THE EFFECT OF BIOOXIDATION ON THE COAGULATION OF DISPERSED COLOR

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I. INTRODUCTION

Large volumes of liquid industrial wastes are produced from the processing, dyeing, and finishing of textile fibers, with the textile dyehouse contributing a large percentage of the total wastes. These dyeing operations are responsible for a highly pollutional and harmful waste from the standpoint of color, biological oxygen demand (B.O.D.), and chemical oxygen demand (C.O.D.). The wastes may also contribute suspended solids, high temperatures, high or low pH values, and toxic materials. In some cases such wastes have created conditions of stream pollution that must be abated.

There have been many research and pilot plant studies concerning the treatment of dyehouse wastes. These studies have included the treatment of wastes separately, the treatment of all the wastes from the textile processes together, and the treatment of the dye wastes and other wastes with domestic sewage. The types of treatment used have included chemical treatment or coagulation, lagoons, and aerated lagoons or bio-aeration processes. Other treatments are activated sludge plants, adsorption processes, trickling filters, and those treatment methods associated with the treating of wastes, such as sedimentation, filtration, and sludge digestion.

Of all the pollutional characteristics of dye wastes, color pollution is the most easily detected, and, therefore, one of the most significant forms of stream pollution. Until recently, little effort has been expended toward the removal of color from wastes or streams. Color in streams is esthetically objectionable, and in the eyes of the public, color is an indication of pollution. Only small quantities of color are removed by current treatment methods devised for eliminating other forms of pollution, but chemical coagulation and adsorption processes have been successful, to different degrees, in removing color from dye wastes. Although the coagulation and adsorption processes result in high color removals, there is an economic consideration involved. The cost of chemicals and adsorbents and the maintenance costs of these processes are great because of the large amount of color involved and the fact that many of the modern dyes contain dispersing and surface active agents. When the chemical coagulation or adsorption methods are used, most of the chemicals or adsorbents are needed to overcome the effects of the dispersing and surface active agents.

Over the past ten years, a new low-cost treatment process has been introduced as a treatment for dye wastes. This process, called the aerated lagoon or bio-aeration process, is based on the development of micro-organisms in a single one-stage prolonged biological process without the use of sludge digesters as compared with current conventional activated sludge processes. The aerated lagoon costs considerably less than most conventional treatment methods, and in many situations, the cost is less than treatment in a joint municipal plant (23). The efficiency of the aerated lagoon is comparable to the efficiencies of conventional treatment methods.

Since high degrees of color removal are achieved by the chemical coagulation process, it was desirable to determine if some form of biological treatment, prior to chemical coagulation, would reduce the chemical coagulant demand of dye wastes, while producing an efficient and economical treatment process.

The following research consists of an investigation employing an aerated lagoon or bio-aeration process as a pretreatment of a dye waste, followed by a chemical coagulation process. The wastes chosen for the investigation were black and brown dispersed dyes, which represent two of the most difficult dyes to treat economically. The purpose of this study was to determine if bio-aeration treatment would effectively reduce the chemical coagulant demand of black and brown dispersed dye wastes, while removing a large percentage of the pollutional characteristics of the wastes. The virtue of such a process would be the removal of a substantial fraction of the color and other pollutional matter from dark dispersed dyes at a minimum cost.

II. LITERATURE REVIEW

Textile waste treatment is one of the most complex problems in the field of industrial waste treatment because seldom are two textile wastes alike in character. In this situation, the different wastes may require quite different treatment processes. Even the wastes from each textile dyehouse differ, because of the widely different dye processes and operations.

Although some textile wastes were treated in this country and abroad, little was accomplished prior to 1925; and most of the information concerning textile waste treatment has been developed since 1930. Prior to 1930, several mills, in regions where stream pollution was most severe, built waste treatment plants at large cost. Many of these plants did not provide successful treatment commensurate with their cost because of the lack of scientific information on the subject.

Beginning in the early 1930's, investigative work has been conducted as an endeavor to develop general principles and procedures, basic in character, which could be used and further developed by the industry in solving waste disposal problems. Over the years, many treatment processes or combinations of treatment processes have been used for the treatment of dyehouse wastes. Among these treatment processes, activated sludge and trickling filter methods have proven efficient in many cases, especially when the dye waste is continuously combined with domestic sewage (1) (3) (4) (5) (10) (12) (19) (20) (21) (24). The adsorption process has been studied and found to be effective in removing color from dye wastes (17) (26). The process that has been most studied and developed to give good reductions in color and pollutional load has been the chemical coagulation method. This is the most widely used method of treating textile dye wastes. In certain situations, chemical coagulation has been effective in removing up to 99 per cent of the color and a large percentage of other pollutional characteristics. Coagulants commonly used are ferrous and ferric sulfate, ferric chloride, aluminum sulfate, lime, calcium chloride, sulfuric acid, and combinations of these. In general, it seems that ferrous sulfate or copperas and lime, together, form the most satisfactory treating material from the standpoint of color removal from dye wastes (2) (14) (16) (18) (20) (21) (22). Porges, Horton, and Baity (13) found that the chemicals that produce optimum coagulation varied according to the waste being treated. Therefore, one reagent or combination of reagents should not be relied upon when investigating chemical coagulation of a waste.

The most significant and comprehensive research investigation on chemical coagulation as a dye waste treatment method was performed by Porges et al (13). They used a systematic approach for studying the coagulation of dye wastes. Most studies on chemical coagulation, prior to Porges et al (13), used a trial and error method to arrive at the best treatment process.

Tests conducted by Porges et al (13) indicated that pH, coagulation procedures, order of addition of chemicals, and heat effects were important variables in the chemical coagulation of textile dye wastes. Some results of their research were as follows:

1. The pH range of coagulation for selected reagents that gave

optimum clarification or color removal for several types of dye wastes was determined. Also, any correlation between the hydrogen-ion concentration and the degree of coagulation for the various coagulants used was noted. The pH range for ferric chloride was below pH 5.6 and above pH 8.3. For ferric chloride and lime, the pH range was above pH 7.7. For ferrous sulfate or copperas, the pH range was above pH 7.0. Finally, for ferrous sulfate and lime, the pH range was above pH 5.6, with an optimum pH range between pH 8.2 and pH 11.5. The close correlation between the chemicals used and the pH range of coagulation in the study was most striking and appeared to be independent of most of the wastes treated. Other works reported similar correlations. Chrisco, White, and Baity (2) and Souther et al (20) (21) (22), experimenting with a variety of dye wastes, obtained final pH values approximating the pH ranges determined by Porges et al (13).

2. Adjustment of the pH of the supernatant liquor prior to discharge was an important factor in the chemical treatment of dye wastes. It was estimated that a pH varying from 6.0 to 8.0 was satisfactory for discharge into streams. This pH range was also advocated as a discharge criterion by Chrisco et al (2).

3. The order in which the chemicals were added in the coagulation test was very important. The order of addition of chemicals depended upon the type of waste being studied. It was shown

that for a greenish-blue indigo waste (initial pH 10.5), lime should be added prior to ferrous sulfate. For a blue vat dye waste (initial pH 11.7) and a blue developed color waste (initial pH 3.3), ferrous sulfate or ferric chloride should be added prior to the addition of lime. Color removal and sludge compaction were the criteria in all the cases studied.

4. Proper mixing and flocculation were determined, and they appeared to be important factors at times. Sludge compaction was influenced by mixing and flocculation. Laboratory findings showed that for a deep blue-black sulfur dye waste (initial pH 11.7), a short rapid mix, followed by little or no flocculation, formed the most compact sludge when ferric chloride was used as the coagulant. For a greenish-blue indigo waste (initial pH 10.5), a short rapid mix, followed by at least 30 minutes flocculation, formed the most compact sludge when lime and ferrous sulfate were used. Finally, for a blue direct dye waste (initial pH 10.8), a 30 second rapid mix, followed by five minutes flocculation, formed the most compact sludge. A blue developed color waste (initial pH 3.3) and a reddish-brown naphthol color dye waste (initial pH 11.6) required the same mixing and flocculation as the direct dye.

5. The temperature of the waste affected the amount of sludge produced from coagulation. The compaction of sludge and

ratio of dewatering increased with increased waste temperature. It was shown that for coagulation of a deep blue-black sulfur dye waste (initial pH 11.7) with ferric chloride, a sharp increase in the settling rate of the sludge occurred at 45°C, and the optimum settling rate occurred at 65°C. In the case of filtering rate, a sharp increase occurred at 45°C, but above 45°C, little change in the dewatering rate was observed. The effect of heat on sludge volume was also observed by Rudolfs and Baumgartner (15). They found that, when coagulating a sulfur dye waste with ferric chloride and hydrochloric acid, the volume occupied by the sludge at 80°C was less than half of the volume at 20°C. With temperatures higher than 80°C, the tendency was to produce a thinner sludge and the floc became smaller.

6. The sludge in the coagulation process was weighted with inert material in an effort to reduce the volume of sludge and induce compaction. For a deep blue-black sulfur dye waste (initial pH 11.7), it was found that clay increased settling rates, but color removal was poor. Coal dust and sand increased settling rates and color removal. With long detention times, such as 24 hours, all sludges tended to reach maximum compaction. Therefore, it was shown that weighting may not be advantageous where long detention periods are available.
7. The ratios of sludge volume to supernatant volume from the coagulation of two dye wastes were determined. For a deep

blue-black sulfur dye waste (initial pH 11.7), the ratio was 1.3:1 after coagulation with ferric chloride and settling for one hour. For a greenish-blue indigo waste (initial pH 10.5), the ratio was between 1:4.1 and 1:4.9 after coagulation with lime and ferrous sulfate and settling for one hour. After settling for 24 hours, the ratio had increased to 1:5.7 for the indigo waste. These results were somewhat different from the results found by Chrisco et al (2). They found that the sludge volume to supernatant volume ratio for an indigo waste varied from 1:3 to 1:20 after coagulation with lime and ferrous sulfate and settling for 24 hours. For a sulfur dye waste, it was shown that the ratio varied from 1:6 to 1:10 after coagulation with ferric chloride and settling for 24 hours. This difference may be contributed to differences in the dye compositions or dyeing procedures.

The results of many pilot plant and complete treatment plant studies have reinforced the results produced by Porges et al (13). One such pilot plant study on sulfur dye waste by Porges, Norton, and Gotaas (14), showed that laboratory studies by Porges et al (13) gave a close indication of plant operation. Laboratory results of color removal may have been slightly better than pilot plant studies because of the possibility of more accurate control in the laboratory. It can now be seen that the two investigations by Porges et al (13) (14) were turning points in the treatment of dye wastes, with the result being the development of a systematic approach to the treatment of dye wastes.

During the last decade, a new treatment process has been developing in the field of textile waste treatment, which may become a second turning point in the treatment of dye wastes. This new process is the low-cost aerated lagoon or bio-aeration process. This aeration process was originally developed by R. H. Souther to treat a mixture of sewage and mill waste at Cone Mills' Haw River plant in North Carolina (8).

> In the aeration system, a lagoon of sufficient size is built to hold the total waste expected for a two-or-three day period. At the inlet end, a mixing basin is fitted with a strainer to remove any undigestible solid matter. At the delivery end, there is a settling basin with or without provision for pumping back a portion of the sludge to the entering end of the lagoon.
>
> In operation, air pumps into the lagoon at the entering and through distribution pipes. The air circulates the waste and provides oxygen for the aerobic bacteria.
>
> Digestion is brought into balance by regulating air flow and, where necessary, by introducing sludge from the settling basin at the delivery end of the lagoon. This sludge is rich in bacteria, which feed on the nutrients in the mill waste-sewage mixture.
>
> The clear effluent from the top of the settling basin discharges into the stream at a B.O.D. reduction rate of 80 to 95%. Sludge does not accumulate in the lagoon because the organic matter is totally destroyed and the soluble salts from the textile waste are, of course, carried through in solution.
>
> Another method of introducing air is by mechanical entrainment produced by agitation in a mixer installed ahead of the lagoon [or in the lagoon] (8).

Souther et al (21) found that a 90 per cent textile mill waste -

10 per cent sewage mixture aerated for 24, 48, and 72 hours in air aerated lagoon, with varying amounts of aerated waste being recirculated, resulted in good removals of B.O.D. and fair removals of color. The 24, 48, and 72 hour removals of B.O.D. were 40.1, 56.6, and 92.3 per cent, respectively, and the removals of color were 11.7, 32.3, and 44.5 per cent, respectively. A seven to 10 per cent domestic wastewater seed was required for satisfactory biological growth. It was found that acid pH adjustment was not required for a highly alkaline textile waste, containing large amounts of hydroxide alkalinity. There was a reduction in pH during the aeration of the waste. Finally, it was shown that the cost of the aerated lagoon process varied from ¼ to ½ that a trickling filters and activated sludge processes. These results were also reported by Jones, Alspaugh, and Stokes (6) in a more detailed study.

Two pilot plant studies by Williams and Hutto (27) have shown that for two different dyeing and finishing wastes, aerated lagoons with detention periods of 48 hours or more, followed by settling, gave B.O.D. removals of 75 to 80 per cent. The wastes were markedly difficult, one having a pH of 5.7 and the other a pH of 11.5. The wastes were aerated without prior seeding with sewage. In the treatment of the waste with pH 11.5, there was a reduction in the pH of the effluent after 48 hours aeration. The final pH was 8.2. All during the aeration study period, the lagoons operated without odors. The only problem encountered was the production of considerable amounts of foam, especially on warm, humid days. The foaming resulted from the large quantities of detergents and soaps used in the dyeing and finishing processes. They found that anti-foam agents and a spraying system using effluent from the lagoon gave favorable results for the reduction of the foam. Although color was not measured, it appeared to be reduced, and the effluent was a uniform grey, regardless of the color of the influent. Sludge accumulations in the settling lagoons were small, as a result of the low amount of suspended solids in the wastes.

Souther (23) reported that as the aeration time increased from 16 hours to 36 hours in the treatment of a vat dye waste-sewage mixture, the B.O.D. removal increased from 65 to 94 per cent and the color removal increased from 60 to 76 per cent. He also showed that for a textile waste with a pH less than 7.0, there was an increase in the pH upon aeration. For a waste with a pH greater than 7.0, a reduction in pH was effected during aeration.

Pinault (12) reported that an aerated lagoon treatment plant at Canton Cotton Mills, Canton, Georgia, was obtaining 93 per cent B.O.D. removal with practically no residual color. The waste was aerated for 24 hours.

According to pilot plant studies, Lauria and Willis (9) and Jones (7) have reported results consistent with those reported by Souther et al (21), Williams et al (27), and Souther (23).

It would appear that a combination of the aerated lagoon and the chemical coagulation process would be an acceptable treatment process for dye wastes. The effects of the aeration system seem to produce a substantial reduction in B.O.D., and the chemical coagulation process produces a large reduction in color. With the B.O.D. reduction, the aerated lagoon may reduce the chemical coagulant demand of the waste; therefore, dye wastes could be effectively treated at a minimum cost.

III. DYEING PROCEDURE AND WASTE COMPOSITION

The textile wastes chosen for this investigation were those that have been offenders because of their volumes, strengths, color properties, and presentment of treatment problems. The two wastes chosen were under the category of dark dispersed dyes which have been increasing in popularity over the past several years. These dye wastes, 701 Black and 803 Brown Dispersed Dyes, were simulated dye wastes and were prepared by Dan River Mills, Danville, Virginia. The formulae of the dye bath mixtures and the dyeing procedure were also obtained from Dan River Mills.

Composition of Wastes

The wastes were very similar, with the main difference having been the type of dye material used.

A. 701 Black Dispersed Dye Waste

The dye was used for yarn dyeing of a 50 per cent Fortrel/ 50 per cent Cotton blend. The color of the dye waste was brown, but the yarn was dyed black. The final pH of the waste was 5.8 at the end of the dyeing run. The dye waste was composed of the following materials:

1. 3.0 per cent, Foron Black K Powder (this includes Foron Navy 2 GL, Foron Yellow Brown 2RFL, and Foron Rubine GFL).
2. 3.8 per cent, Dispersing Agent 8 (this is a 33 per cent solution of Tamol SN).
3. 1.0 per cent, Calgon.

4. 1.0 per cent, Monosodium Phosphate.
5. 1.15 per cent, Sodium Nitrite.
6. Water

B. <u>803 Brown Dispersed Dye Waste</u>

This dye was also used for yarn dyeing of a 50 per cent Fortrel/50 per cent Cotton blend. The color of the dye waste was reddish-brown, but the yarn color was brown. The pH of the waste was 6.2 at the end of the dyeing run. The dye waste was composed of the following compounds:

1. 0.95 per cent, Amacron Orange LS.
2. 1.14 per cent, Amacron Blue RLS.
3. 0.35 per cent, Eastman Polyester Red B.
4. 3.8 per cent, Dispersing Agent 8 (this is a 33 per cent solution of Tamol SN).
5. 1.0 per cent, Calgon.
6. 1.0 per cent, Monosodium Phosphate.
7. 1.15 per cent, Sodium Nitrite.
8. Water

Dyeing Procedure

The dyeing procedures for yarn dyeing of Fortrel/Cotton blends using 701 Black and 803 Brown Dispersed Dyes were the same. The procedure was as follows:

1. Yarn was washed in an 80 liter dye machine with hot water until all traces of tint were removed.

2. The dye machine, with the yarn inside, was set at 130°F, and two grams per liter of Dispersing Agent 8 was put into the machine. The machine was operated to provide agitation and a 130°F temperature for five minutes.
3. The dispersed dyes, which included the dye and dispersing agents, were added at the end of the five minute run.
4. 1.0 per cent of the Monosodium Phosphate and 1.0 per cent of the Calgon were then added. Both were in aqueous solutions and diluted to approximately 10 per cent solutions before adding to the machine. The Monosodium Phosphate was used to control the pH of the dye.
5. The dye machine was run for 10 minutes, and the pH of the dye bath was recorded. The pH of the 701 Black Dispersed Dye was 6.0, and the pH of the 803 Brown Dispersed Dye was 6.1.
6. After the pH reading, one gram per liter of Sodium Nitrite was added to the dye machine. This chemical was needed to prevent the decomposition of the dye constituents.
7. The dye machine was heated to 200°F over a 20 minute period.
8. The machine was closed off and heated to 265°F.
9. The yarn was dyed for 30 minutes at 265°F.
10. At the end of this 30 minute period, the dye machine was cooled to 200°F. The dye liquor was then dropped from the machine with the final pH of the 701 Black Dispersed Dye at 5.8, and the pH of the 803 Brown Dispersed Dye at 6.2.

This final dye liquor was the waste used in this investigation.

IV. METHODS AND MATERIALS

Experimental Materials

A. Reagents

1. Dextrose, anhydrous, ($C_6H_{12}O_6$), (d-glucose), certified reagent, (Fisher). Used in obtaining a standard curve for C.O.D. analysis with the Technicon Auto-Analyzer.
2. Ferric Chloride, ($FeCl_3 \cdot 6H_2O$), lump, C.P., (Fisher). Used in a 1 Normal solution as a coagulant for removal of color from black and brown dye wastes in the coagulation test.
3. Ferroin Indicator Solution, (1,10-Phenanthroline Ferrous Sulfate Complex), (Fisher). Used as an indicator for conventional C.O.D. analysis.
4. Ferrous Ammonium Sulfate, ($Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$), granular, certified reagent, (Fisher). Used as a titrant for conventional C.O.D. analysis.
5. Ferrous Sulfate, ($FeSO_4 \cdot 7H_2O$), crystal, certified reagent, (Fisher). Used in a 1 Normal solution as a coagulant for removal of color from the black dye waste in the coagulation test.
6. Lime, high calcium hydrate, ($Ca(OH)_2$), chemical grade, (National Gypsum Company). Used in a 1 Normal solution as a coagulant in combination with ferric chloride and ferrous sulfate for the removal of color from black and brown dye wastes in the coagulation test. Also used to adjust the pH

of black and brown dye wastes for coagulation test.

