with a fixed procedure oil determinations and flavor could be correlated.

SUMMARY

Thirteen packs of frozen Temple orange concentrates were prepared during three seasons, all of which were products of good quality. However, a concentrate with the typical Temple flavor was obtained only when cold-pressed peel oil from the fruit was added to the concentrate. The intensity of Temple flavor did not correlate directly with the recoverable oil content of the concentrates, since oil determinations showed little relationship to the characteristic Temple flavor.

ACKNOWLEDGMENT

Acknowledgment is made to Mr. W. M. McSweeney, Mr. W. Storey, and Mr. R. C. Wooten who supplied the Temple oranges for this investigation.

Mr. J. W. Kesterson prepared the cold-pressed Temple orange oil used in this study and the authors wish to thank him for his assistance.

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THE RESULTS OF RESEARCH ON CITRUS PROCESSING WASTE DISPOSAL

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The value of citrus fruit and citrus processing to Florida is probably better known to this audience than to the writer. However, a few statistics may help to remind us of the magnitude of the industry.

During the crop year 1952-53, Florida produced 109,600,000 standard boxes of citrus fruit valued at $218,828,993. Of that amount 62,000,396 boxes of fruit were consumed by processing into juice, sections, concentrate, fruit salad, etc. In other words, processing plants used just slightly over 50 percent of all the citrus fruit picked that year. It was estimated that the gross value of the processed fruit at the canner's door was $86,857,519.00. Needless to say, all Florida has a stake in those figures.[1]

However, the processing of citrus fruit can produce a liability as well as an asset. I refer, of course, to the waste products which flow just as steadily into the streams and lakes of Florida as the golden fruit flows into the chutes of the processing plants. Much has been done since citrus canning first started in 1921 to reduce or eliminate wastes of various kinds and by-products of cow feed, citrus molasses, peel oil, alcohol, pectin, are ample evidence of the ingenuity and technical skill of the American industrialist and his staff of experts as they constantly strive to leave not even the "squeal" in the case of citrus fruit. There does remain, though, in spite of their efforts, a residual of dissolved sugars and a small amount of suspended solids in the huge volumes of process water which ultimately, up to now, has found its way into the surface and underground streams. This residual probably seems to a person used to thinking in terms of per cent or brix to be too slight to deserve consideration. The quantities in many cases are so slight that they are expressed in parts per million by weight; however, these slight residuals may make one good-sized concentrating plant the equivalent in terms of organic content to a city of many thousands of persons.

The sanitary engineer or sanitary chemist expresses the polluting potential of a waste in terms of its ability to reduce dissolved oxygen in a stream. Loss of oxygen is not by any means the only manifestation of pollution but in the absence of disease bacteria or toxic chemicals it becomes the dominant effect since its depletion may not only mean death to fishes and lower forms of aquatic life, but it may also indicate that the stream is approaching an anaerobic state of organic decomposition which is frequently coupled with discoloration, odors, sludge banks and floating sludge islands. We, therefore, use this oxygen depleting power or so-called *Biochemical Oxygen Demand* as a comparative measure of the polluting potential of a given waste and since the consumption of oxygen is dependent on time and temperature as well as on quantity of pollutants, sanitary engineers have agreed to express the Biochemical Oxygen Demand in terms of the quantity of oxygen consumed in a five-day period which is held at 20° C in the presence of an excess of oxygen. This is commonly expressed as the five day, 20° C BOD.

Almost all organic substances that are above the state of carbon dioxide or elemental carbon have some BOD, because in almost every case they oxidize in the presence of oxygen to simpler, more stable forms. Domestic sewage in the absence of industrial waste has a very consistent BOD, and it is estimated that the contribution of each person to the BOD of sewage each day amounts to about 0.17 lbs. of BOD. It may be diluted in a large quantity of water in which case the parts per million (ppm) will be low but the pounds of oxygen consumed at 20° C in five days will still be about 0.17 lbs. This gives us a basis for the so-called equivalent population basis of comparison. This is merely a handy device to compare two sources of waste in a unit easily understood by the layman. For example, one million gallons of a waste containing 100 parts per million of five day, 20° C BOD is equivalent approximately to sewage discharged from a city of 4,906 persons each day. That isn't always true so far as the stream is concerned, because different wastes of the same five day, 20° C BOD may have vastly different total oxygen depleting ability and may have different rates of depletion but it does serve as a rough method of comparison.

