 | 788 | | | |
| 9/10 | | | 6.6 | 6,460 |

TABLE C-13. LABORATORY REACTOR 1 AT $\theta_c$ = 1.09 DAYS

| Date | Feed COD mg/L | Filtered Feed COD mg/L | Mixed Liquor Filtrate COD mg/L | Flow Rate mL/day | D.O. mg/L | pH | Suspended Solids mg/L |
|---|---|---|---|---|---|---|---|
| 9/10 | | | | | 1.3 | 5.5 | |
| 9/11 | 21,674 | 19,328 | 4,703 | | | | |
| 9/12 | 20,712 | 19,028 | 7,157 | 3,240 | 3.8 | 4.8 | 6,453 |
| 9/13 | | | | 3,190 | 1.0 | 4.4 | 4,678 |
| 9/14 | 19,958 | 17,618 | 9,168 | | 1.6 | | |

TABLE C-14. LABORATORY REACTOR 2 AT $\theta_c$ = 1.09 DAYS

| Date | Mixed Liquor Filtrate COD mg/L | D.O. mg/L | pH | Suspended Solids mg/L |
|---|---|---|---|---|
| 9/10 | | 2.5 | 7.2 | |
| 9/11 | 947 | | | |
| 9/12 | 990 | 2.5 | 7.5 | 7,850 |
| 9/13 | | 1.0 | 7.5 | 6,870 |
| 9/14 | 1,157 | 1.5 | | |

TABLE D-1. BATCH SETTLING TEST RESULTS FOR 1/3, 2/3 MIXTURE OF PURIFLOC C-41 AND AMOCO CATIONIC COAGULANT AIDS*

| Time | Interface Height, cm | | | | | |
|---|---|---|---|---|---|---|
| | Coagulant Concentration, mg/L | | | | | |
| min. | 0 | 7 | 15 | 24 | 36 | 60 |
| 0 | 280 | 280 | 280 | 280 | 280 | 280 |
| 30 | 269 | 276 | 276 | 276 | 276 | 274 |
| 60 | 255 | 271 | 271 | 270 | 271 | 266 |
| 135 | 196 | 260 | 267 | 251 | 241 | 238 |
| 255 | 140 | 244 | 246 | 210 | 176 | 176 |
| 345 | 123 | 230 | 232 | 182 | 146 | 146 |
| 490 | 109 | 207 | 211 | 150 | 120 | 121 |
| 1440 | 92 | 134 | 136 | 107 | 101 | 99 |

* Stillage COD = 42, 400 mg/L

TABLE D-2. EFFECT OF COAGULANT ADDITION ON SUPERNATANT LIQUOR*

| Characteristic | Coagulant Concentration mg/L | | | | | |
|---|---|---|---|---|---|---|
| | 0 | 7 | 15 | 24 | 36 | 60 |
| COD, mg/L | 19,540 | 16,160 | 16,260 | 16,130 | 15,860 | 15,620 |
| Susp. solids mg/L | 1,270 | 222 | | 175 | 149 | 88 |

* Stillage COD = 42, 400 mg/L

TABLE D-3. EFFECT OF CENTRIFUGATION ON CAKE MOISTURE CENTRATE SUSPENDED SOLIDS* AND CENTRATE COD**

| Run | 1200 RPM | | | 1800 RPM | | | 2400 RPM | | | 3000 RPM | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Cake Moist % | Susp. Solids mg/L | COD mg/L | Cake Moist % | Susp. Solids mg/L | COD mg/L | Cake Moist % | Susp. Solids mg/L | COD mg/L | Cake Moist % | Susp. Solids mg/L | COD mg/L |
| 1 | 90.6 | | 19,800 | 87.7 | 1,625 | 19,470 | 87.9 | 1,147 | 18,630 | 86.0 | 1,138 | 18,975 |
| 2 | 88.0 | 1,685 | 18,870 | 86.8 | 1,446 | 18,830 | 88.7 | 968 | 18,220 | 85.4 | 872 | 17,600 |
| 4 | 87.6 | 1,255 | 18,120 | 86.4 | 944 | 18,220 | 85.5 | 609 | 17,720 | 86.0 | 621 | 17,540 |
| 8 | 87.3 | 1,003 | 18,290 | 86.8 | 765 | 18,080 | 84.4 | 648 | 18,110 | 84.4 | 418 | 17,250 |
| 15 | 87.1 | 872 | 17,550 | 84.5 | 538 | 17,540 | 83.7 | 501 | 17,540 | 82.6 | 394 | 17,540 |