7. Mercuric Sulfate, ($HgSO_4$), powder, analytical grade, (Baker). Used in the conventional and Technicon Auto-Analyzer C.O.D. analysis to eliminate interference due to chlorides in the dye wastes.

8. Potassium Dichromate, ($K_2Cr_2O_7$), fine crystals, certified reagent, (Fisher). Used as an oxidant in the conventional and Technicon Auto-Analyzer C.O.D. analyses.

9. Potassium Hydroxide, 45 per cent, (KOH), (Hach). Used for removing carbon dioxide in the manometric B.O.D. determinations.

10. Purifloc C31 Flocculant, cationic polyelectrolyte, (Dow). Used in a one per cent solution as a coagulant for the removal of color from the brown dye waste in the coagulation test.

11. Silver Sulfate, (Ag_2SO_4), powder, reagent grade, (Baker and Adamson). Used as a catalyst in the conventional and Technicon Auto-Analyzer C.O.D. analysis to assist in oxidizing straight-chain compounds in the dye wastes.

12. Sodium Hydroxide Solution, 50 per cent W/W, (NaOH), certified reagent, (Fisher). Used in neutralizing dye waste samples that have been acidified for storage.

13. Standard pH Buffer, (Monopotassium Phosphate-Sodium Hydroxide Buffer), 0.05 Molar, ($pH\ 7.00 \pm 0.02$ @ 25°C), certified reagent, (Fisher). Used as a buffer for standardizing the

pH meter.

14. Standard pH Buffer, (Potassium Biphthalate Buffer), 0.05 Molar, (pH 4.00 ± 0.02 @ 25°C), certified reagent, (Fisher). Used as a buffer for standardizing the pH meter.
15. Sulfamic Acid, (H_2NSO_2OH), granular, certified reagent, (Fisher). Used in the conventional and Technicon Auto-Analyzer C.O.D. analysis to eliminate interference due to nitrites in the dye waste.
16. Sulfuric Acid, concentrated, (H_2SO_4), reagent grade, (Fisher). Used in digestion mixture for Technicon Auto-Analyzer C.O.D. analysis; used in the conventional C.O.D. analysis; used to acidify dye waste samples for storage; and used in a 1 Normal solution to adjust the pH of black and brown dye wastes for the coagulation test.

B. Apparatus

1. Analytical Balance, (Sartorius). Used for determining accurate weights in experiments.
2. Beakers, 1000 ml, (Corning), Pyrex Brand Glass. Used in "jar tests" or coagulation tests.
3. Burettes, 25 ml, (Fisher). Used in titrations.
4. Condensers, (Friedrichs), Pyrex Brand Glass with 24/40 Ground-Glass Joints. Used as part of the reflux apparatus in the conventional C.O.D. analysis.
5. Desiccator, (Fisher). Used to cool the reagents, the residues, and their containers to room temperature after drying in the

drying oven.

6. Drying Oven, (Precision). Used to dry reagents and samples from suspended solids tests.
7. Extraction Heating Unit and Support, Six Electrically Heated Units, (Precision). Used as part of the reflux apparatus in the conventional C.O.D. analysis.
8. Flat-Bottom Flasks, 500 ml, (Corning), Pyrex Brand Glass with Long Neck and 24/40 Ground-Glass Joints. Used as part of the reflux apparatus in the conventional C.O.D. analysis.
9. Gas Diffuser Stones, (Fisher). Used to diffuse the air in the aerated lagoon process.
10. Glass Fiber Filters, (Reeve Angel). Used in filtration for determining suspended solids.
11. Gooch Crucibles, (Coors). Used in filtration for determining suspended solids.
12. Hach Manometric B.O.D. Apparatus, Model 1791, (Hach). Used for B.O.D. determinations.
13. Harvard Trip Balance, (Ohaus). Used for determining less accurate weights in experiments.
14. Magnetic Mixer, (Wilkens-Anderson). Used to mix solutions.
15. Multiple Stirrer, (Phipps and Bird). Used for "jar tests" or coagulation tests.
16. Oxygen Meter, YSI Model 51, (Yellow Spring Instrument Company). Used to measure the dissolved oxygen content of the dye wastes during aeration.

17. <u>pH Meter with Glass Electrodes</u>, (Leeds and Northrup). Used to measure pH of samples.
18. <u>Spectronic 20 Colorimeter/Spectrophotometer</u>, (Bausch and Lomb). Used to determine color removals from the dye wastes after treatment.
19. <u>Technicon Auto-Analyzer</u>, (Technicon). Used to determine C.O.D. of samples.

<u>Experimental Methods</u>

The purpose of this investigation was to determine if aerated lagoon treatment would effectively reduce the chemical coagulant demand of dark dispersed dye wastes, while removing a large percentage of the color and other pollutional matter from the wastes. Two simulated dye wastes, 701 Black and 803 Brown Dispersed Dyes, were prepared by Dan River Mills, Danville, Virginia. The wastes, at 200°F after leaving the dye machine, were cooled to 20°C (68°F) before being treated.

Initial tests were performed on the raw dye wastes. The wastes were then aerated in a simulated aerated lagoon. Samples were taken from the aerated lagoon at specified periods of from one to five days, and tests were performed on these samples. The tests conducted on the raw and aerated samples included the manometric B.O.D. determination, conventional C.O.D. analysis, Technicon Auto-Analyzer C.O.D. analysis, and chemical coagulation tests or "jar tests." Other tests that were performed were the suspended solids tests, color removal analysis, and pH tests. Color removal analysis, pH tests, and a few conventional C.O.D. analyses were performed on the supernatant from the coagulation tests. The amount of sludge and appearance of supernatant were noted in each coagulation test.

The following is a description of the manner in which the experiments and tests were conducted:

Experiment 1: Treatment of 701 Black Dispersed Dye Waste

Aerated Lagoon Treatment

The biological process used in this investigation was a simulated aerated lagoon, except that the lagoon was a batch rather than a continuous-flow system. For the investigation of the effect of time of aeration on effluent properties (C.O.D., coagulant demand, etc.), the batch process was considered to be more practical.

The aeration tank or lagoon consisted of a 20 gallon plastic trash can. Air was supplied through four porous gas diffuser stones. The air was first passed through a cotton plug filter to remove oil, moisture, and other contaminants. The entire aeration apparatus was placed in a constant temperature (20°C or 68°F) room to insure that the treatment would not be affected by fluctuations in the ambient temperature.

Nine gallons (34.07 liters) of dye waste and one gallon (3.79 liters) of biological seed were placed in the trash can or aeration tank. The biological seed used was trickling filter effluent from the Stroubles Creek Sewage Treatment Plant of the Blacksburg - VPI Sanitation Authority. An air flow was provided to produce a dissolved oxygen (D.O.) concentration greater than one part per million (p.p.m.). The D.O. was checked periodically with an Oxygen Meter and D.O. probe.

There was a substantial amount of foaming of the waste in the aeration tank, but enough freeboard above the waste was provided to prevent the foam from becoming a problem. The foam subsided considerably after the first few days of aeration.

Samples of waste were taken from the aeration tank after 32.5, 49.5, 66.5, 112.5, and 184.5 hours of aeration. The size of each sample taken was approximately seven liters.

pH Determination

A Leeds and Northrup pH Meter with a glass-calomel electrode system was employed to measure the pH value of samples directly. The pH value was recorded for the raw and aerated samples. Also, the pH value was found for the supernatant of the coagulation tests after 45 minutes and after 24 hours settling. The pH meter was standardized with buffer solutions of pH 4.0 and 7.0.

Suspended Solids Test

The concentration of suspended solids was determined for the raw and aerated samples. The suspended solids test was performed as described in Standard Methods for the Examination of Water and Wastewater (25), with the following exceptions. The Gooch crucible method was employed using a 2.1 centimeter (diameter) glass fiber filter in place of the asbestos mat. Also, the drying time for determining suspended solids was increased from one hour to 24 hours. A 25, 50, or 100 milliliter sample was used in the experiments, with the size of sample decreasing from 100 to 25 milliliters as the suspended solids concentration became greater.

B.O.D. Analysis

The B.O.D. test was conducted on the raw and aerated samples. The method employed was the direct B.O.D. determination using the Hach Manometric B.O.D. Apparatus (Model 1791). The samples were not buffered or neutralized before performing the B.O.D. tests. The samples were not buffered because the original dye was buffered during the dyeing procedure (see Chapter III, page 22). They were not neutralized because the pH values ranged between pH 6.0 and 7.0. Each sample was diluted nine to one with distilled water for the B.O.D. tests. A mixture of 14.1 milliliters of sample, 127.2 milliliters of oxygen saturated distilled water, and 15.7 milliliters of biological seed was added to the sample bottle on the apparatus. The mixture totaled 157 milliliters. The seed was effluent from the previously mentioned trickling filter. This seed was chosen because its B.O.D. was low, but it was biologically active. A wick and cup containing four drops of potassium hydroxide was put into each sample bottle on the apparatus. After each sample was set up on the B.O.D. apparatus, the system was closed, and the utilization of oxygen was observed daily over a period of seven to eight days at a temperature of 20°C. Manometer readings were taken periodically (once a day), recorded, and plotted on a graph.

The proper B.O.D. value was found by increasing the manometer reading by a factor of 10 because the sample was diluted to 10 per cent.

A more detailed discussion of the operation of the B.O.D. apparatus is given in the manual accompanying the apparatus.

Conventional C.O.D. Analysis

The conventional C.O.D. analysis was conducted on the raw and aerated samples. The C.O.D. analysis was performed as described in *Standard Methods for the Examination of Water and Wastewater* (25), with the following exceptions. Flat-bottomed flasks were used as the refluxing flasks in place of the erlenmeyer flasks. The sulfamic acid, used to eliminate interference due to nitrites, was added to the C.O.D. reagents in each test, instead of being added to the potassium dichromate solution. One gram of silver sulfate was added to the C.O.D. reagents in each test, instead of adding it to a nine-pound bottle of sulfuric acid.

The samples that were tested were diluted to 10 and 20 per cent, and one to four tests were run on each sample. The results from the tests for each sample were averaged and recorded.

Technicon Auto-Analyzer C.O.D. Analysis

A C.O.D. analysis was conducted on the raw and aerated samples using the Technicon Auto-Analyzer. This method of analysis was used as a check on the conventional C.O.D. tests. The raw and aerated samples were acidified with concentrated sulfuric acid. The final pH values of the acidified samples ranged between pH 0.8 and 1.2. The samples were then stored until after the last sample was taken from the aerated lagoon. At this time, the samples were neutralized with 50 per cent W/W sodium hydroxide. All the neutralized samples were diluted to 10 and 20 per cent. Two 10 per cent dilutions and two 20 per cent dilutions were tested for each sample. Mercuric sulfate, sulfamic acid, and silver sulfate were added to the other reagents in this test, and they were added in the same quantities as described for the conventional C.O.D. analysis in the preceding section. In this method for the determination of C.O.D., each sample was digested with known amounts of potassium dichromate and sulfuric acid. The depletion in color due to the oxidation of organic materials in the sample was then measured colorimetrically and recorded on the chart of a recorder. The C.O.D. of the samples was found by comparing the responses of unknown samples with those of known glucose standards, which were plotted to give a standard curve (see Figure 3, page 47). For this investigation, a flow diagram of the system for the measurement of C.O.D. is shown in Figure 1.

A more detailed discussion of the operation of the Technicon Auto-Analyzer for C.O.D. determination is given in the manual accompanying the machine.

Coagulation Test

The coagulation tests or jar tests were performed to obtain the optimum chemical coagulant demand of the raw and aerated samples.

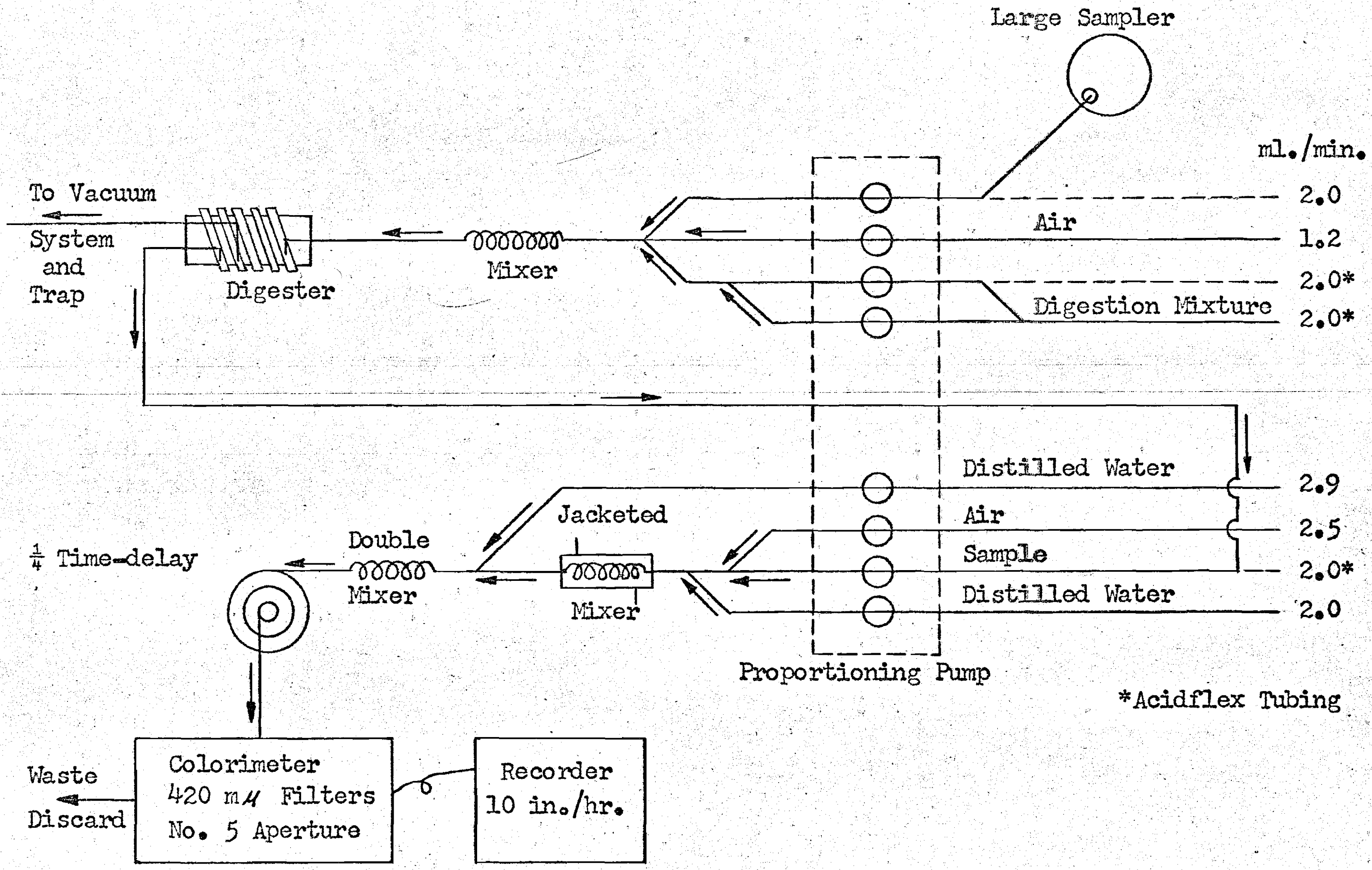


FIGURE 1. FLOW DIAGRAM OF AUTO-ANALYZER FOR C.O.D. DETERMINATION

Two coagulant combinations, ferric chloride and lime, and ferrous sulfate and lime, were used for this test. In both cases, the lime was added first because a preliminary investigation showed that the best results were obtained by this procedure. All the chemical coagulants were made up into 1 Normal solutions. The lime was prepared daily, but the ferric chloride and ferrous sulfate were prepared every one to four days.

The experimental procedure was conducted in the following manner (11). The experiment was carried out in a constant temperature (20°C) room. A preliminary investigation of the coagulant demand of each sample was conducted by varying the coagulant dosage in 25 milliliter samples. From the results of this investigation, the range of chemical dosage was decided upon for the regular coagulation tests. This investigation was performed for the raw and aerated samples. A 500 milliliter sample was now added to each of six one liter beakers accommodated in the jar test apparatus. The jar test apparatus was a laboratory Multiple Stirrer manufactured by Phipps and Bird. The stirrers on the apparatus were started and run at 100 revolutions per minute (r.p.m.). The coagulants were added to the beakers with increasing dosages from left to right, and with the third or fourth beaker receiving a dosage estimated to be correct. Equal amounts (normality basis) of lime and ferric chloride or ferrous sulfate were used in the tests. The coagulants were mixed with the samples at 100 r.p.m. for one minute. The stirring rate was reduced to 45 r.p.m., and the mixing was continued for an additional

30 minutes. The stirrer was turned off, and the mixtures were settled for 45 minutes. At this time, the appearance of the supernatant was observed, and the amount of sludge was measured. The sludge was measured by the graduations on the side of the beakers. The graduations were accurate to within ± five milliliters. The pH of the supernatant was measured, as was the amount of color removal effected by the chemical coagulation. From these results, the optimum coagulant dosage was determined.

For the last sample taken from the aeration tank, the effect of pH on coagulation with ferric chloride and lime was determined. A coagulant dosage was arbitrarily decided upon for the optimum pH test. A preliminary test was performed to obtain a titration curve for the aerated sample with the dosage of coagulant added. The sample and coagulants were mixed well and divided into two beakers. One beaker was titrated with 1 Normal sulfuric acid. The second beaker was titrated with 1 Normal lime. From the titration curve obtained, the amount of lime or sulfuric acid needed to produce a certain pH was found. A 300 milliliter sample was added to each of six one liter beakers. The samples were mixed at 100 r.p.m. while the dosage of ferric chloride and lime was added to the six beakers. The pH was varied by adding sulfuric acid in the first two beakers and lime in the last three beakers. The third beaker was not treated with acid or alkali. The samples were dosed so as to provide a pH in each beaker varying in increments of 0.5 to 1.5 pH units. The samples were mixed for one minute at

100 r.p.m. and stirred for 30 minutes at 45 r.p.m. The mixtures were settled for 45 minutes and for 24 hours. At the end of the settling times, the appearance was observed, and the color removal, pH, and sludge volume were measured. From these results, the optimum pH for the coagulation was determined.

Color Removal Analysis

The amount of color removal was determined indirectly for the aerated samples (after one hour sedimentation) and for the supernatant samples from the coagulation tests. The apparatus used to determine the color removal was the Spectronic 20 Colorimeter/Spectrophotometer manufactured by Bausch and Lomb. The manual accompanying this instrument was helpful in providing information as to the operation of the machine.

The method used for obtaining color removal values involved performing an absorption spectral analysis and getting the maximum absorption of light in a sample from an absorption spectrum curve. This light absorption value was then entered into a standard curve from which the amount of color removal was obtained.