With that explanation then, let's look at some results obtained from some specific citrus waste analyses. Note, the number of plants on which these studies were made are very few and the sampling period is quite limited. These may or may not, therefore, represent typical wastes from plants of these types. Table I shows the results obtained from the study of several plants with different combinations of processes and with the results expressed in parts per million, pounds of five day BOD, population equivalent and pounds of BOD per unit of production where such a breakdown is possible.

The effects of discharging raw sewage from a city of 68,350 persons into one of our small fresh water lakes can easily be imagined. The comparison isn't perfect because the citrus waste does not contain the suspended solids or bacteria that the sewage contains, but the effect on our small streams and lakes is still devastating. In a number of instances one's nose is quite sufficient to determine whether the plant is operating or closed down.

Note that in general, as industrial wastes go, these are not strong wastes. These are much too diluted to make evaporation feasible and while McNary & Associates (3) have successfully produced methane by anaerobic decomposition of cannery wastes, the volume and strength of these wastes are such that there is little chance of profitable by-product recovery.

There have been a number of studies made on the treatment of wastes of this type. Von Lossecke & Associates (4) found in 1941 that a standard rate trickling filter could be used successfully and suggested a formula for deriving the size of the filter. If this formula is applied to the average results for the concentrate plant shown in Table I, the required filter for 75 per cent BOD removal would be 4,875 sq. ft. in area by 6 ft. deep or 29,200 cu. ft. of stone. At a very conservative $10.00 per cu. yd., that means that the filter rock alone would cost about $11,000.00. That's not impossible but it is to say the least discouraging to management.

Mention has been made of McNary's experiments with anaerobic fermentation which were quite successful but again very expensive for a dilute waste.

With all these facts in mind, the Florida State Board of Health in 1951, under the stimulation of a $5,000.00 grant from the U.S. Public Health Service under Public Law 845, entered into an agreement with the Florida Canners Association to conduct a research project to study several suggested methods of treating citrus waste sufficiently to permit its satis-

The Results of Research on Citrus Processing Waste Disposal

TABLE I

-4-

CITRUS PROCESSING WASTE DATA*

	Single Strength Juice Plant (2 plants)	Grapefruit Sectionizing Plant (2 plants)	Orange Juice Concentrate Avg. (4 Plants)	Cow Feed and Molasses Mill	Combination Single Strength and Sectionizing	Combination Single Strength Cow Feed and Mollasses
Production Per Day	18,850 Cases 28,744 Cases	4,420 Cases 3,718 Cases	20,042 gal. Concentrate	103.6✓ Tons	S.S. 11,247 Cases Sections-4,598 Cases	S.S. 7148 Cases Feed, 47 Tons Molasses, 5490 gal.
Waste Flow (gpd)	158,611 813,200	211,700 420,260	2,396,500	2,308,770	853,000	1,459,300
5-day-20°C. B.O.D. (p.p.m.)	182 182	873 945	82	498	460	395
Lb.B.O.D./1000 Cases	12.7 43.1	384 887	57.1 lb./1000 gal. Conc.	113 lb./ Ton✓		
Pop. Equiv. B.O.D.	1408 7280	9070 19,400	6,675	68,350	19,160	28,200
Suspended Solids (p.p.m.)	25 85	140 124	27	30	96	56 to 144
Settleable Solids ml./l	— 00	30 —	4.1	0.1	10	0.05
Total Solids (p.p.m.)	532 1,030	2,498 1,798	296	442	895	430 to 1605

* Data from the files of the Florida State Board of Health Stream Sanitation Laboratory. All cases are for No. 2 cans.

✓ Dry Feed only. Does not include liquid molasses sold as feed.

factory disposal into surface streams. The investigator on this project was Ben F. O'Neal, Chemical Engineer from the staff of the Bureau of Sanitary Engineering of the Florida State Board of Health and the work was done under the general supervision of the writer. The combined $5,000.00 grant from the U.S. Public Health Service and a $10,000.00 grant from the Florida Canners Association, together with the generous cooperation of a number of individuals and companies in loaning equipment and giving the benefit of their counsel permitted an 18 month's study during which the following methods of treatment were studied by means of pilot plant scale models.