* Stillage Susp. Solids Conc. = 11,952

** Stillage COD conc. = 36,100



TABLE D-4. EFFECT OF POLYELETROLYTE ADDITION* ON CENTRATE CHARACTERISTICS AT 2400 RPM

| Run Time Min. | Cake Moist % | Susp. Solids mg/L | COD mg/L |
|---|---|---|---|
| 0 | - | 23,010 | 46,400 |
| 1 | 85.8 | 1,610 | 21,105 |
| 3 | 83.9 | 897 | |
| 6 | 84.5 | 644 | 21,120 |
| 10 | 83.8 | 506 | 20,955 |
| 15 | 81.8 | 598 | 20,715 |

* 8 mg/L Amoco Cationic

TABLE D-5. EFFECT OF DETARTRATION ON SUPERNATANT COD*

| $C_aCl_2$ conc. g/L | $C_a(OH)_2$ conc. g/L | pH | Filtered COD |
|---|---|---|---|
| 0 | 0 | 3.45 | |
| 0.50 | 0.50 | 4.95 | 13,060 |
| 0.75 | 0.75 | 5.50 | 12,830 |
| 1.00 | 1.00 | 6.25 | 12,520 |
| 1.25 | 1.25 | 6.90 | 12,400 |
| 1.50 | 1.50 | 7.70 | 12,160 |
| 1.75 | 1.75 | 8.25 | 12,780 |
| 2.00 | 2.00 | 8.65 | 12,320 |
| 2.50 | 2.50 | 9.05 | 12,520 |
| 3.00 | 3.00 | 9.90 | 12,620 |
| 4.00 | 4.00 | 10.7 | 12,720 |
| 4.50 | 4.50 | 11.4 | |

* Stillage COD = 34,230

TABLE D-6. SETTLING OF DETARTRATED STILLAGE USING PURIFLOC A-23*

| 1 mg/L | | 10 mg/L | | 14 mg/L | | 20 mg/L | |
|---|---|---|---|---|---|---|---|
| Time min | Interface height cm | Time min | Interface height cm | Time min | Interface height cm | Time min | Interface height cm |
| 0 | 280 | 0 | 263 | 0 | 268 | 0 | 263 |
| 20 | 279 | 10 | 256 | 10 | 260 | 10 | 259 |
| 40 | 272 | 20 | 246 | 20 | 247 | 20 | 254 |
| 50 | 261 | 30 | 236 | 30 | 241 | 30 | 248 |
| 80 | 262 | 40 | 225 | 40 | 230 | 40 | 241 |
| 140 | 249 | 50 | 207 | 50 | 220 | 50 | 235 |
| 170 | 241 | 60 | 196 | 60 | 211 | 60 | 229 |
| 200 | 234 | 70 | 187 | 70 | 202 | 70 | 221 |
| 230 | 227 | 80 | 182 | 80 | 192 | 80 | 213 |
| 250 | 221 | 100 | 174 | 100 | 184 | 90 | 207 |
| 1440 | 138 | 110 | 167 | 110 | 178 | 105 | 196 |
| | | 120 | 163 | 120 | 171 | 125 | 183 |
| | | 130 | 157 | 130 | 168 | 135 | 178 |
| | | 1440 | 123 | 1440 | 129 | 1440 | 129 |

* Untreated Detartrated Stillage did not form interface.