The details of the absorption spectral analysis were performed in the following manner. It was found that for the dye waste used, the red part of the wavelength scale on the Spectronic 20 was needed for the color removal determinations. This scale required the use of an accessory red filter (Cat. No. 33-29-18) and a red sensitive phototube (Cat. No. 33-29-72). First, the Spectronic 20 was adjusted with a distilled water blank so that between the wavelengths of 600 and 800 millimicrons (mμ), the per cent transmittance of light through the water increased from 25 per cent to 100 per cent. From 800 to 975 mμ the per cent transmittance decreased from 100 per cent to nine per cent. A curve resulting from a plot of this information was obtained for each sample tested for color removal. This curve was called a spectrum curve, and all curves prepared in the same manner were called spectrum curves. The other spectrum curves were all made with the Spectronic 20 adjusted with the distilled water blank. The distilled water spectrum curve acted as a base line with all the sample spectrum curves falling below it.

The effect of pH on the color removal analysis was determined by using three samples of waste with varying pH values. The pH values of the three samples were 4.65, 6.15, and 8.00. The pH was varied by adding 1 Normal sulfuric acid or 1 Normal sodium hydroxide. A spectrum curve was obtained for each of the samples using the same wavelengths as used for the distilled water blank. The curves were very similar, so a pH adjustment of any future samples was not needed.

A standard curve was drawn using dilutions of the raw dye waste (see Figure 7, page 50). Samples of waste were diluted to 75.0, 50.0, 37.5, 25.0, 12.5, 5.0, and 1.0 per cent. A spectrum curve was obtained for each diluted sample and for a raw sample. These spectrum curves were plotted on the same graph as the distilled water spectrum curve. The maximum absorption of light was found for each sample. This was done by determining the maximum vertical distance between each of the sample curves and the distilled water curve, and dividing this distance by the total vertical distance under the distilled water curve. It was found that the maximum light absorption for each sample occurred at different wavelengths, usually between 675 and 825 mμ. Because the maximum light absorption occurred at different wavelengths, it was decided that a spectrum curve should be obtained for all samples used in the color removal analysis. This method was used instead of choosing a median wavelength, such as 750 mμ, and using the vertical distance between the two curves divided by the total distance under the distilled water curve at the 750 mμ wavelength. The maximum light absorption values for the raw and diluted samples were plotted against the per cent dilution to form a standard curve.

The per cent color removal of the aerated and supernatant samples was obtained by plotting a spectrum curve, determining the maximum light absorption, and referring this light absorption value to the standard curve. The per cent color removal was the complement of the per cent dilution value obtained from the curve.

At the end of the experiment on the 701 Black Dispersed Dye, an alternate method for determining the per cent color removal was evaluated. In the alternate method, the median wavelength of 750 mμ was chosen as the wavelength where the maximum light absorption occurred. The maximum light absorptions for the raw and diluted samples used in making the standard curve were obtained from the previously drawn spectrum curves of the samples. These light absorption values were plotted against the per cent dilution of the samples to give an alternate standard curve. From the spectrum curves of several of the aerated and supernatant samples, the maximum light absorption was determined for the wavelength of 750 mμ. The per cent color removal was determined from the alternate standard curve by the same method as described previously. It was found that the two methods of determining the per cent color removal gave very similar results. The results differed from between zero and two per cent color removal. It was decided that the alternate method would be satisfactory for determining color removals in any following experiments. The alternate method was much faster than the original method.

Examples of the original and alternate methods of color removal analysis are given in Appendix A.

Experiment 2: Treatment of 803 Brown Dispersed Dye Waste

Aerated Lagoon Treatment

The procedures for the aerated lagoon in "Experiment 1" were used for this experiment, with the following exceptions. The type of seed used, and the amount of waste aerated were changed. The biological seed contained a mixture of acclimated dye waste seed and effluent from the previously mentioned trickling filter. The mixture contained 50 per cent of each biological seed. Also, the amount of waste and seed placed in the aeration tank were increased to 13 gallons (49.27 liters) and 1.3 gallons (4.93 liters), respectively.

Samples of waste were taken from the aeration tank after 51, 76, 103.5, 124.5, 172, and 244 hours of aeration. The size of samples taken were approximately 8.5 liters.

pH Determination

The same procedures were used as those under pH Determination in "Experiment 1."

Suspended Solids Test

The same procedures were used as those under Suspended Solids Test in "Experiment 1."

B.O.D. Analysis

The same procedures were used as those under B.O.D. Analysis in "Experiment 1," with the following exceptions. The B.O.D. readings were taken once a day over a period of seven to eight days. Only two drops of potassium hydroxide were used in the wick and cup placed in each manometric monitored bottle.

Conventional C.O.D. Analysis

The same procedures were used as those under Conventional C.O.D. Analysis in "Experiment 1." The C.O.D. analysis was conducted on raw, aerated, and three supernatant samples. The C.O.D. of the three supernatant samples was obtained to determine approximately how much C.O.D. the coagulation process removed after 45 minutes settling. Two of the supernatant samples were from a ferric chloride and lime coagulation test. The other sample was from a Purifloc C31 coagulation test.

Technicon Auto-Analyzer C.O.D. Analysis

The same procedures were used as those under Technicon Auto-Analyzer C.O.D. Analysis in "Experiment 1." The same flow diagram was used as that shown in Figure 1, with the exception that a Number 7 aperture was used in place of the Number 5 aperture in the colorimeter.

Coagulation Test

The same procedures were used as those under Coagulation Test in "Experiment 1," with the following exceptions. A new coagulant, Purifloc C31 (Purifloc is a registered trade name), was used in place of ferrous sulfate and lime. Purifloc C31, a cationic polyelectrolyte, was made up into a one per cent solution. This solution was prepared every two to five days. The ferric chloride and lime were prepared daily. Three-hundred milliliter samples were used instead of 500 milliliter samples. For all the samples, the optimum coagulant dosage and the optimum pH for the coagulation tests were determined. All the coagulated samples were settled for 45 minutes and for 24 hours. The appearance was observed, and the color removal, pH, and sludge volume were measured at the end of each settling time.

Color Removal Analysis

The alternate method for color removal that was explained in "Experiment 1," under Color Removal Analysis, was used in this experiment. This method was used because its accuracy was comparable to that of the original method. Also, the alternate method was less time consuming. The standard curve used in this experiment is shown in Figure 16, page 74.

V. EXPERIMENTAL DATA AND RESULTS

A. Experiment 1: Treatment of 701 Black Dispersed Dye Waste

The objective of this experiment was to determine if aerated lagoon treatment would effectively reduce the chemical coagulant demand of 701 Black Dispersed Dye Waste.

1. Conventional C.O.D. Analysis

The purpose for conducting the conventional C.O.D. analysis was to obtain immediate performance figures for the aerated lagoon treatment after 0, 32.5, 49.5, 66.5, 112.5, and 184.5 hours of aeration. The procedure was to obtain a C.O.D. value for the raw dye waste and for the aerated lagoon samples after the above aeration periods. The samples were analyzed as described in the Methods and Materials chapter.

The results of the conventional C.O.D. analysis on the raw and aerated samples are shown graphically in Figure 2. These results indicated that the aerated lagoon produced reductions in the C.O.D. of the dye waste. Figure 2 shows that 66.5 hours of aeration effected the greatest C.O.D. reduction.

It was noted in Figure 2 that the samples taken after 112.5 and 184.5 hours of aeration did not follow the decreasing C.O.D. trend as found in the previous samples. This may be possibly attributed to changes in the chemical structures making up the dye waste. Aromatic hydrocarbons are not oxidized to any appreciable extent by the C.O.D. test. The

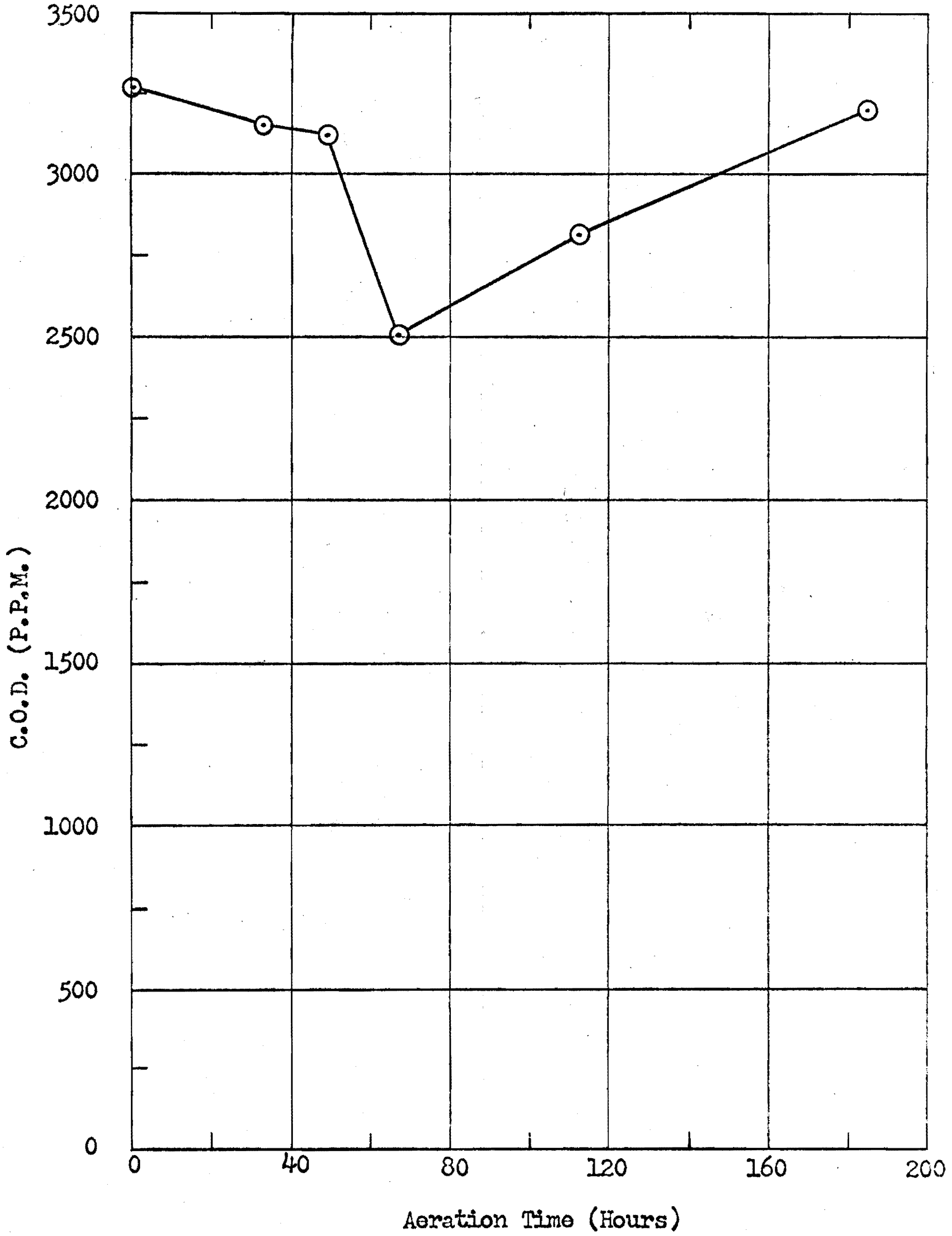


FIGURE 2. THE EFFECT OF AERATION TIME ON THE C.O.D. OF 701 BLACK DISPERSED DYE WASTE USING THE CONVENTIONAL C.O.D. ANALYSIS

dye waste contained large amounts of aromatic hydrocarbons, which may have been broken down by the aeration process. This breakdown would produce more C.O.D., the result being an increase in the C.O.D. curve shown in Figure 2.

2. Technicon Auto-Analyzer C.O.D. Analysis

The purpose of this analysis was to verify the results showing a reduction in C.O.D. by the conventional C.O.D. analysis. The C.O.D. values were found by an automated colorimetric method using the Technicon Auto-Analyzer. The raw and aerated samples were stored and then analyzed as described in the Methods and Materials chapter. A standard curve was prepared using known glucose standards. The glucose standards (100, 250, 500, 750, and 1000 p.p.m. C.O.D.) were digested with known amounts of potassium dichromate and sulfuric acid. The depletion of color due to the oxidation of the glucose was measured colorimetrically in terms of absorbance of light. A standard curve was plotted showing the relationship between C.O.D. concentration and absorbance. This curve is shown in Figure 3. Since the standard curve has a maximum C.O.D. value of 1000 p.p.m., it was necessary to dilute the unknown samples so that resulting C.O.D. values were less than 1000 p.p.m.

The results of the Technicon Auto-Analyzer C.O.D. analysis on the raw and aerated samples are shown in Figure 4. This curve indicates that 49.5 hours of aeration had the greatest

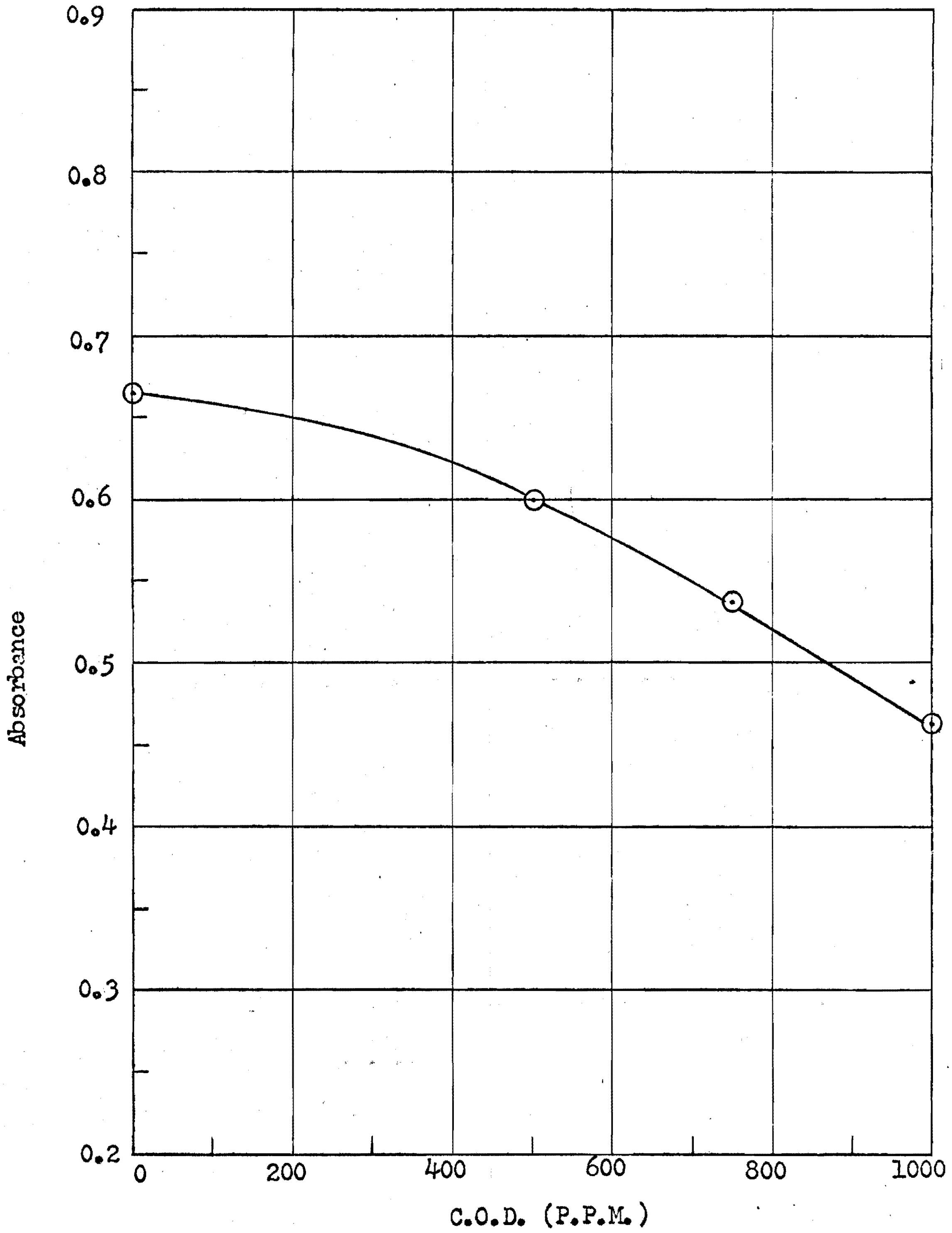


FIGURE 3. STANDARD CURVE USED FOR DETERMINATION OF C.O.D. OF 701 BLACK DISPERSED DYE WASTE BY THE TECHNICON AUTO-ANALYZER C.O.D. ANALYSIS

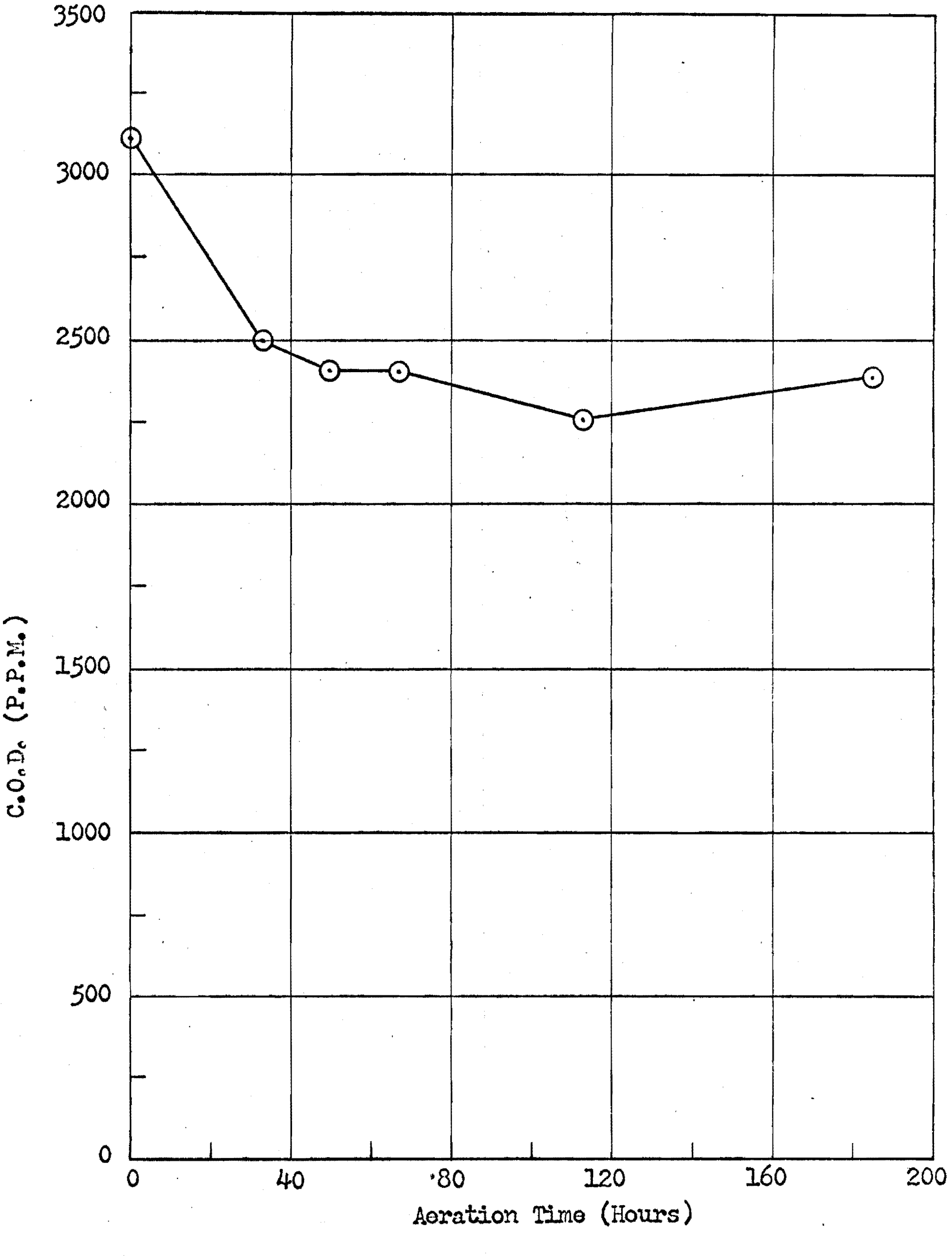


FIGURE 4. THE EFFECT OF AERATION TIME ON THE C.O.D. OF 701 BLACK DISPERSED DYE WASTE USING THE TECHNICON AUTO-ANALYZER C.O.D. ANALYSIS

practical effect on the reduction in C.O.D. This time was about one day less than the aeration time obtained by the conventional C.O.D. analysis for the best reduction in C.O.D.