The methods of treatment of the waste investigated were:

1. High rate trickling filter
2. Activated sludge
3. Spray irrigation
4. Chemical flocculation—air flotation

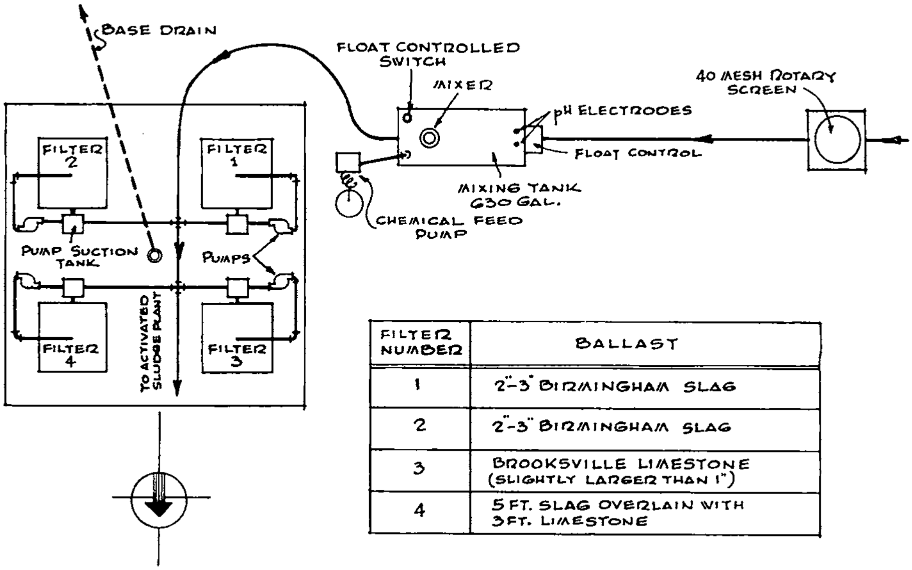
Discussion of Methods of Treatment Investigated (5)

1. *High Rate Trickling Filter.* A battery of four experimental trickling filters, each 3 sq. ft. and 8 ft. deep, were used. See Figure 1. A portion of the combined waste from the Plymouth plants was taken after passing through a 40 mesh rotary screen.

The waste flowed into a 400 gallon tank which was provided with a float controlled valve, a mixer and automatic pH controlling equipment. The waste then flowed into four small tanks, each of which served as a pump sump for one of the filters. It was found in an earlier attempt with this filter that it was absolutely necessary to have pH adjustment. Wash-up periods frequently sent waste having a pH of 11.0 to the filters killing off the filter organisms and delaying further work until the filter beds could recover. This required a period of two weeks in several instances.

The actual operation of the four filters in O'Neal's research project covered conditions of hydraulic loading varying from 20 million gallons per acre per day (Mgad) to 96 million gallons per day. Recirculation ratios varied from 4/1 to 15/1. These values represented units of recirculated waste per unit of raw waste added to the system. Raw waste feed to the filters varied from 4-12 mgad. BOD reaching the filters varied from 0.0096 lbs/day/cu. ft. filter volume. Filters No. 1 and

FIGURE I
TRICKLING FILTERS



FILTER NUMBER	BALLAST
1	2"-3" BIRMINGHAM SLAG
2	2"-3" BIRMINGHAM SLAG
3	BROOKSVILLE LIMESTONE (SLIGHTLY LARGER THAN 1")
4	5 FT. SLAG OVERLAIN WITH 3FT. LIMESTONE

No. 2, both of which were filled with 2″-3″ slag, were operated in series under various hydraulic loading conditions, recirculation ratios, and raw waste loadings. Filter No. 3 was filled with limestone and the raw waste loading was kept constant throughout the season with the hydraulic loading and recirculation ratio being varied for comparative purposes. Filter No. 4 was filled with a 5′ layer of limestone overlain by a 3′ layer of 2″-3″ slag. Filters No. 4 and No. 1 were operated under identical conditions for comparative purposes.