TABLE D-6. (continued) SETTLING OF DETARTRATED STILLAGE USING PURIFLOC A-23

| 26 mg/L | | 30 mg/L | | 40 mg/L | | 50 mg/L | |
|---|---|---|---|---|---|---|---|
| Time min | Interface height cm | Time min | Interface height cm | Time min | Interface height cm | Time min | Interface height cm |
| 0 | 263 | 0 | 274 | 0 | 269 | 0 | 272 |
| 10 | 258 | 10 | 241 | 10 | 232 | 11 | 221 |
| 20 | 249 | 20 | 204 | 20 | 185 | 26 | 165 |
| 30 | 241 | 30 | 185 | 30 | 159 | 36 | 151 |
| 40 | 231 | 40 | - | 40 | 148 | 46 | 142 |
| 50 | 222 | 50 | 162 | 50 | 141 | 56 | 138 |
| 60 | 213 | 60 | 157 | 60 | 136 | 66 | 136 |
| 70 | 206 | 70 | 151 | 70 | 133 | 76 | 132 |
| 80 | 196 | 80 | 146 | 80 | 131 | 86 | 131 |
| 90 | 191 | 90 | 145 | 90 | 129 | 96 | 131 |
| 105 | 182 | 95 | 144 | | | 101 | 131 |
| 125 | 171 | | | | | | |
| 135 | 168 | | | | | | |
| 1440 | 129 | | | | | | |

TABLE D-7. COAGULATION OF CENTRATE* WITH NALCO 610 POLYELECTROLYTE AND BENTONITE.

| Nalco 610 | Supernatant Liquor COD, mg/L | |
|---|---|---|
| mg/L | 10 mg/L Bentonite | 20 mg/L Bentonite |
| 5 | 17,840 | 17,070 |
| 15 | 17,770 | 16,510 |
| 35 | 17,070 | 16,200 |
| 60 | 16,480 | 15,900 |
| 100 | 16,410 | 15,610 |

*Stillage COD = 39,100 mg/L
Centrate COD = 19,080 mg/L
Fitrate COD = 17,470 mg/L

TABLE D-8. DETARTRATION OF CENTRATE*

| $C_a(OH)_2$ g/L | $C_aCl_2$ g/L | COD mg/L | pH |
|---|---|---|---|
| 0.00 | 0.00 | 18,960 | 3.60 |
| 0.25 | 0.25 | 16,240 | 4.10 |
| 0.50 | 0.50 | 16,080 | 4.31 |
| 0.75 | 0.75 | 16,120 | 4.72 |
| 1.00 | 1.00 | 15,690 | 5.60 |
| 1.30 | 1.30 | 15,930 | 6.40 |
| 1.50 | 1.50 | 15,760 | 6.98 |
| 1.75 | 1.75 | 15,230 | 8.55 |
| 2.00 | 2.00 | 15,340 | 9.10 |
| 2.25 | 2.25 | 14,990 | 9.60 |
| 2.50 | 2.50 | 14,750 | 10.05 |
| 2.75 | 2.75 | 14,790 | 10.05 |
| 3.00 | 3.00 | 14,630 | 10.65 |
| 4.00 | 4.00 | 13,500 | 11.5 |

TABLE D-9. COAGULATION OF DETARTRATED CENTRATE WITH PURIFLOC A-23

| Purifloc A-23 mg/L | Residual Conc mg/L | |
|---|---|---|
| | Suspended Solids | COD |
| 0 | 5820 | 15,040 |
| 2.5 | 2872 | 13,260 |
| 5.0 | 2430 | 13,840 |
| 10 | 2020 | 12,520 |
| 25 | 1480 | 11,900 |
| 35 | 1210 | 11,450 |
| 50 | 1002 | 11,700 |
| 75 | 819 | 10,800 |

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**16. ABSTRACT**

Pilot and laboratory scale studies were run on aerobic and anaerobic biological treatment of winery stillage over a two year period. The pilot scale studies included work with aerobic lagoons and anaerobic packed towers. Laboratory systems studied were aerobic reactors without recycle and batch fed anaerobic processes. Because suspended solids removal proved to be a key factor in successful biological treatment, centrifugation, detartration, coagulation and flocculation, and combinations of these methods were included in the studies.

Centrifugation proved to be the best method of removing solids prior to biological treatment. Solids removal in combination with an aerobic treatment process can be expected to produce final filtrate chemical oxygen demands of about 700 mg/L and a final filtrate BOD of about 75 mg/L. Anaerobic processes studied did not operate well but produced effluents with chemical oxygen demands of the order of 4000 mg/L.

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