The results of this C.O.D. analysis shown in Figure 4 were considerably different from the results of the conventional C.O.D. analysis shown in Figure 2. The Auto-Analyzer gave C.O.D. values that were less than those obtained by the conventional method. It was believed that the results should have been different because the procedures of the two tests were different. The main difference in the two analyses was that the samples were only digested in the Auto-Analyzer for a short time, e.g. two to five minutes, while the conventional analysis had a digestion period of two hours. The shorter digestion time would cause lower C.O.D. readings for the Auto-Analyzer, even though the Auto-Analyzer digests at a higher temperature.

The Auto-Analyzer analysis only determined C.O.D. values between 500 and 1000 p.p.m. for this experiment. The samples and standards with C.O.D. values below 500 p.p.m. did not produce results, although they were run through the machine. Points were not obtained for the 100 and 250 p.p.m. C.O.D. standards on the standard curve (see Figure 3). Also, results were not obtained for the raw and aerated samples that were diluted to 10 per cent. The reason for this limitation in the procedure was believed to have been the size of aperture used in the colorimeter. A Number 5 aperture was used, but a larger aperture probably would have produced results for all the samples.

3. B.O.D. Analysis

The B.O.D. tests were performed as a second performance parameter for the aerated lagoon. The B.O.D. performance data was obtained for the aerated lagoon after 0, 32.5, 49.5, 66.5, 112.5, and 184.5 hours of aeration. Daily B.O.D. readings were obtained for the raw and aerated samples after the above aeration periods. The procedures followed those described in the Methods and Materials chapter.

The results of the B.O.D. analysis on the raw and aerated samples are given in Table 1, and the five-day B.O.D. values are shown graphically in Figure 5. The B.O.D. results indicated that the aerated lagoon produced substantial reductions in the B.O.D. of the dye waste. Figure 5 shows that 32.5 hours of aeration had the greatest practical effect on the B.O.D. reduction. It was noted in Table 1 that the samples taken after 49.5 and 112.5 hours resulted in B.O.D. values of zero. This may be attributed to the possibility that potassium hydroxide may have spilled out of the cup and wick in the B.O.D. bottles. This would have killed the biological seed in the B.O.D. bottle, therefore causing zero B.O.D. readings.

4. Suspended Solids Test

The concentration of suspended solids was determined as a

TABLE 1

B.O.D. (P.P.M.) OF 701 BLACK DISPERSED DYE WASTE AFTER VARIOUS PERIODS OF AERATION

Time of Incubation (Days)	Aeration Time (Hours)					
	0	32.5	49.5	66.5	112.5	184.5
1	170	110	0	96	0	142
2	354	140	0	157	0	110
3	384	160	0	150	0	141
4	428	180	0	122	0	160
5	568	180	0	186	0	160
6	550	180	0	165	0	160
7	550	132	0	150	0	160
8	• •	115	0	150	0	• •

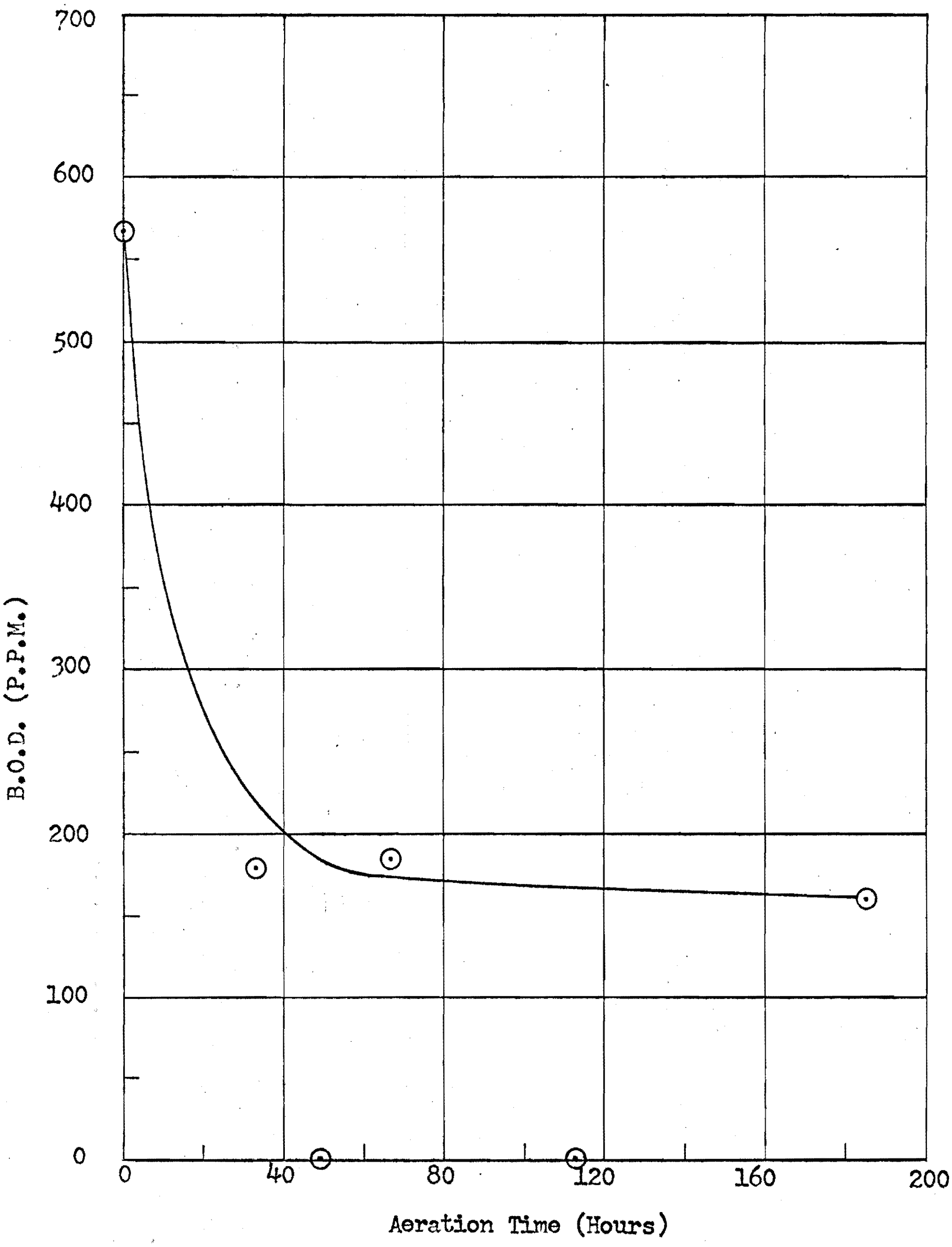


FIGURE 5. THE EFFECT OF AERATION TIME ON THE B.O.D. OF 701 BLACK DISPERSED DYE WASTE

third performance parameter for the aerated lagoon. The amount of suspended solids was an indication of the quantity of biological growth or active sludge in the lagoon. Suspended solids data were obtained for the aerated lagoon after 0, 32.5, 49.5, 66.5, 112.5, and 184.5 hours of aeration. The procedure used was the same as that described in the Methods and Materials chapter.

The suspended solids results are shown graphically in Figure 6. The increased concentration of suspended solids with time of aeration indicated that biological growth was accumulating in the aerated lagoon. This observation was an indication that the lagoon was capable of reducing the C.O.D. and B.O.D.

A suspended solids value was not obtained for the sample taken at 112.5 hours of aeration. Also, a decrease in the suspended solids was observed for the sample taken at 184.5 hours of aeration. These last two observations, the failure to obtain one suspended solids value and the decrease in another, were attributed to incorrect readings from the analytical balance.

5. Coagulation Test and Color Removal Analysis

The amount of color removal was found for each aeration sample so as to evaluate the ability of the aerated lagoon to remove color from the dye waste. The coagulation tests were used to obtain the optimum coagulant demand of the raw

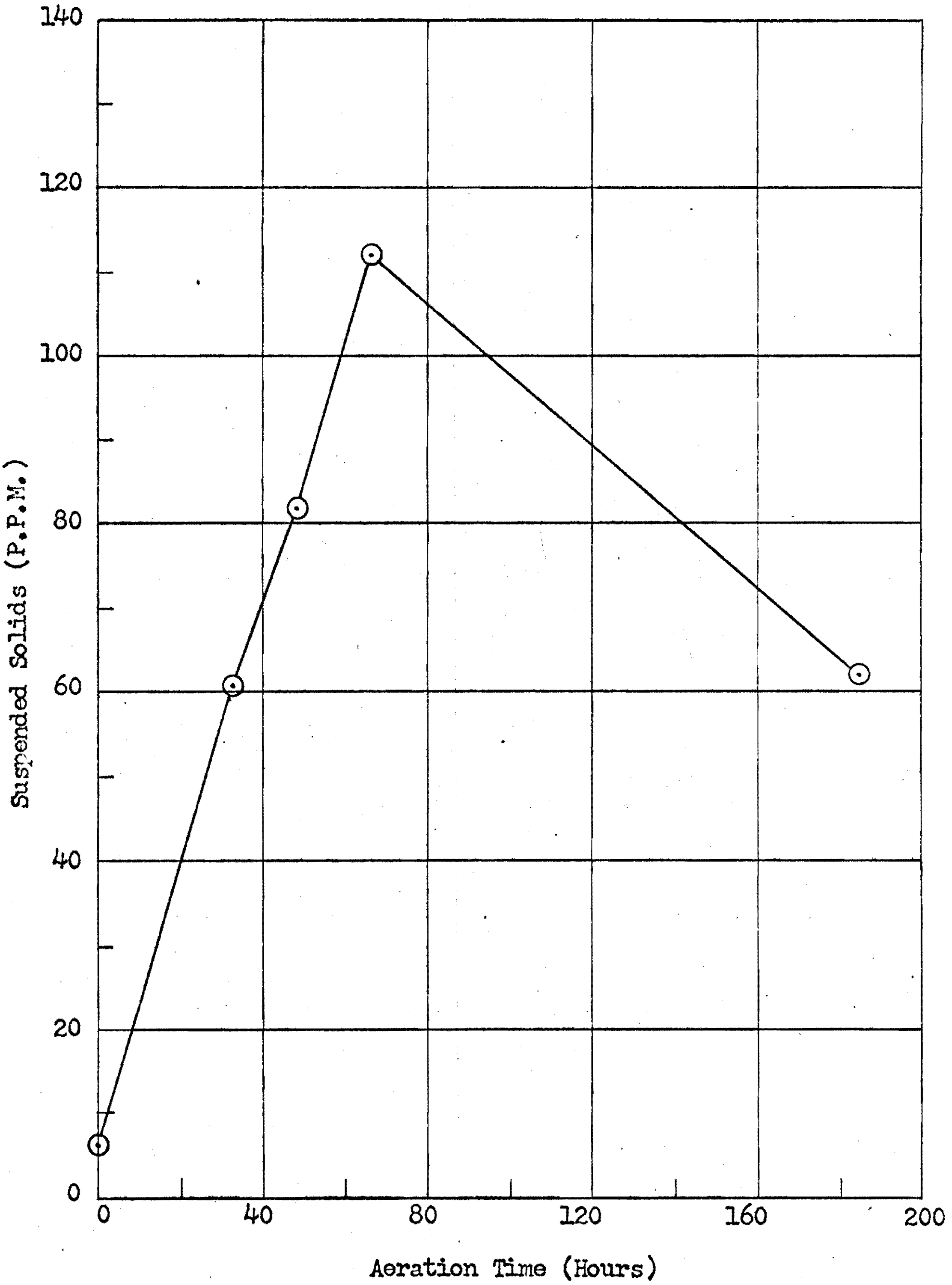


FIGURE 6. THE EFFECT OF AERATION TIME ON THE SUSPENDED SOLIDS OF 701 BLACK DISPERSED DYE WASTE

and aerated waste. The tests were also used to evaluate the aerated lagoon's ability to reduce the coagulant demand of the waste. The methods employed for coagulation and determination of color removal were as described in the Methods and Materials chapter.

The standard curve obtained for the color removal analysis is shown in Figure 7. The effect of aeration on color removal from the dye waste is shown graphically in Figure 8. The results shown in Figure 8, indicated that the aerated lagoon was relatively ineffective for the removal of color. The lower values for the removal of color at 112.5 and 184.5 hours of aeration may be attributed to the increased suspended solids concentration that developed during aeration of the mixture. Suspended solids cause less light transmittance through a sample of waste, therefore, a lower color removal was observed. Another possible reason for the lower color removal was a chemical and biological change in the dye waste, which may have produced more color in the waste.

The results of the chemical coagulation tests are tabulated in Tables 3, 4, and 5 in Appendix B. The effect of aeration time on the optimum coagulant dosage and per cent sludge volume from the coagulation tests is shown in Figures 9 and 10. The coagulant dosage giving 95 per cent color removal was designated as the optimum dosage which was listed in Table 3 and plotted in Figure 9 for ferric chloride and

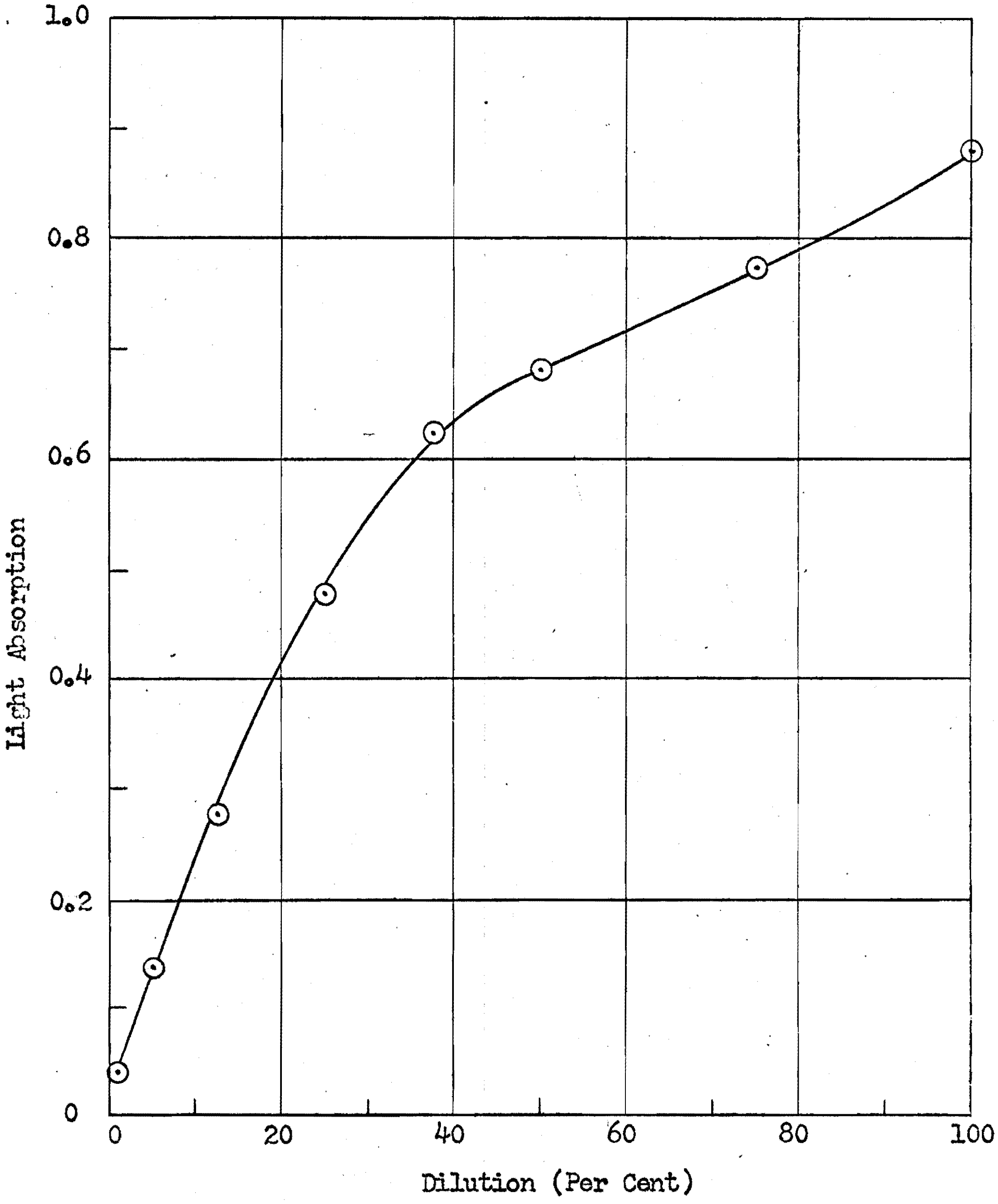


FIGURE 7. STANDARD CURVE USED FOR DETERMINATION OF PER CENT COLOR REMOVAL FOR TREATED SAMPLES OF 701 BLACK DISPERSED DYE WASTE