In an attempt to find an optimum depth for filters treating this waste, samples were collected from various filter depths and analyzed. These results indicate that an average of 84 per cent of the performance occurred in the upper 4′ of the filters. The data collected on the operation of the trickling filters definitely indicate better performance for filters No. 3 and No. 4. Filter No. 3 contained all limestone and No. 4 contained 5′ of limestone overlain with 3′ of slag. Filters No. 1 and No. 2 contained slag. The pHs of the effluent from the limestone filters as compared to those of the effluent from the slag filters were usually higher. At the beginning of the season, these differences were as much as one pH unit. As the filters built up biological coatings on the ballast, this difference decreased.

Data obtained by O'Neal reveals that the high rate trickling filters removed an average of as much as 0.182 pounds of BOD per cu. ft. of filter volume. This accounted for 76 per cent of the applied BOD which totaled 0.239 pounds per cu. ft. of filter volume. This filter was receiving a total hydraulic loading of 42 million gpad, a raw waste loading of 6 mgad and was being operated with a recirculation ratio of 6/1. The raw waste was being fortified with 50 ppm nitrogen and 20 ppm P_2O_5. There were many other conditions of operation which also gave very gratifying results for the filters. For a summary of the results for the four trickling filters, please see Graph VI. On the basis of 0.239 lbs. of BOD per cu. ft. the example previously used, in connection with the standard rate filter, would require a filter containing only 6,861 cu. ft. of rock or a bed 47 ft. in diameter by 4 ft. deep.

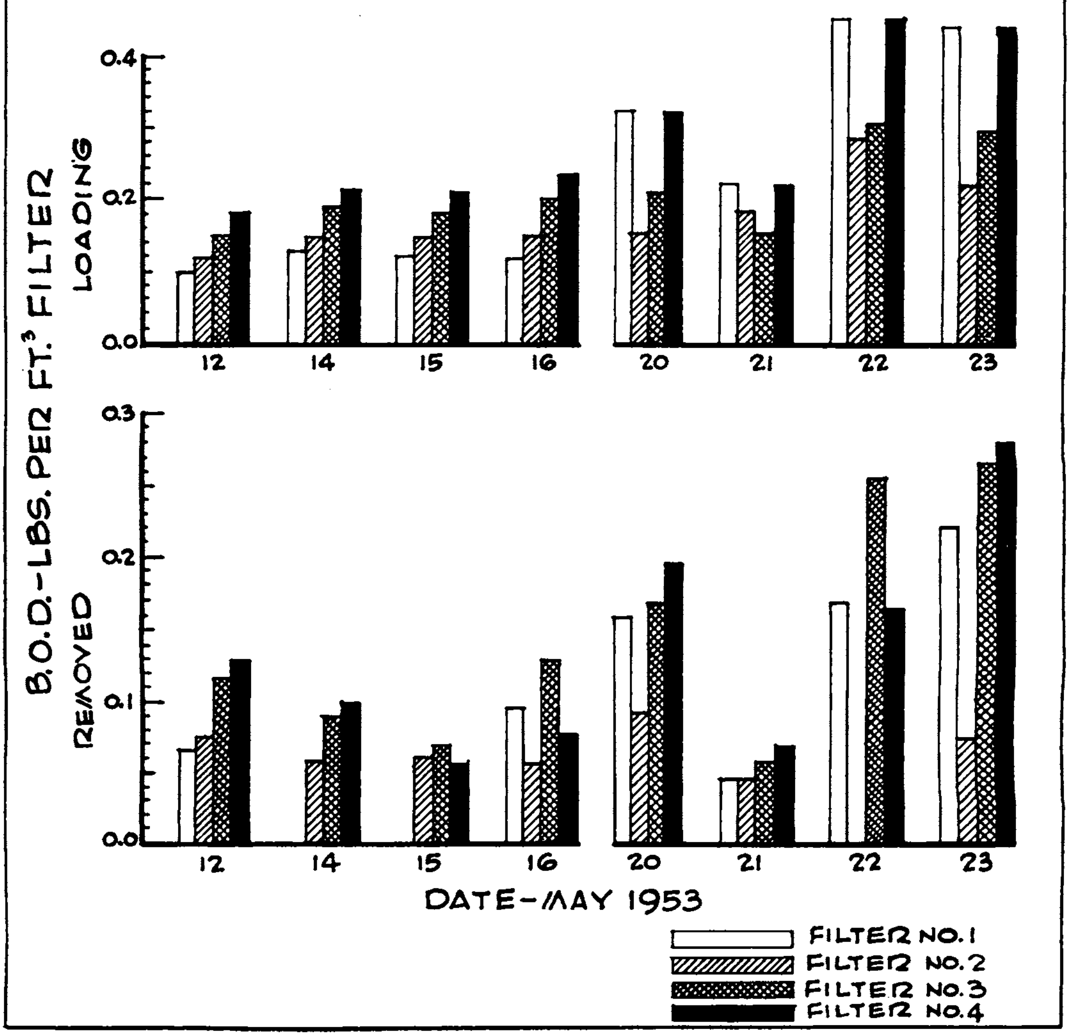
The addition of nitrogen and phosphorus (50 ppm nitrogen and 20 ppm P_2O_5) gave the