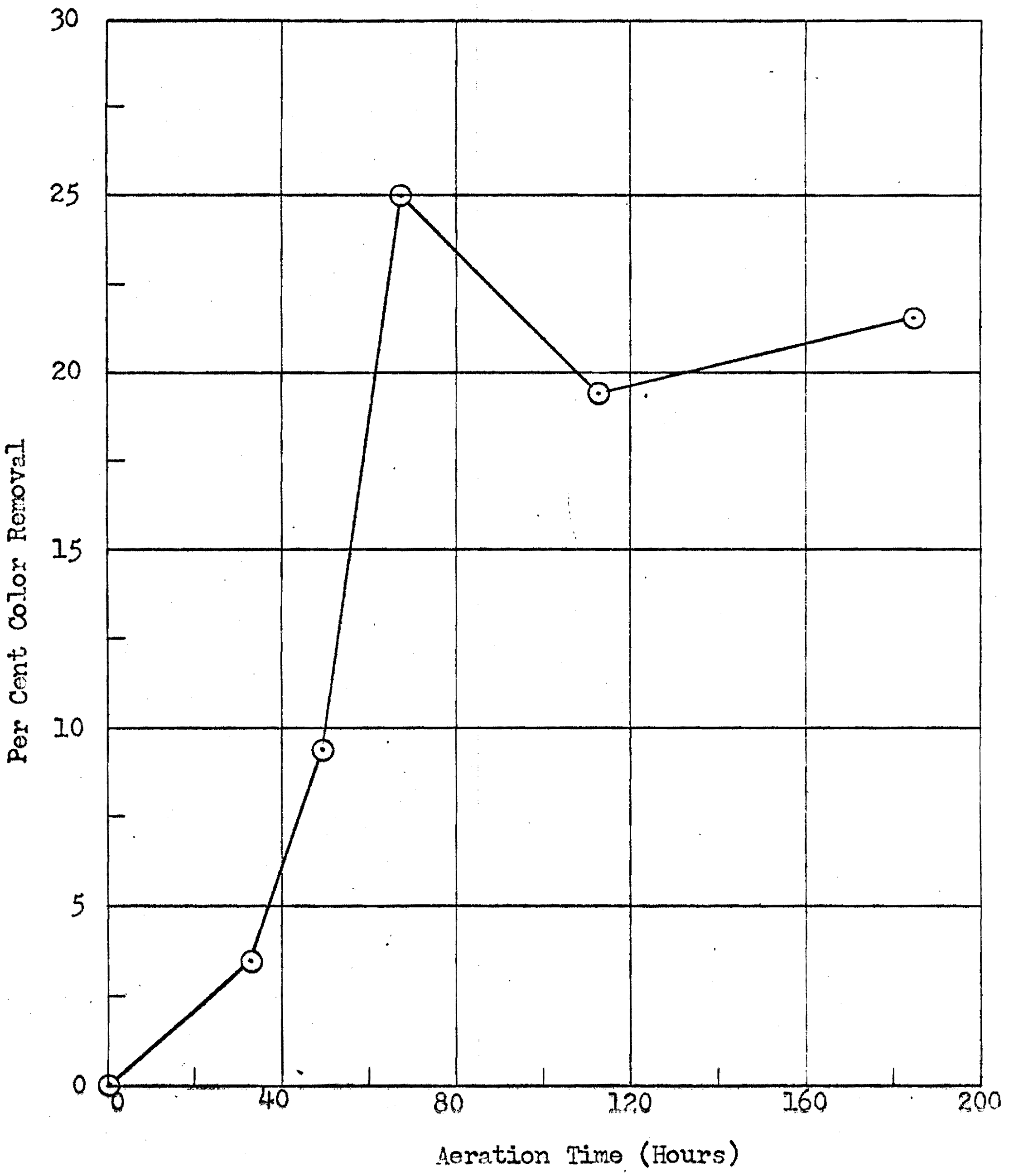


FIGURE 8. THE EFFECT OF AERATION TIME ON PER CENT COLOR REMOVAL OF 701 BLACK DISPERSED DYE WASTE

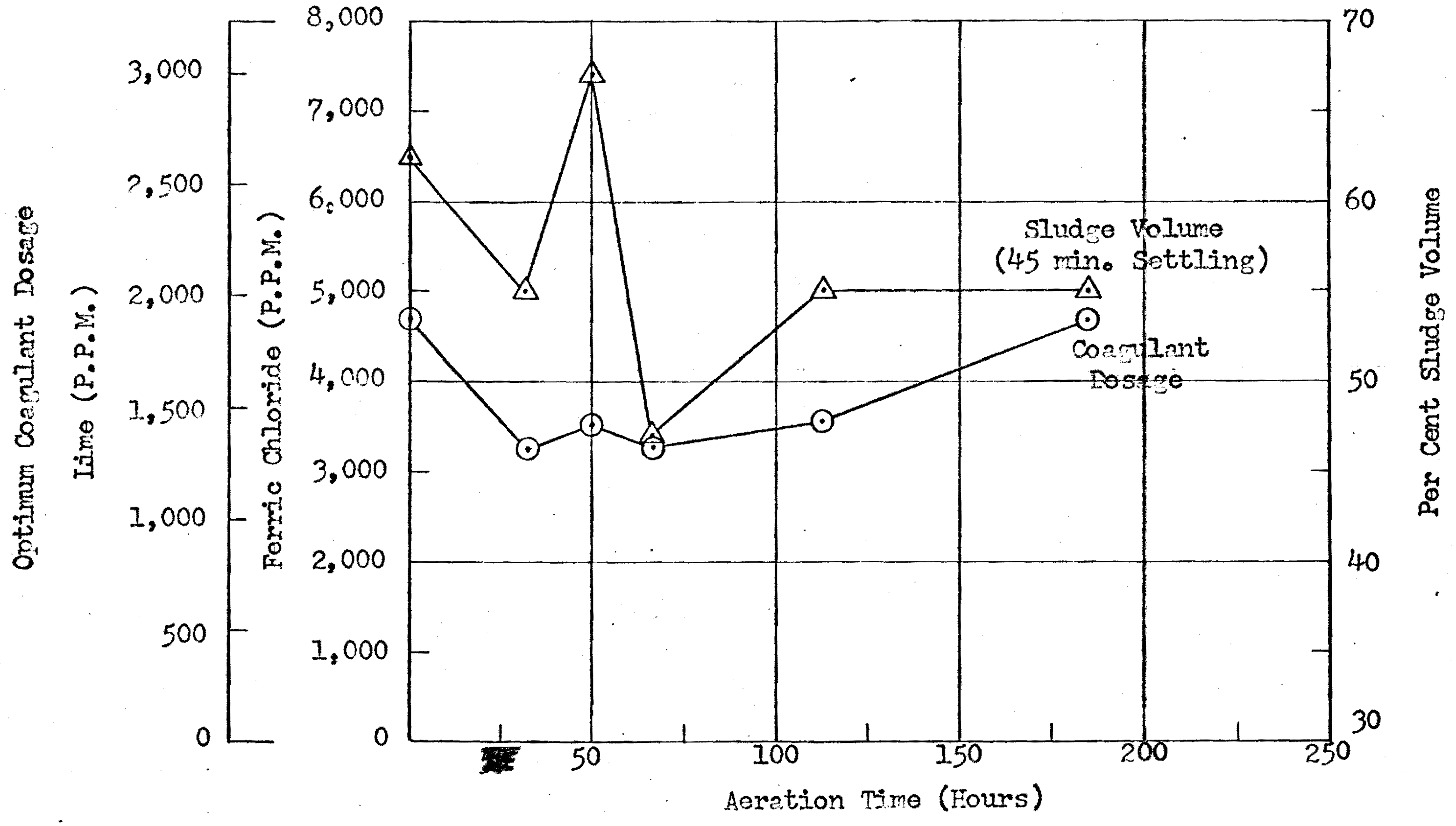


FIGURE 9. THE EFFECT OF AERATION TIME ON THE OPTIMUM COAGULANT DOSAGE AND PER CENT SLUDGE VOLUME OF 701 BLACK DISPERSED DYE WASTE USING FERRIC CHLORIDE AND LIME AS THE COAGULANT COMBINATION

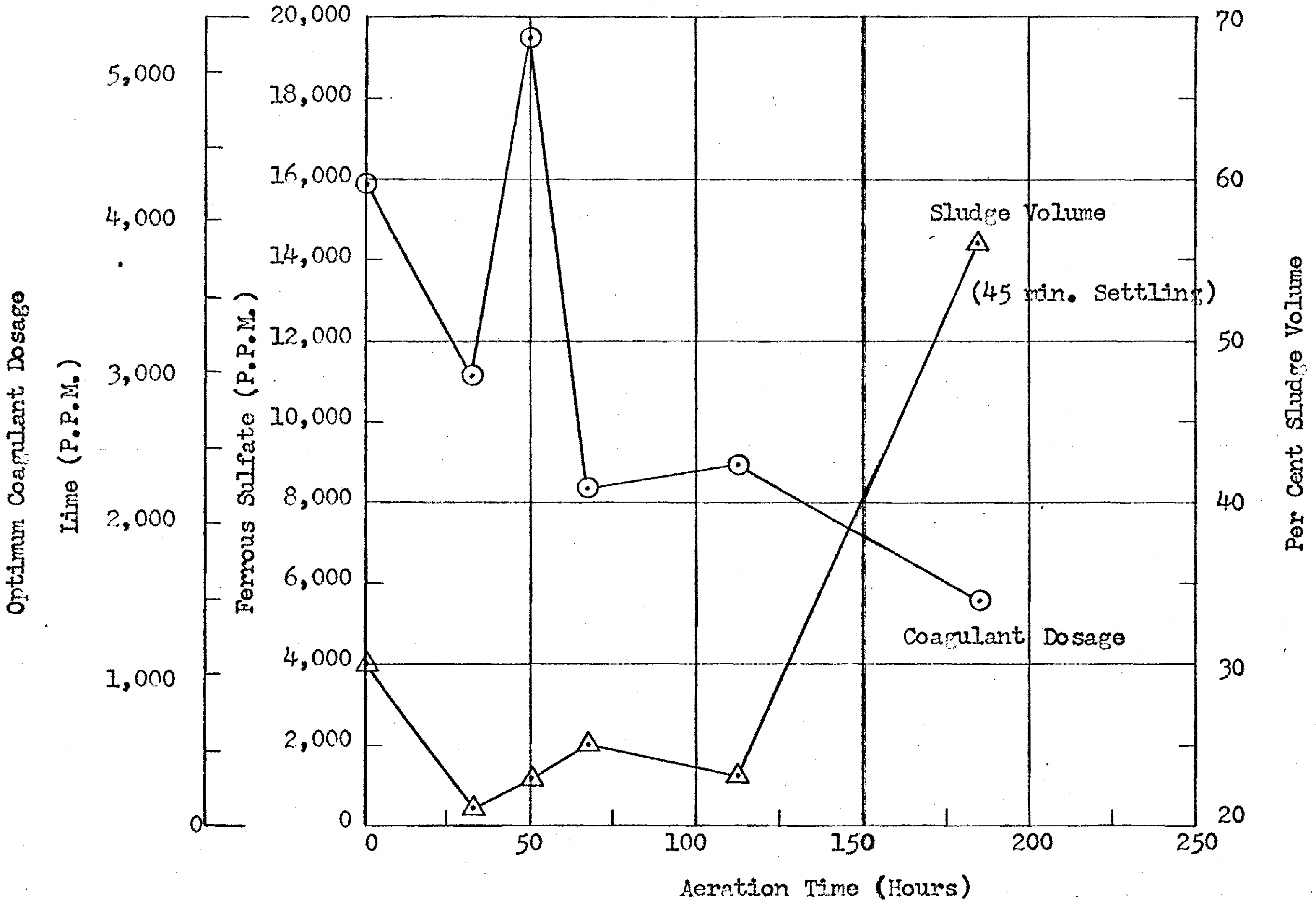


FIGURE 10. THE EFFECT OF AERATION TIME ON THE OPTIMUM COAGULANT DOSAGE AND PER CENT SLUDGE VOLUME OF 701 BLACK DISPERSED DYE WASTE USING FERROUS SULFATE AND LIME AS THE COAGULANT COMBINATION

lime. In cases where there were more than one 95 per cent color removal reading, the lowest coagulant dosage was chosen as the optimum. As shown in Table 3, the optimum coagulant dosages were usually interpolated from the data, if the coagulant dosages used did not result in exactly 95 per cent color removal. In Figure 10 and Table 4, the optimum coagulant dosages were obtained in the same way as described above, except the dosages were found at 89 per cent color removal. The per cent sludge volume readings at the optimum coagulant dosages were also interpolated from tables to correspond with the values for color removal.

The results shown in Figure 9 and given in Table 3 implied that 32.5 hours of aeration effected the greatest reduction in the optimum coagulant dosage. Also, the amount of sludge produced by coagulation of the sample at 32.5 hours of aeration was low. In Figure 10 and Table 4, the results indicated that the greatest reduction in the optimum coagulant dosage was obtained with 184.5 hours of aeration. The sludge volume produced by coagulation of the sample at 184.5 hours of aeration was low. But, since the results for the optimum coagulant dosage were erratic, it was felt that the best combination of aeration time and optimum coagulant dosage could not be obtained clearly.

It was observed in Figure 9 that the samples tested after 112.5 and 184.5 hours of aeration possessed an increased optimum coagulant dosage. These values were attributed to a possible chemical change in the dye during aeration. Two other causes were thought to have brought about the increase in coagulant dosage, but these were rejected after a small investigation. It was thought that the coagulants had become weak in strength which would have effected an indicated increase in the coagulant dosage. But, by testing the coagulants against fresh coagulant solutions, it was established that this was not the cause. It was also speculated that the pH may have caused the increased coagulant dosage. Therefore, the pH was varied for arbitrary coagulant dosages, as given in Table 4. The outcome was that similar results were obtained at all pH values, and the pH was not responsible for the increase.

Although Beer's Law was not employed in the construction of the standard curve in Figure 7 used for all the color removal analyses in "Experiment 1", it was found that the law could have been applied to the color removal analyses. Beer's Law states that the intensity of transmitted light is affected by a change in the concentration of the absorbing material. Beer's Law gives the following relation:

$dI/I = -K'' dC$

where I = Intensity of light

C = Concentration of absorbing material

K" = Absorption coefficient

The equation is such that a straight line is produced when the logarithm of the fraction of light transmitted, I_o/I, is plotted against the concentration of absorbing material. The light transmitted through distilled water is represented by the symbol I_o, and I is the light transmitted through the sample being tested. The $\log_{10}(I_o/I)$ is called the absorbance. In this investigation, absorbance values were obtained from the light absorption values plotted in Figure 7 by using the following relation: $\log_{10}(I_o/I) = \log_{10}(1/(1-\text{Light Absorption}))$. From this relation and a plot of the resulting absorbance values, it was shown that the color removal analysis used in this investigation conformed to Beer's Law.

B. Experiment 2: Treatment of 803 Brown Dispersed Dye Waste

The object of this experiment was to determine if aerated lagoon treatment would effectively reduce the chemical coagulant demand of 803 Brown Dispersed Dye Waste.

1. Conventional C.O.D. Analysis

The purpose for conducting the conventional C.O.D. analysis was to obtain immediate performance data for the aerated lagoon treatment after 0, 51.0, 76.0, 103.5, 124.5, 172.0, and 244.0 hours of aeration. The procedure used was as described in the Methods and Materials chapter.

The results of the conventional C.O.D. analysis on the raw and aerated samples are shown graphically in Figure 11. The results given in Figure 11 indicated that the C.O.D. of the dye waste mixture varied with the aeration time. Figure 11 also shows that 103.5 hours of aeration produced the greatest reduction in the C.O.D. A set of C.O.D. tests were also performed in supernatant samples from the coagulation tests with ferric chloride and lime, and Purifloc C31 as the coagulants. The coagulation tests were performed on samples of waste taken after 244.0 hours of aeration. The C.O.D. results were 832 p.p.m. and 882 p.p.m. for the supernatants from the ferric chloride and lime, and Purifloc C31 coagulation tests, respectively. Therefore, chemical treatment with ferric chloride and lime, and Purifloc C31 gave 68.4 and 66.5 per cent reduction in C.O.D., respectively. These C.O.D. results indicated that the chemical treatment process was responsible for a large reduction in C.O.D.

It was noted in Figure 11 that the samples taken after 124.5, 172.5, and 244.0 hours of aeration did not follow the decreased C.O.D. trend. This may be attributed to changes in the chemical structures making up the dye waste. Aromatic hydrocarbons are not oxidized to any appreciable extent by the C.O.D. test. The dye waste contained large amounts of aromatic hydrocarbons, which may have been broken down by the aeration process. This breakdown would produce more C.O.D., the result being an increase in the C.O.D. curve as

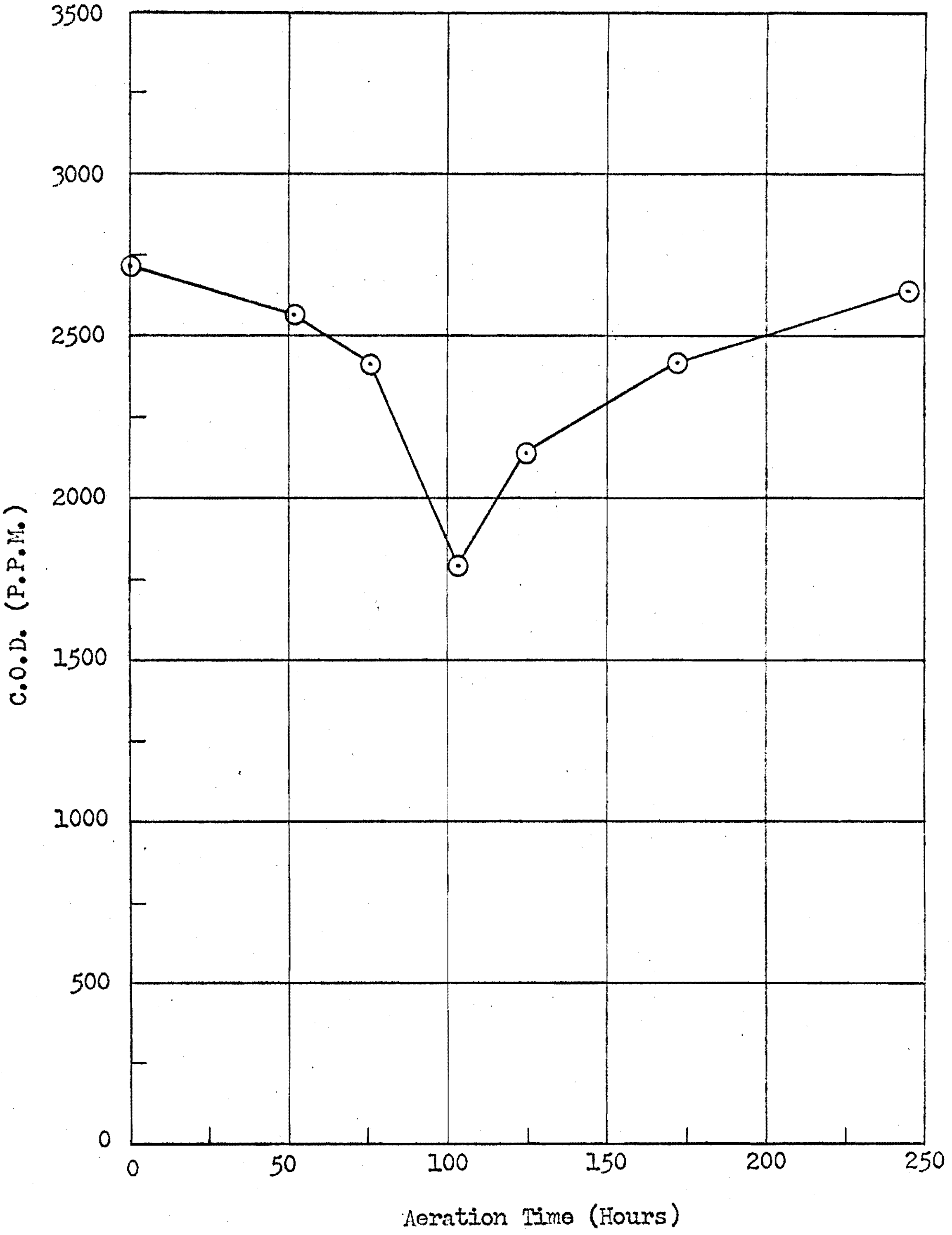


FIGURE 11. THE EFFECT OF AERATION TIME ON THE C.O.D. OF 803 BROWN DISPERSED DYE WASTE USING THE CONVENTIONAL C.O.D. ANALYSIS

shown in Figure 2.

2. Technicon Auto-Analyzer C.O.D. Analysis

The purpose of this experiment was to verify the results showing a reduction in C.O.D. by the conventional C.O.D. analysis by an automated colorimetric method using the Technicon Auto-Analyzer. The procedure used was as described in the Methods and Materials chapter and in "Experiment 1" of this chapter. A Number 7 aperture was used in the colorimeter instead of a Number 5 aperture. As a result, C.O.D. values were obtained for all the standards and unknown samples. The use of the Number 7 aperture helped in verifying that a small aperture was the cause of not obtaining all the results in the Technicon Auto-Analyzer C.O.D. Analysis section in "Experiment 1".

The standard curve used for the C.O.D. analysis is shown in Figure 12. The results of the C.O.D. analysis using the Auto-Analyzer are shown graphically in Figure 13. The results in Figure 13 indicated that the greatest practical effect on the C.O.D. reduction was obtained with an aeration time ranging from 76.0 and 124.5 hours. The aeration time of 103.5 hours, for the greatest C.O.D. reduction obtained in the conventional C.O.D. analysis, fell within the range found above for the auto-Analyzer C.O.D. analysis.

The results of the C.O.D. analysis shown in Figure 13 were considerably different from the results of the conven-

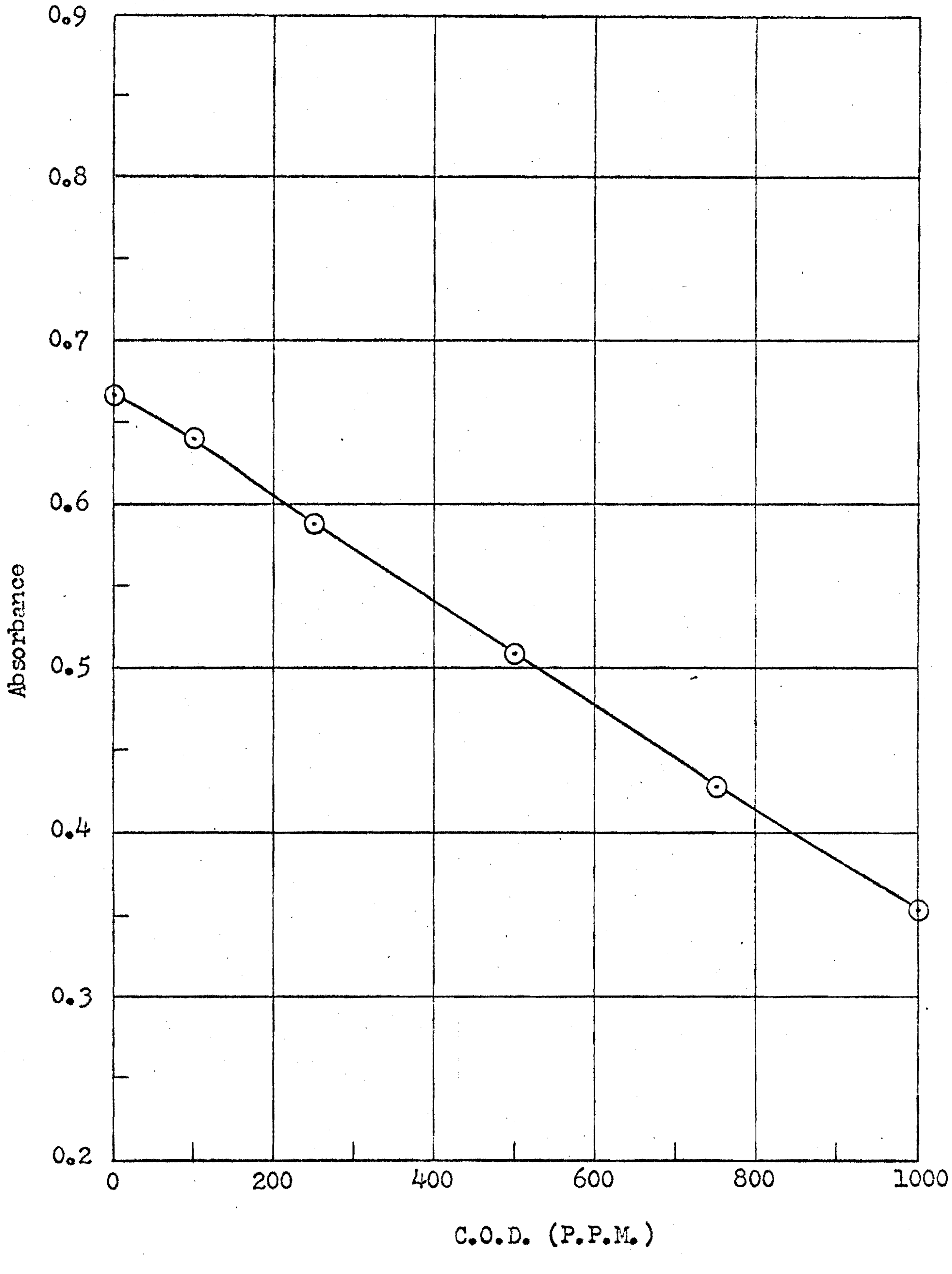


FIGURE 12. STANDARD CURVE USED FOR DETERMINATION OF C.O.D. OF 803 BROWN DISPERSED DYE WASTE BY THE TECHNICON AUTO-ANALYZER C.O.D. ANALYSIS

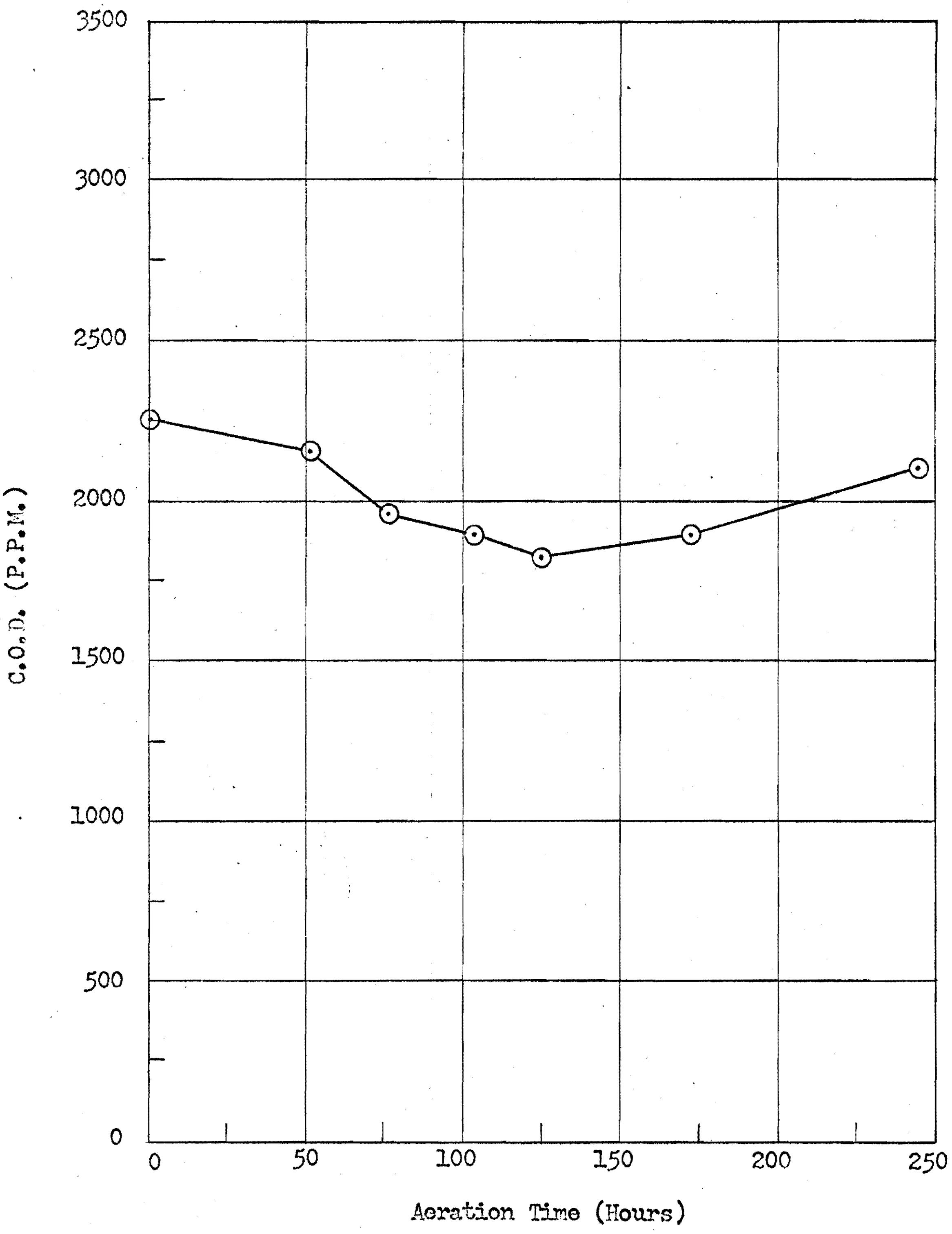


FIGURE 13. THE EFFECT OF AERATION TIME ON THE C.O.D. OF 805 BROWN DISPERSED DYE WASTE USING THE TECHNICON AUTO-ANALYZER C.O.D. ANALYSIS

tional C.O.D. analysis shown in Figure 11. The Auto-Analyzer gave C.O.D. values that were less than those obtained by the conventional method. It was believed that the results should have been different because the procedures of the two tests were different. The main difference in the two analyses was that the samples were only digested in the Auto-Analyzer for a short time, e.g. two to five minutes, while the conventional analysis had a digestion period of two hours. The shorter digestion time would cause lower C.O.D. readings for the Auto-Analyzer, even though the Auto-Analyzer digests at a high temperature.

3. B.O.D. Analysis

The B.O.D. tests were performed as a second performance parameter for the aerated lagoon. The B.O.D. data were obtained for the aerated lagoon after 0, 51.0, 76.0, 103.5, 124.5, and 244.0 hours of aeration. The procedures followed were as described in the Methods and Materials chapter.

The results of the B.O.D. analysis on the raw and aerated samples are given in Table 2, and the five-day B.O.D. values are shown in Figure 14. The B.O.D. results shown in Figure 14 were erratic. Therefore, it was felt that the effect of aeration time on the B.O.D. reduction of the waste was not obtainable from the results shown in Figure 14. The reason for the erratic results is unknown, unless potassium hydroxide spilled out of the cup and wick in the B.O.D. bottles and killed some of the biological seed.

TABLE 2

B.O.D. (P.P.M.) OF 803 BROWN DISPERSED DYE WASTE AFTER VARIOUS PERIODS OF AERATION

Time of Incubation (Days)	Aeration Time (Hours)					
	0	5	76	103.5	124.5	244
1	96	170	175	30	115	115
2	280	290	333	73	198	143
3	393	335	411	176	339	170
4	465	329	510	108	430	193
5	508	329	510	107	476	210
6	530	320	510	141	508	211
7	588	320	510	200	541	235
8	600	• •	• •	200	560	249

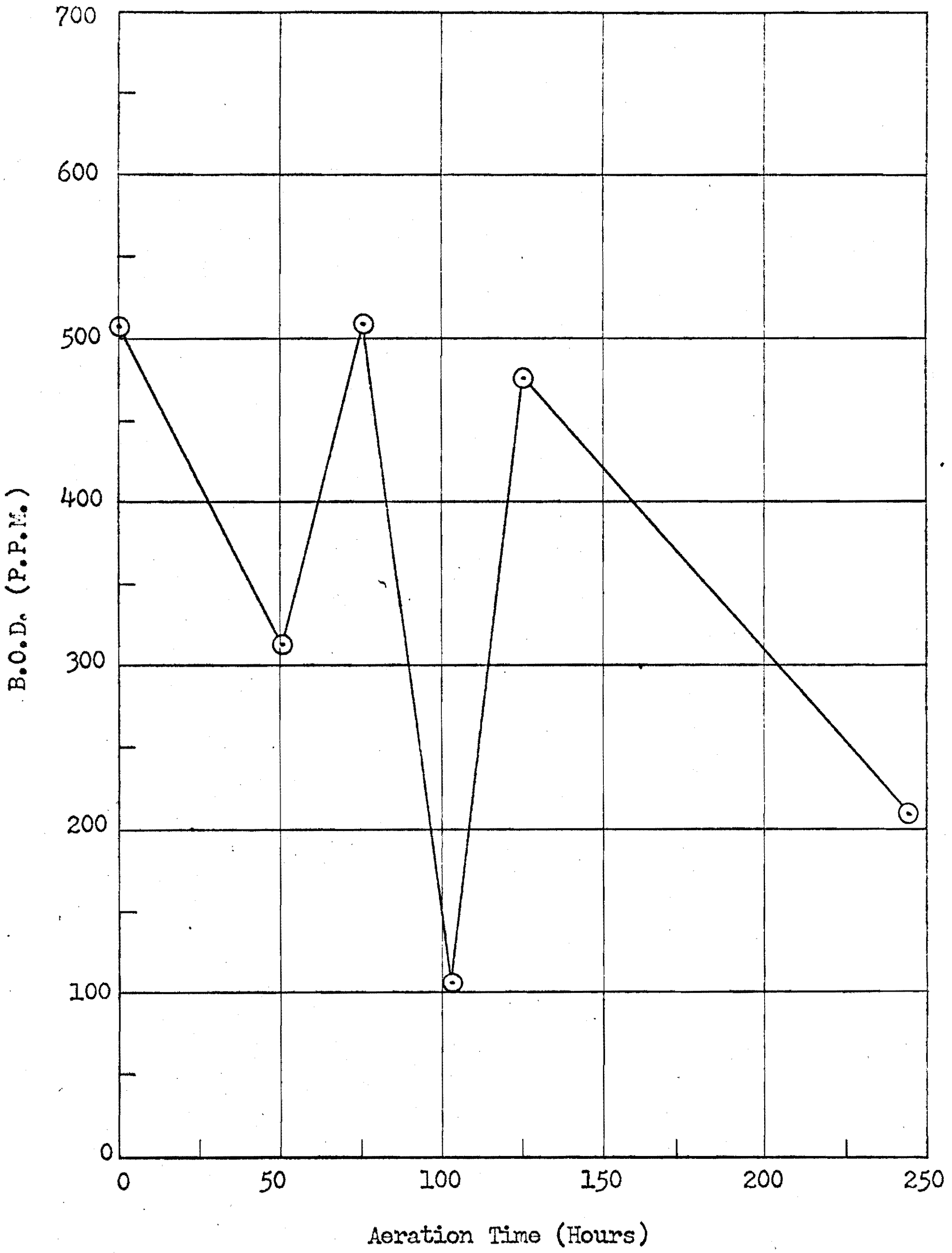


FIGURE 14. THE EFFECT OF AERATION TIME ON THE B.O.D. OF 803 BROWN DISPERSED DYE WASTE

4. Suspended Solids Test

The concentration of suspended solids was determined as a third performance parameter for the aerated lagoon. Suspended solids data were obtained for the aerated lagoon after 0, 51.0, 76.0, 103.5, 124.5, 172.0, and 244.0 hours of aeration. The procedure used was the same as that described in the Methods and Materials chapter.

The suspended solids results are shown in Figure 15. The results of these tests implied that the biological growth was accumulating in the aerated lagoon, but not very rapidly. A decrease in the suspended solids was observed for the sample taken after 172.0 hours of aeration. This decrease was believed to have been caused by an incorrect reading obtained from the analytical balance.

5. Coagulation Test and Color Removal Analysis

The coagulation tests were used to obtain the optimum coagulant dosage for the raw and aerated waste, and were used to evaluate the ability of the aerated lagoon to reduce the coagulant demand of the waste. The color removal for each sample drawn from the aeration tank was determined to evaluate the ability of the aerated lagoon to remove color from the dye waste. The methods followed were as described in the Methods and Materials chapter. The color analysis in this experiment was determined to follow Beer's Law, as described in "Experiment 1".

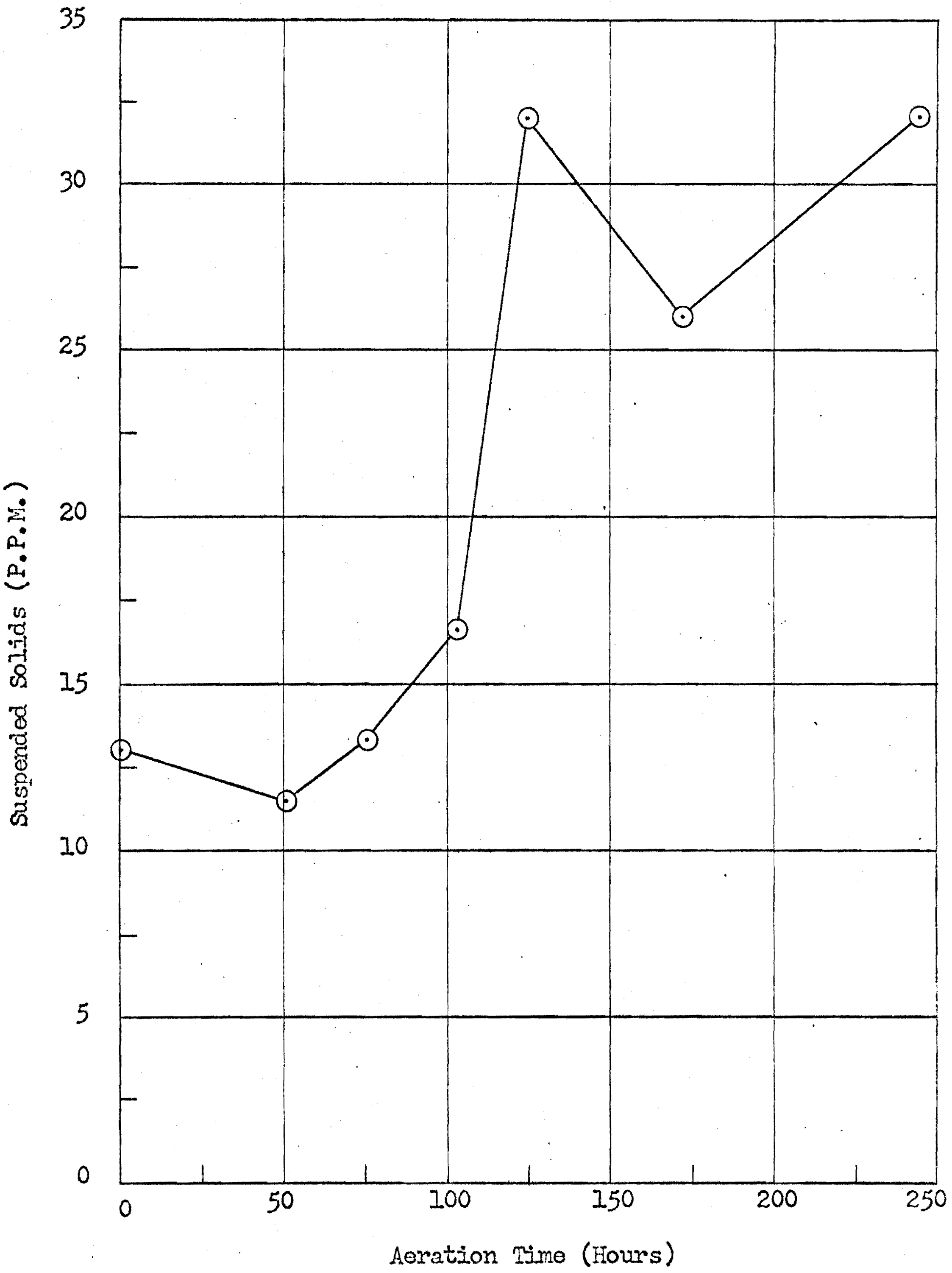


FIGURE 15. THE EFFECT OF AERATION TIME ON THE SUSPENDED SOLIDS OF 803 BROWN DISPERSED DYE WASTE

The standard curve used for the color removal analysis is shown in Figure 16. The performance of the aerated lagoon as to color removal is shown in Figure 17. The results shown in Figure 17, indicated that the aerated lagoon had a very low color removal performance. The lower values for the removal of color at 103.5, 124.5, 172.0 and 244.0 hours of aeration may be attributed to the increased suspended solids concentration that developed during aeration of the mixture. Suspended solids cause less light transmittance through a sample of waste, therefore, a lower color removal was observed. Another possible reason for the lower color removal was a chemical and biological change in the dye waste, which may have produced more color in the waste.