GRAPH-VI
FILTER LOADING & PERFORMANCE

FILTERS 1 & 2 OPERATED IN SERIES

FILTER	HYDRAULIC LOADING RAW	HYDRAULIC LOADING TOTAL	REC. RATIO	FILTER	HYDRAULIC LOADING RAW	HYDRAULIC LOADING TOTAL	REC. RATIO
1	4 (mgad)	20 (mgad)	4/1	1	9 (mgad)	45 (mgad)	4/1
2	5 "	25 "	4/1	2	9 "	45 "	4/1
3	6 "	30 "	4/1	3	6 "	42 "	6/1
4	7 "	35 "	4/1	4	9 "	45 "	4/1

NUTRIENT: ELEMENT	AMT. ADDED	SOURCE	NUTRIENT: ELEMENT	AMT. ADDED	SOURCE
N_2	10 (ppm)	NH_4NO_3	N_2	50 (ppm)	NH_4NO_3
P	20 (ppm) P_2O_5	SUPERPHOSPHATE	P	20 (ppm) P_2O_5	SUPERPHOS.



highest most uniform performance of the filters. The use of nitrogen and phosphorus at the rates noted above does not make their cost prohibitive. Based upon actual costs of these compounds, it amounts to $57.95 per million gallons of waste. Chemical costs would be $10,400 per season for a plant operating 180 days and discharging one million gallons of treatable waste per day. This is more of this type waste than any plant should create and hence be required to treat. Unlike domestic waste which contains enteric organisms, this waste will not require disinfection of the effluent nor digestion of the solids.

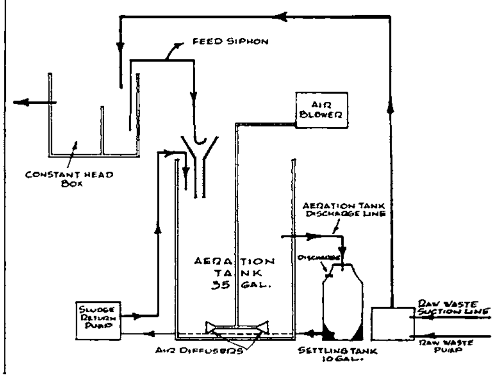
This will amount to savings not experienced by municipalities. O'Neal concluded that trickling filters operated either in series or at high recirculation ratios (6/1 and 8/1) offer practical means of treating citrus processing liquid waste. The addition of nitrogen and phosphorus not only increased the performance of the filters in terms of lbs. of BOD removed per unit volume of filter, but also increased the percentage reduction of BOD.

There are large volumes of water relatively free of BOD discharged from all concentrate plants. It is believed that under the most extreme conditions the waste which needs treating should not be over 1/10 the volume of the clear water which has a usual BOD of around 20 ppm. Based upon this, if a filter effluent containing 500 ppm BOD should be mixed with clear water containing 20 ppm BOD, the resulting BOD would be 64 ppm. In most cases, this should be satisfactory treatment and not cause subsequent trouble.

2. *Activated Sludge.* O'Neal experimented with a conventional type activated sludge plant to test the biological sludge type of treatment on citrus waste. (See Figure II). The shallow depth, 28", of the aeration tank resulted in inefficient use of the air supplied to the aeration tank. This was due to the short period of contact of the air with the liquid as the air bubbles rose through the liquid to the surface from which they escaped. Greater depths would increase this contact time and hence permit more of the oxygen to go into solution.

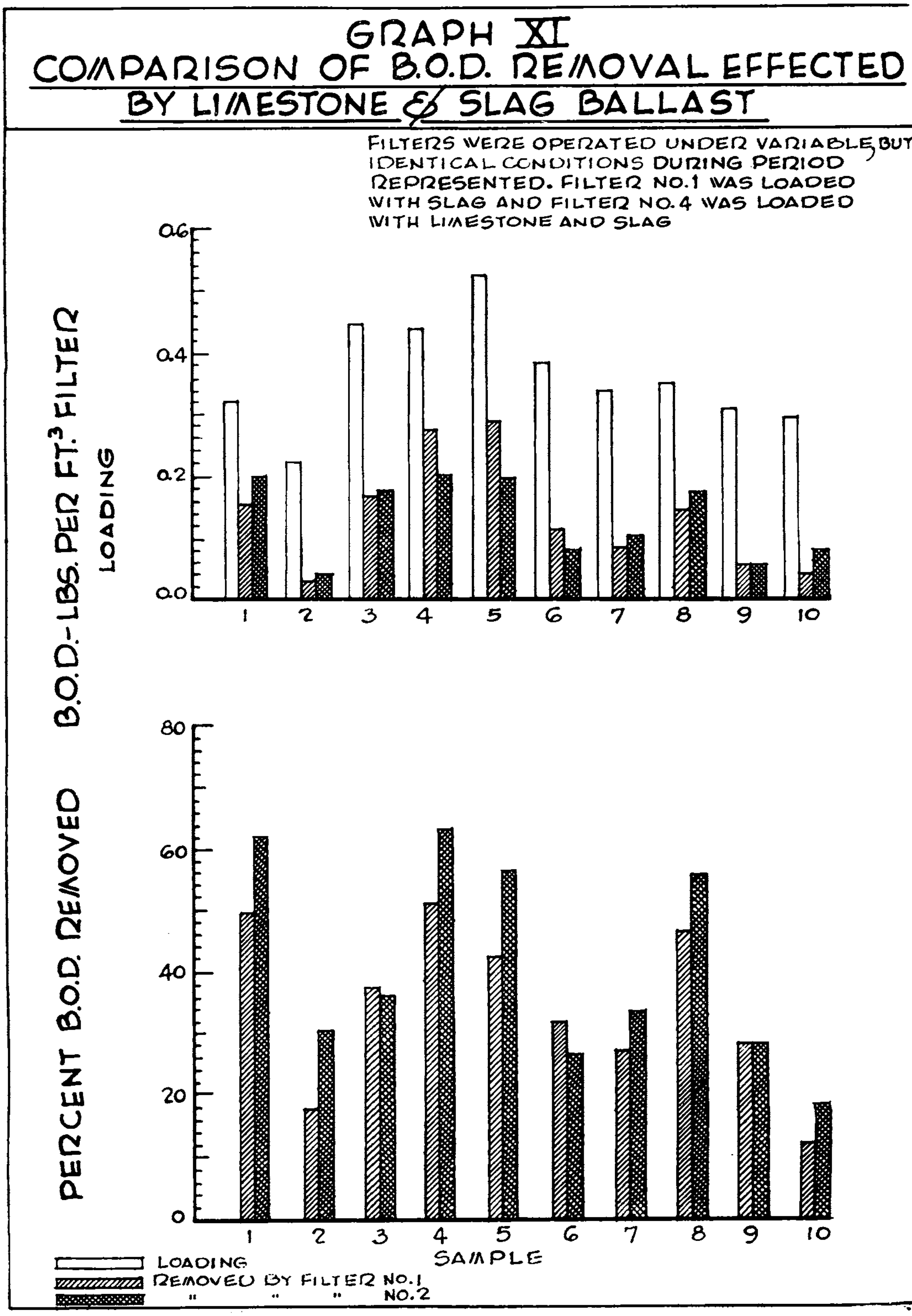
Phosphorus, in the form of superphosphate, and nitrogen, as sodium nitrate or ammonium nitrate, was added to the waste in various amounts before the waste was treated. This was directed at determining if there was necessity for such nutrients and, if so, the optimum amount and proportions that were necessary.