The results of the chemical coagulation tests are presented in Tables 6, 7, 8, and 9 in Appendix B. The effect of aeration time on the optimum coagulant dosage and per cent sludge volume from the coagulation tests is shown in Figures 18 and 19. The coagulant dosage giving 98 per cent color removal was designated as the optimum coagulant dosage listed in Table 6 and plotted in Figure 18. In cases where there were more than one 98 per cent color removal reading, the lowest coagulant dosage was chosen as the optimum. As shown in Table 6, the optimum coagulant dosages were usually interpolated from the data, if the coagulant dosages did not result in exactly 98 per cent color removal. In Figure 19

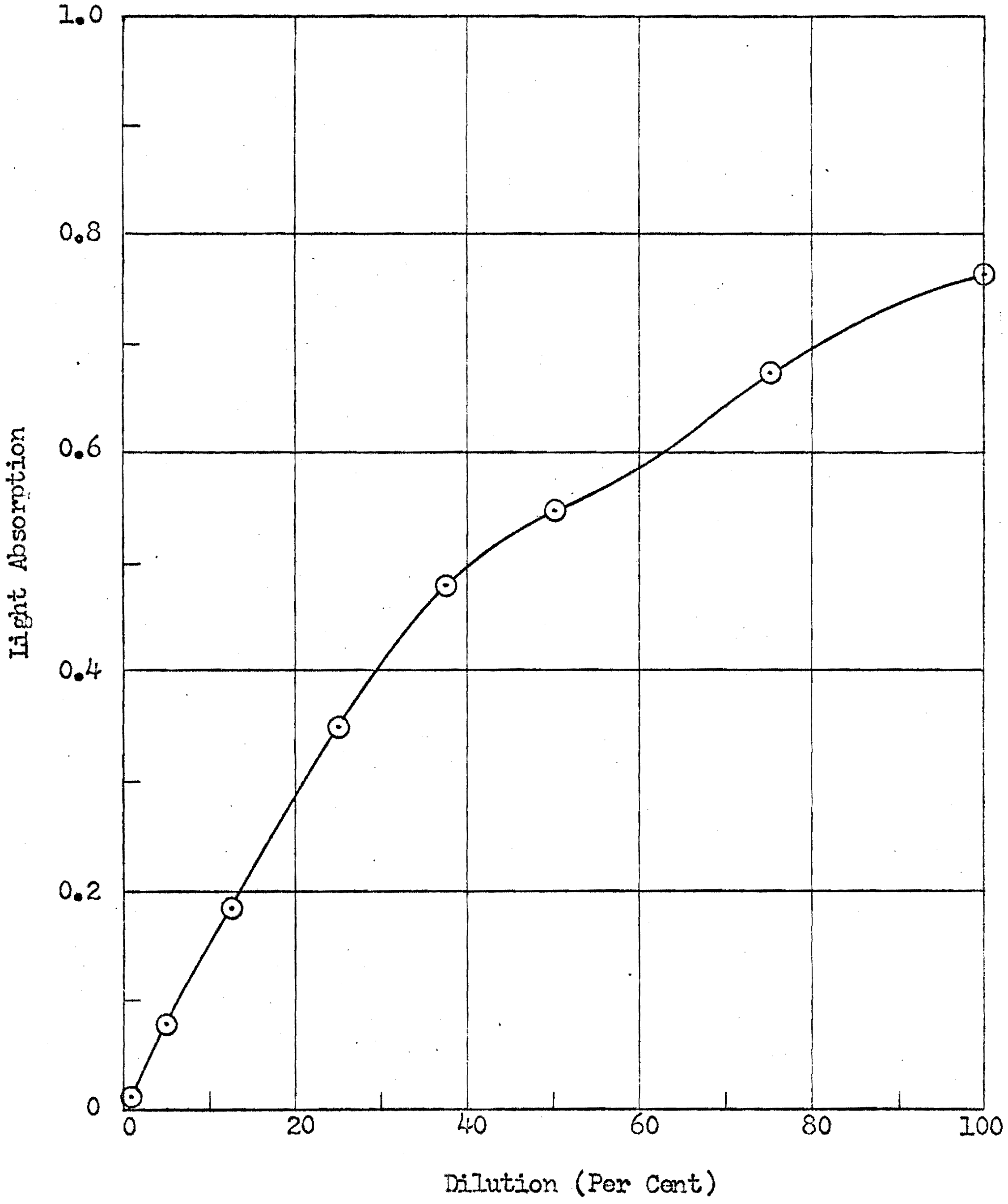
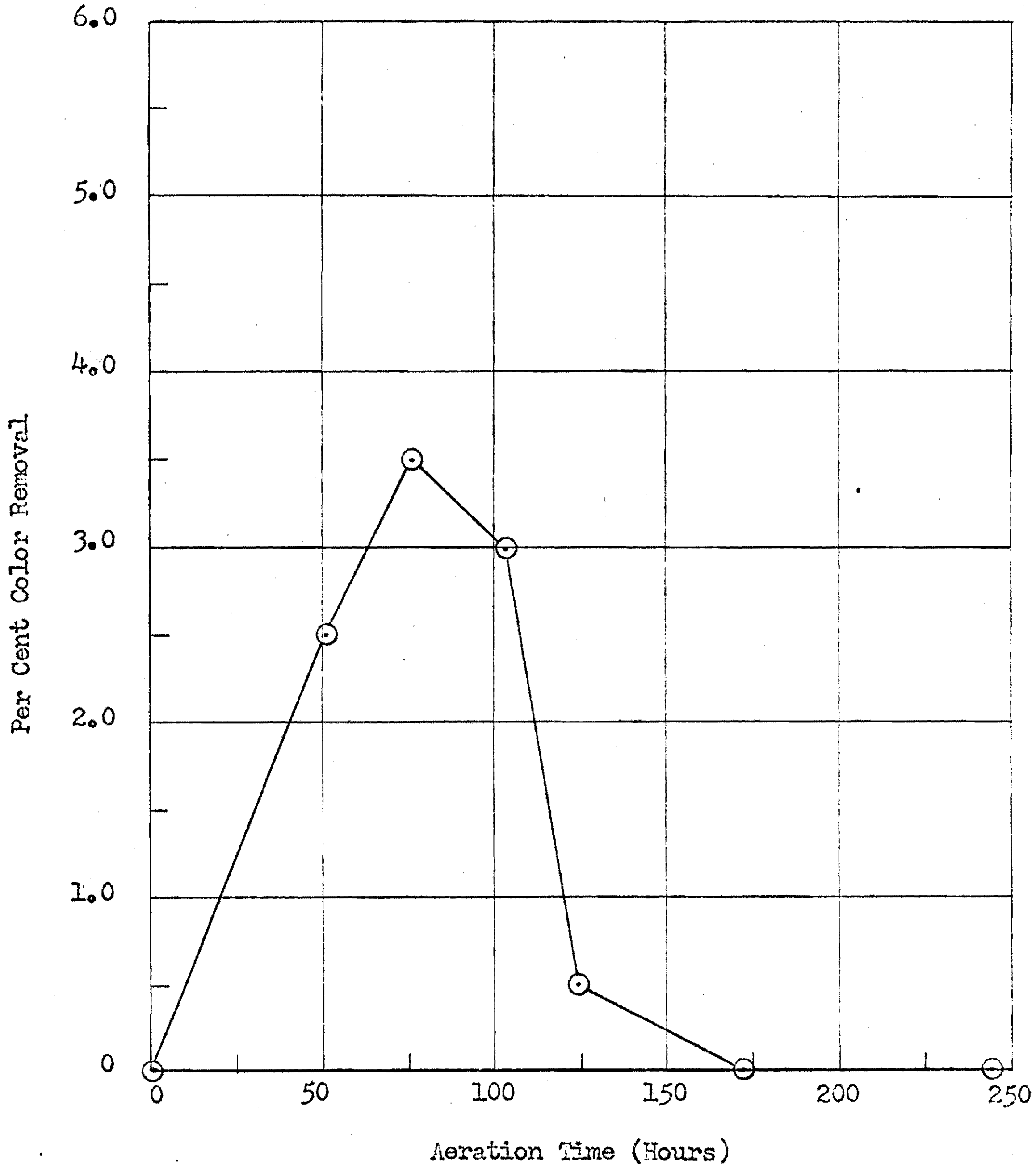


FIGURE 16. STANDARD CURVE USED FOR DETERMINATION OF PER CENT COLOR REMOVAL FOR TREATED SAMPLES OF 803 BROWN DISPERSED DYE WASTE



. FIGURE 17. THE EFFECT OF AERATION TIME ON PER CENT COLOR REMOVAL OF 803 BROWN DISPERSED DYE WASTE

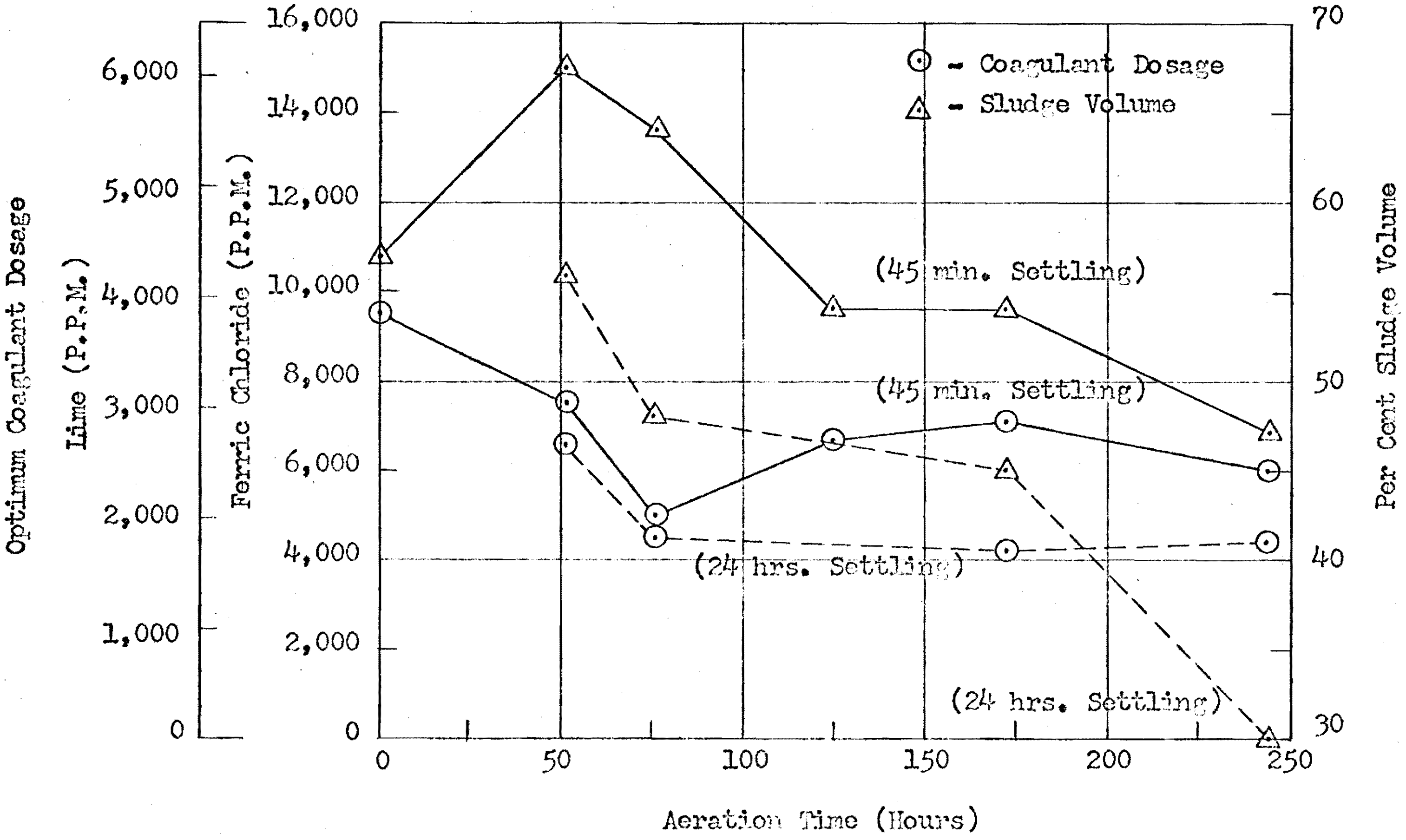


FIGURE 18. THE EFFECT OF AERATION TIME ON THE OPTIMUM COAGULANT DOSAGE AND PER CENT SLUDGE VOLUME OF 803 BROWN DISPERSED DYE WASTE USING FERRIC CHLORIDE AND LIME AS THE COAGULANT COMBINATION

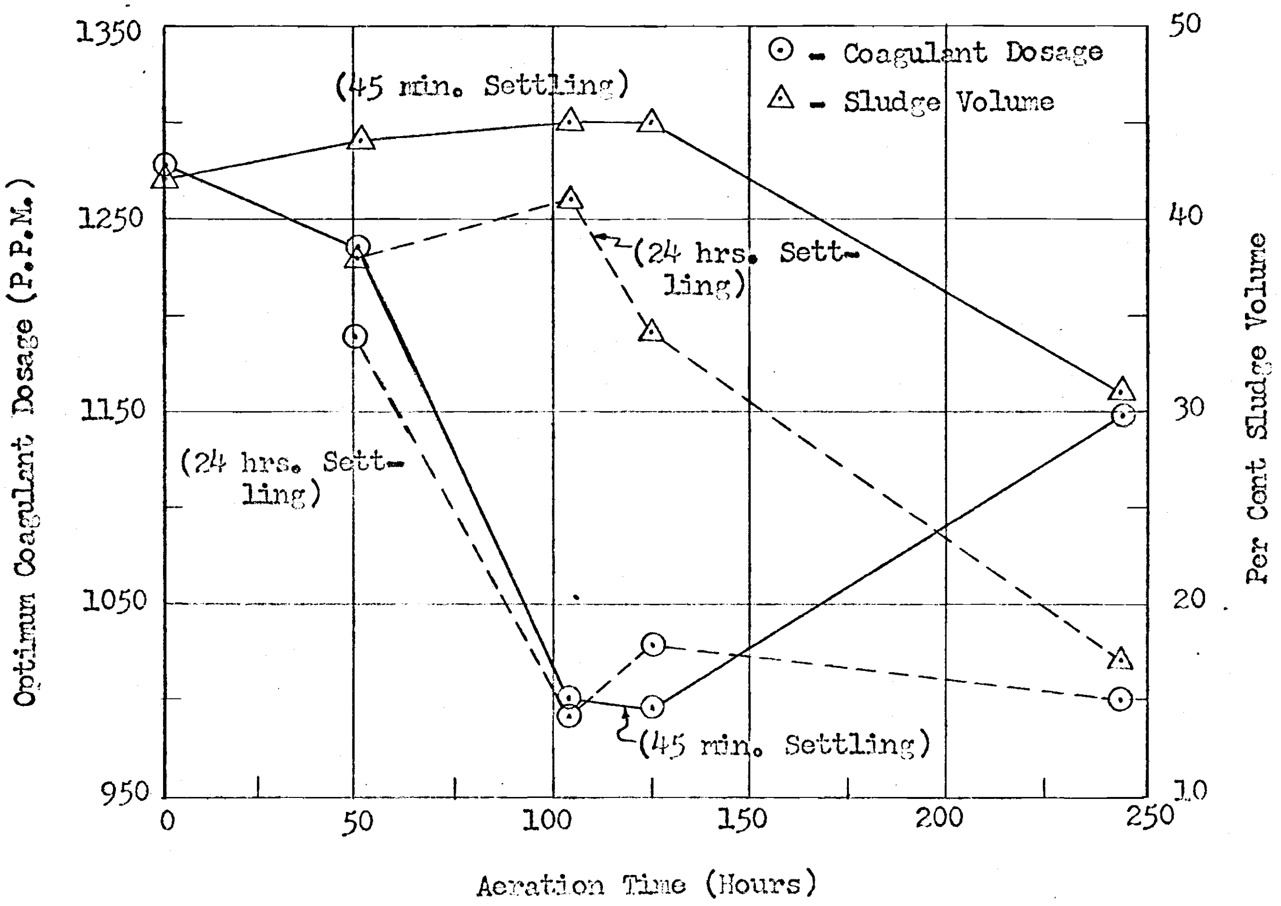


FIGURE 19. THE EFFECT OF AERATION TIME ON THE OPTIMUM COAGULANT DOSAGE AND PER CENT SLUDGE VOLUME OF 803 BROWN DISPERSED DYE WASTE USING PURIFLOC C31 AS THE COAGULANT

and Table 8, the optimum coagulant dosages were obtained in the same way as described above, except the dosages were found at 93 per cent color removal. The per cent sludge volume readings at the optimum coagulant dosages were also interpolated from tables to correspond with the values for color removal.

The results shown in Figure 18 and plotted in Table 6 implied that 76.0 hours of aeration effected the greatest reduction in the optimum coagulant dosage, after settling for 45 minutes. The amount of sludge produced by coagulation of the sample at 76.0 hours of aeration was high. When settling was extended to 24 hours, it was shown that 76.0 hours of aeration continued to have the greatest effect on the optimum coagulant dosage. The sludge volume was reduced by an additional seven to 16 per cent with sedimentation for 24 hours instead of 45 minutes. As illustrated in Table 7, the pH was varied for arbitrary coagulant dosages for the various samples of aerated dye waste mixture. The outcome was that similar results were obtained at all pH values.

In Figure 19 and Table 8, the results indicated that the greatest practical effect on the reduction of the optimum coagulant dosage, after 45 minutes of settling, was obtained at 103.5 hours of aeration. The amount of sludge produced by coagulation of the sample at 103.5 hours of aeration was high. When the settling was extended to 24 hours, it was shown that 103.5 hours of aeration also effected the greatest reduction in the optimum coagulant dosage. The sludge volume percentages shown in Figure 19 were reduced by an additional four to 14 per cent with sedimentation for 24 hours instead of 45 minutes. The pH was varied for an arbitrary coagulant dosage for the various aeration samples, as presented in Table 9. In most cases, the outcome was that similar results were obtained at pH values below 7.0.

It was noted in Figure 18 that the samples taken after 124.5 and 172.0 hours of aeration, and in Figure 19 that the sample taken after 244.0 hours of aeration showed an increase in the optimum coagulant dosage. These high values were attributed to a possible chemical change in the dye waste during aeration.