FIGURE II
ACTIVATED SLUDGE PLANT



A summary of O'Neal's results contained in Graph XI definitely reveals that the waste reaching this plant was effectively treated under practically all conditions of operation. The effluent discharged from the activated sludge presented three distinct appearances during this period of tests. During the time of very little or no perceptible D.O. content, it usually presented a brown cloudy appearance. At certain times this effluent was also slimy. During times when the D.O. was high and nutrient was added, the effluent was usually very clear with some small sludge suspensions visible. During these periods, the effluent never gave a slimy appearance. After nutrient was discontinued, but the D.O. was still high, the effluent was clear but brown in color. The odor of the effluent was musty upon certain occasions; however, it was usually odorless.

Final data indicated that of 10 conditions of operation tested, seven of them resulted in BOD reductions of 80 per cent or more. The lowest average percentage reduction was 69 per cent. Another observation is that, again, of the ten conditions tried, seven of them resulted in average final BODs less than 300 ppm. Based upon all results, an aeration time of 9 hours and a chemical feed of 50 ppm nitrogen and 20 ppm P_2O_5 is indicated for maximum logical results while 22 cu. ft. of air per gallon of waste were required to maintain a D.O. of 2-3 ppm during this experiment. It


GRAPH XI
COMPARISON OF B.O.D. REMOVAL EFFECTED
BY LIMESTONE & SLAG BALLAST
FILTERS WERE OPERATED UNDER VARIABLE, BUT IDENTICAL CONDITIONS DURING PERIOD REPRESENTED. FILTER NO. 1 WAS LOADED WITH SLAG AND FILTER NO. 4 WAS LOADED WITH LIMESTONE AND SLAG
B.O.D.-LBS. PER FT.³ FILTER
LOADING
PERCENT B.O.D. REMOVED
0.0
0.2
0.4
0.6
0
20
40
60
80
1 2 3 4 5 6 7 8 9 10
SAMPLE
LOADING
REMOVED BY FILTER NO. 1
" " " NO. 2

is felt that this amount could be reduced to 1/4 this amount in a tank of proper depth. The greatest difficulty which would result in the operation of an activated sludge plant treating citrus waste would be in removing the solids from the effluent. While it was not tried on this particular activated sludge experiment, it is believed that if air flotation would serve as a means of separating activated sludge solids from the liquid effluent that a desirable degree of treatment could be effected on citrus waste with shorter periods of aeration and less nutrient. This is based upon the fact that the magnitude of D.O., aeration time and nutrient feed had little effect on percentage of the BOD removed, but rather gave a more bulky sludge. Increased bulkiness of the sludge seemed to be the greatest effect of variable loading or of shock loading. With a process where this bulky sludge could be removed without depending upon its settleability, approximately as good results should be obtained with a bulky sludge as could be obtained with a more rapidly settling sludge.

Dougherty and McNary have also experimented with activated sludge with a laboratory model set-up and report (6) that removal of 90 per cent BOD or better was obtained following a considerable period of floc development without the use of nutrients. If that can be demonstrated to hold true on larger models, activated sludge would probably become the method of choice.

Another activated sludge investigation is also being made in Texas by personnel of the Texas Department of Health. Only progress reports have been seen by the writer but it appears that good treatment was secured in a drum-sized pilot plant utilizing pre-aeration and the addition of brewers yeast. A final report has not yet been prepared.

3. *Spray Irrigation.* O'Neal determined that in general the initial ability of the soil to absorb citrus waste was very great but decreased as the soil became saturated. Small growths such as grass, weeds and bushes were immediately destroyed by the waste, with larger growths such as pine trees and scrub oaks showing an indicated tolerance for the waste. It was observed that many of the scrub oaks and pine trees later died. In view of this, it seems reasonable to assume that these trees were destroyed by the waste previously applied. Land covered by vegetation and/or a heavy mulch exhibited greater ability to absorb the waste than did barren land. In all cases, flooding occurred around the irrigation pipe joints during intermittent operation and in depressions in the ground. Waste was applied to the soil in quantities varying from 5,770 gpad to 145,100 gpad. Reference is also made to reports by the National Canners Association on the use of this method of treatment for other types of fruit (7).

4. *Chemical Flocculation.—Air Flotation.* Experiments were conducted with this process during O'Neal's project with the thought that this method of solids removal might be beneficial to precede seepage lagoons and/or spray irrigation projects.

Chemical flocculation—air flotation treatment of citrus waste to remove solids did not prove successful to any surprising degree. An average of 61 per cent of the suspended solids were removed from the waste during an 8-day period of testing this method of treatment. Laboratory settling of the composited raw samples resulted in 64 per cent of the suspended solids being removed. Approximately 100 ppm lime was added during this experiment for flocculation.

Conclusion

It is believed that the studies described herein have amply demonstrated that citrus waste can be treated without an unusually large outlay of capital funds. The use of high rate trickling filters has been studied to the point that satisfactory results can be predicted with reasonably conservative organic loadings. Brooksville limerock appears to be completely satisfactory and its production in the proper size for trickling filters would materially reduce the cost of constructing these units.

The activated sludge process holds great promise but it is not possible to accurately predict the quantity of air required or the type of secondary clarifier needed. It has been suggested that activated sludge followed by air flotation might be a good combination that would take advantage of the apparent lightness of the activated sludge floc.

In both trickling filters and activated sludge O'Neal's study indicates that nitrogen and phosphorous additions will be required but the costs should not be prohibitive.

The spray irrigation process is especially attractive for plants located near high, dry land and where such land is cheap to buy or rent. A suitable cover crop is needed that can withstand the low pH found in citrus waste and when this is found, automatic pH control will probably be necesary to prevent killing the cover crop during clean up periods. This same control is necessary with all the other methods likewise. The use of a different type of pipe joint or the use of a smaller pump permitting longer periods of operation would eliminate or reduce the nuisance of ponding of waste at each pipe joint. The use of easily moved pipe is, however, necessary since any area will eventually become saturated.

Additional study of all these methods of waste treatment is needed and efforts are continuing to find the funds required for such a study.

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NOTES ON FACTORS ASSOCIATED WITH GELATION IN FROZEN CONCENTRATED ORANGE JUICE

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Frozen orange concentrate has been observed to gel when stored at temperatures higher than 0° F. (−17.8° C.). This phenomenon has been studied by a number of investigators (4, 9, 11, 12) and they generally agree that the basic factors involved are the pectin concentration, sugar content, pectinesterase activity, pH, and divalent ion content. In the preparation of the concentrate, the sugar content and pH are quite uniform. Baker and Goodwin (2) observed that pectin gels will form over a wide range of pH values. These include the pH range of 42° Brix commercial orange concentrate. Based on the results of Roberts and Gaddum (10), Wenzel, Moore, Rouse, and Atkins (12) came to the conclusion that citrus juices contain more than the optimum amount of calcium necessary for the formation of enzyme-demethylated pectin gels.

Recent investigations by Atkins, Rouse, Huggart, Moore, and Wenzel (1) have shown that heating the juice to inactivate the enzyme will stabilize the concentrate with respect to gelation.

Rouse (11) has shown that the loss of cloud and gelation in citrus juice are related to the low methoxyl pectin produced by the action of pectinesterase on pectin. Baker and Goodwin (2) have found that as the methoxyl content decreased from 7.1% to 4.5%, the amount of pectin required for gel formation in a given sugar solution also decreased. At 4.5% methoxyl content, the minimum amount of pectin was required. Other factors such as molecular weight of the pectin, the extent of esterification, and the distribution of the ester groups on the pectinic acid molecule have been listed by Kertesz (5) as important factors in the gel forming power of low methoxyl pectin.

The purpose of this study is to explore the relationship between pectinesterase activity and pectin content in commercial frozen orange concentrates. The observations made from the data obtained in the preliminary study are tentative, since the number of samples and analyses that are necessary in this type of study are insufficient to draw positive conclusions.

Experimental Methods

During the 1950-51 Valencia season samples of commercial concentrate were obtained from

[1]/Present address: Redwood Arsenal, Huntsville, Ala.

[2]/One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.