VI. DISCUSSION OF RESULTS

The primary method of treating dye wastes has been chemical coagulation, but because of the use of dispersing agents in dye bath formulations, the coagulant dosages to effect color removal are high and a large volume of sludge is produced. Since the chemical requirements or chemical coagulant demands of dye wastes have been as high, biological treatment was considered as a possible means of reducing the coagulant demand of the raw wastes. The results of this investigation indicated that biological treatment would reduce the coagulant demand.

There are a few possible mechanisms by which biological treatment or aerated lagoon treatment may reduce the chemical coagulant demand of the dye wastes. The wastes, 701 Black and 803 Brown Dispersed Dye wastes, used in this investigation were relatively high in phosphates. As described in Chapter III, Dyeing Procedure and Waste Composition, monosodium phosphate and Calgon were added to the dye during the dyeing procedure. Calgon is a polymerized metaphosphate compound that was used to sequester metal ions and aid in the dispersion of the dyestuff. The monosodium phosphate was added to control the pH of the dyeing operation. Phosphates produce a chemical coagulant demand by reacting with the coagulants to form phosphate complexes or precipitates, therefore causing a large amount of coagulant to be used before the color of the waste can be removed or reduced. Phosphates may be removed during biological treatment with an aerated lagoon through assimilation by microorganisms, by hydrolysis of polyphosphates to insoluble orthophosphate, and possibly by adsorption on biological flocs formed during aeration. The reduction in phosphates would effect a reduction in chemical coagulant demand. Further reductions in the coagulant demand may be attributed to the assimilation of organic materials in the waste by aerobic organisms in the aerated lagoon.

The mechanisms involved in the removal of color from dye wastes by the coagulation process are not fully understood. Three mechanisms are believed to be responsible for the coagulation of colloidal particles in liquid wastes, e.g. color in dye wastes (11):

1. The coagulants may react with constituents in the dye waste, usually hydroxide ions or organic matter, to form microflocs of hydroxypolymers, which are complex gelatinous precipitates of colloidal size. The color particles probably possess a negative charge, and the microflocs possess positive charges on surface sites. Therefore, aggregation of the microflocs and dispersed color particles of the waste can be induced by electrostatic attraction.
2. The multivalent cations possessed by the coagulants may partially neutralize the charge on the dispersed color particles. The charge may be reduced to the point of inducing coagulation.
3. An increase in the concentration of coagulant ions in solution reduces the electronegative forces of repulsion that stabilize the color dispersions with a negative

> charge. If ion exchange occurs between the negative color particles and the coagulant ions in solution, the dispersion may be destabilized and coagulation induced.

The dark dispersed dyes used in this investigation had large chemical coagulant demands for two reasons. Firstly, since the concentration of phosphates was high, there was a high coagulant demand. Secondly, the color in the dye waste was dispersed by the organic dispersing agent that was added during the dyeing procedure. This agent increased the surface charges of the color particles. Enough coagulant had to be added to precipitate or otherwise overcome the action of the dispersing agent.

In this investigation, aeration was found to produce a substantial reduction in the chemical coagulant demand of the dye wastes. It was shown in the Experimental Data and Results chapter that the aeration time needed to produce a practical reduction in the coagulant demand was dependent upon the type of coagulants and waste used. The coagulant dosages for 90 to 100 per cent removal of color from 701 Black Dispersed Dye waste were found to decrease 31 per cent after 32.5 hours of aeration, when treated with ferric chloride and lime, and settled for 45 minutes. Coagulant dosages for 90 to 100 per cent removal of color from 803 Brown Dispersed Dye waste were found to decrease 21 per cent after 103.5 hours of aeration, and 47 per cent after 76.0 hours of aeration, when treated with Purifloc C31, or with ferric chloride and lime, and settled for 45 minutes. The aerated lagoon also effected a substantial reduction in the C.O.D. and B.O.D. of the waste. The reductions in C.O.D. were 68 per cent for ferric chloride and lime treatment and 67 per cent for Purifloc C31 treatment. The samples tested were 803 Brown Dispersed Dye waste.

Very little color removal was obtained by the aerated lagoon process, which was not consistent with the results reported in the literature. Souther et al (21) and Souther (23) reported color removals from mixed dye wastes by aerated lagoon treatment of between 32 and 76 per cent. Williams et al (27) reported that the color of a mixed dye waste was reduced to a uniform grey. The mixed dye wastes in the investigations above were mostly from the dyeing and finishing of cotton textiles, and probably did not include dispersed dyes, since they are primarily used for the dyeing of synthetic textiles. Therefore, the dye wastes reported in the literature were different from the dispersed dye wastes used in this study. The color removals obtained in this investigation of dispersed dye wastes ranged from 3.5 to 25 per cent, with very little change in the appearance of the waste. The aerated lagoon removed 25 per cent of the color after 66.5 hours of aeration, and 3.5 per cent of the color after 76.0 hours of aeration for 701 Black Dispersed Dye waste and 803 Brown Dispersed Dye waste, respectively. The samples used for the color analysis were settled for 45 minutes before analyzing. The low color removals experienced from the wastes used in this study were probably caused by the biological refractory properties of the dispersed dyestuff.

The coagulation tests produced large color removal percentages in this investigation. The percentages obtained by treatment with ferric chloride and lime, and Purifloc C31 were usually high, e.g. between 90 and 100 per cent color removal. Coagulation with ferrous sulfate and lime produced color removal percentages ranging from 87 to 92 per cent color removal. The chemical coagulation with ferric chloride and lime, and Purifloc C31 effected a substantial reduction in C.O.D., as shown by two C.O.D. tests that were conducted on supernatant samples of 803 Brown Dispersed Dye waste. The reductions in C.O.D. were 68 per cent for ferric chloride and lime, and 67 per cent for Purifloc C31. The reduction in C.O.D. by chemical coagulation may be attributed to adsorption of organic material on to the flocs formed during chemical treatment, and possibly precipitation of organic dispersing agents.

Chrisco et al (2) and Porges et al (13) have reported sludge volume to supernatant volume ratios between 1:20 and 1:3, which resulted in sludge volume percentages between five and 25 per cent, respectively. These results were for a variety of dye wastes and coagulants, and the settling time was 24 hours. The dye wastes in the investigations above were mostly from the dyeing of cotton textiles, and some of them were dyes that are out of date today. The dye wastes did not include any dispersed dyes. Therefore, the dye wastes reported in the literature were different from the dispersed dye wastes used in this study. The sludge volume percentages, obtained in this investigation after 24 hours settling, were between 41 and 48 per cent, and were somewhat higher than those reported above. This increase in sludge volume may be due to the greater coagulant demand of the waste. A settling time of 45 minutes gave even greater increases in sludge volume. At 45 minutes settling, the sludge volumes percentages were between 45 and 64 per cent.

The treatment of dye wastes is certainly an area in which further research is needed. This investigation dealt with the effects of aerated lagoon treatment on the chemical demand of dark dispersed dye wastes. This study was considered to be of value because it indicated that aerated lagoon treatment would produce substantial reductions in the chemical coagulant demand of the dye wastes, which in turn produced large reductions in color. Also, significant reductions in the B.O.D. and C.O.D. were produced by the aerated lagoon. It is not certain whether the process combination investigated in this thesis will be economical enough to be used as a treatment process in the textile industry. It would be advantageous, therefore, to study the cost aspects of a treatment process using an aerated lagoon and chemical coagulation process and compare this cost with the cost of other treatment processes.

VII. CONCLUSIONS

The results of this investigation led to the following conclusions:

1. Biological treatment using an aerated lagoon produced substantial reductions in the chemical coagulant dosages of dark dispersed dye wastes. The aeration time needed to produce a reduction in the coagulant dosage was dependent upon the type of coagulants and the character of the waste. The coagulant dosages for 90 to 100 per cent removal of color from 701 Black Dispersed Dye waste for 90 to 100 per cent removal of color were found to decrease 31 per cent after 32.5 hours of aeration, when treated with ferric chloride and lime, and settled for 45 minutes. Coagulant dosages for 90 to 100 per cent removal of color from 803 Brown Dispersed Dye waste were found to decrease 21 per cent after 103.5 hours of aeration, and 47 per cent after 76.0 hours of aeration, when treated with Purifloc C31, or with ferric chloride and lime, and settled for 45 minutes.
2. Sludge accumulation from the coagulation of dispersed dye waste was high. The per cent sludge volume produced by treatment of 701 Black Dispersed Dye waste with the optimum coagulant dosage was found to be 55 per cent after 32.5 hours of aeration, when treated with ferric chloride and lime, and settled for 45 minutes. The sludge volume

percentages produced by treatment of 803 Brown Dispersed Dye waste with the optimum dosages were found to be 45 per cent after 103.5 hours of aeration, and 64 per cent after 76.0 hours of aeration, when treated with Purifloc C31, and ferric chloride and lime, respectively, and settled for 45 minutes.

3. Settling was complete in 45 minutes, but the sludge continued to compact for 24 hours.
4. Addition of the coagulants to biologically treated dye waste produced substantial reductions in the chemical oxygen demand. The reductions in C.O.D. were 68 per cent for ferric chloride and lime treatment, and 67 per cent for Purifloc C31 treatment. The samples tested were 803 Brown Dispersed Dye waste.
5. Aerated lagoon biological treatment produced substantial reductions in the C.O.D. of dye wastes. The C.O.D. of 701 Black Dispersed Dye waste was found to decrease five and 23 per cent after 32.5 and 66.5 hours of aeration, respectively. The C.O.D. of 803 Brown Dispersed Dye waste was found to decrease 11 and 34 per cent after 76.0 and 103.5 hours of aeration, respectively.
6. Very little color removal was obtained by the aerated lagoon process. The lagoon removed 25 per cent of the color after 66.5 hours of aeration, and 3.5 per cent of the color after 76.0 hours of aeration for 701 Black

and 803 Brown Dispersed Dye wastes, respectively. The samples used for the color analysis were settled for 45 minutes.

VIII. SUMMARY

The object of this investigation was to determine the effect of aerated lagoon biological treatment on the chemical coagulant demand required to effect removal of color from two dark dispersed dye wastes. Chemical coagulation was desirable because it effectively reduced the color and C.O.D. of the dispersed dye waste. Aerated lagoon biological treatment was thought to be a possible means of reducing the chemical coagulant demand of dye wastes.

A simulated aerated lagoon was constructed utilizing dark dispersed dye wastes, 701 Black or 803 Brown Dispersed Dye waste, and a biological seed. The lagoon was operated on a batch basis. Samples were drawn from the aerated lagoon after various periods of aeration, and treated with coagulants and coagulant combinations to produce high reductions in color. Ferric chloride and lime, ferrous sulfate and lime, and Purifloc C31 were the coagulants used in the coagulation tests. The optimum coagulant dosage, color removal, volume of sludge, and pH were measured for each sample after specified settling periods. The C.O.D. of the supernatant after coagulation of a sample of 803 Brown Dispersed Dye waste with ferric chloride and lime and Purifloc C31 was determined. The effect of aeration time on color removal, B.O.D., C.O.D., optimum coagulant dosage, and suspended solids was also determined for the dispersed dye wastes.

The chemical coagulant demand was substantially reduced by aerated lagoon biological treatment. Coagulant dosages for 90 to

100 per cent removal of color from 701 Black Dispersed Dye waste were found to decrease 31 per cent after 32.5 hours of aeration, when treated with ferric chloride and lime and settled for 45 minutes. Coagulation dosages for 90 to 100 per cent removal of color from 803 Brown Dispersed Dye waste were found to decrease 21 per cent after 103.5 hours of aeration, and 47 per cent after 76.0 hours of aeration, when treated with Purifloc C31, or with ferric chloride and lime, and settled for 45 minutes. A large sludge volume was produced from the coagulation of dispersed dye wastes. The per cent sludge volume produced by treatment of 701 Black Dispersed Dye waste with the optimum coagulant dosage was found to be 55 per cent after 32.5 hours of aeration, when treated with ferric chloride and lime, and settled for 45 minutes. The sludge volume percentages produced by treatment of 803 Dispersed Dye waste for the optimum dosages were found to be 45 per cent after 103.5 hours of aeration, and 64 per cent after 76.0 hours of aeration, when treated with Purifloc C31 or with ferric chloride and lime, and settled for 45 minutes. Settling was complete in 45 minutes, but the sludge continued to compact for 24 hours.

Reductions in C.O.D. were observed when coagulants were used to treat the biologically treated dye waste. The C.O.D. reductions were 68 per cent for ferric chloride and lime treatment, and 67 per cent for Purifloc C31 treatment. The samples tested were composed of 803 Brown Dispersed Dye waste.

Significant reductions in the C.O.D. of the dye wastes were produced by the aerated lagoon. The C.O.D. of 701 Black Dispersed

Dye waste was found to decrease five and 23 per cent after 32.5 and 66.5 hours of aeration, respectively. The C.O.D. of 803 Brown Dispersed Dye waste was found to decrease 11 and 34 per cent after 76.0 and 103.5 hours of aeration, respectively. Very little color removal was obtained by the aerated lagoon process. The lagoon removed 25 per cent of the color after 66.5 hours of aeration for the 701 Black Dispersed Dye waste. The lagoon only removed 3.5 per cent of the color after 76.0 hours of aeration for the 803 Brown Dispersed Dye waste.

IX. ACKNOWLEDGMENTS

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APPENDIX A

Illustrated Examples of the Original and Alternate Methods for Color Removal Analysis

A. Example 1: Original Method for Color Removal Analysis

1. This example was performed using a sample of supernatant from the treatment of 701 Black Dispersed Dye waste with the coagulation test and ferric chloride and lime as the coagulants.
2. The Spectronic 20 was adjusted with a distilled water blank so that a distilled water spectrum curve was obtained as shown in Figure 20.
3. With the Spectronic 20 adjusted with the distilled water blank, the sample was put into the instrument. A spectrum curve was then obtained for the sample by varying the wave length of light. The curve is shown in Figure 20.
4. The maximum absorption of light was found for the sample by measuring the maximum vertical distance between the sample spectrum curve and the distilled water spectrum curve, and dividing this distance by the total vertical distance under the distilled water spectrum curve. The vertical distance between the curves is shown in Figure 20.

   Vertical Distance Between Curves = 57.0 units

   Total Distance Under Distilled Water Curve = 89.5 units

   Maximum Light Absorption = 57.0/89.5 = 0.637
5. The maximum light absorption was entered into the standard curve shown in Figure 7. From Figure 7, a per cent dilution value was obtained for the maximum light absorption value above. This dilution value was subtracted from 100

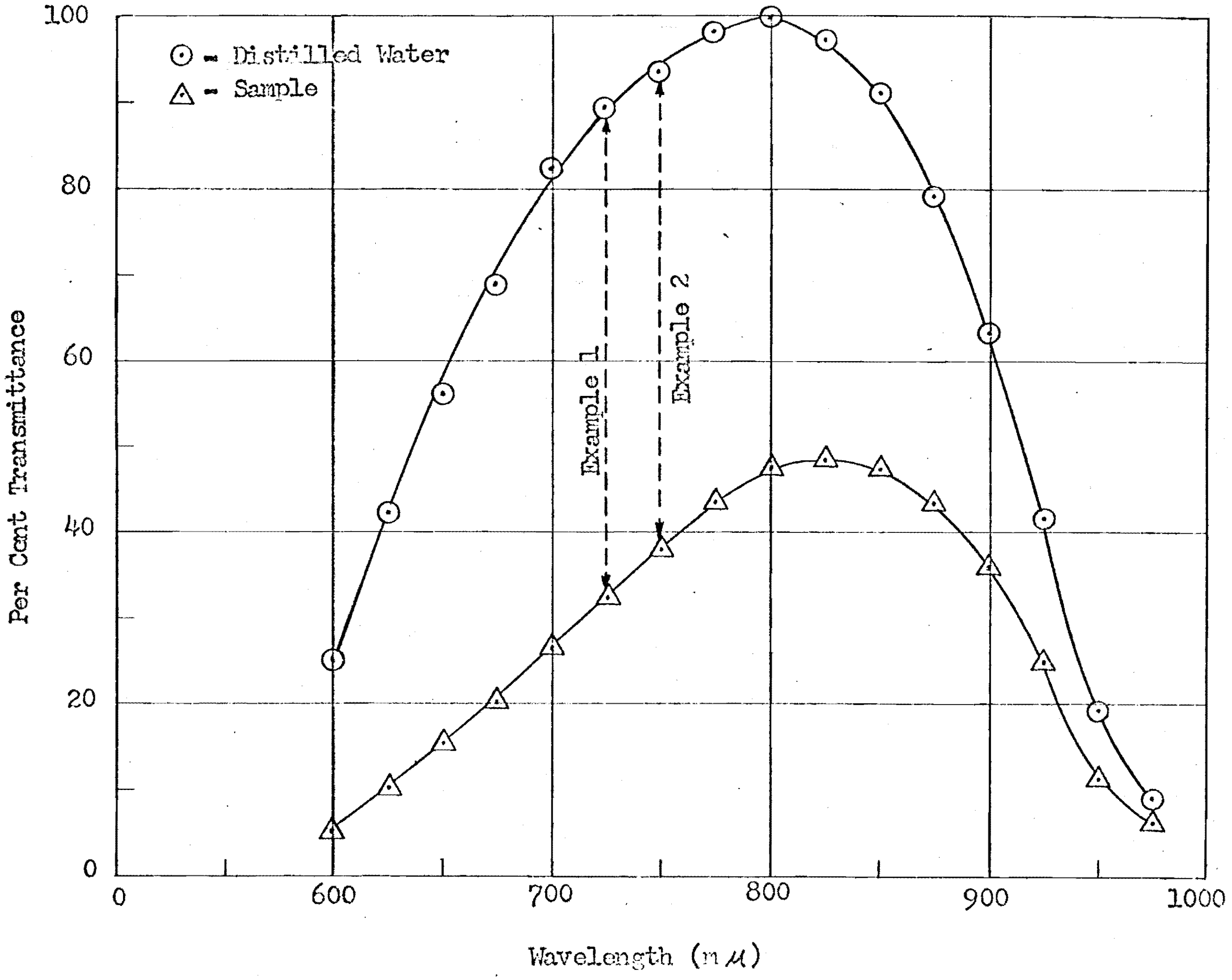

FIGURE 20. SPECTRUM CURVES FOR COLOR REMOVAL ANALYSIS

per cent to obtain the per cent color removal.

Per Cent Dilution = 40.0 per cent

Per Cent Color Removal = 100.0 - 40.0 = 60.0 per cent

B. Example 2: Alternate Method for Color Removal Analysis

1. This example was performed using a sample of supernatant from the treatment of 701 Black Dispersed Dye waste with the coagulation test and ferric chloride and lime as the coagulants. The spectrum curves for the distilled water and the sample are shown in Figure 20.
2. The maximum light absorption was found for the sample by measuring the vertical distance between the sample spectrum curve and the distilled water spectrum curve shown in Figure 20, and dividing this distance by the total vertical distance under the distilled water spectrum curve. These vertical distances were measured at a wavelength of 750 m . The vertical distance between the curves is shown in Figure 20.

   Vertical Distance Between Curves = 55.8 units

   Total Distance Under Distilled Water Curve = 93.8 units

   Maximum Light Absorption = 55.8/93.8 = 0.595
3. The maximum light absorption was then entered into the standard curve shown in Figure 21. This standard curve was obtained by the same procedure described for the alternate method standard curve in the Methods and Materials chapter. A per cent dilution was obtained for the maximum light

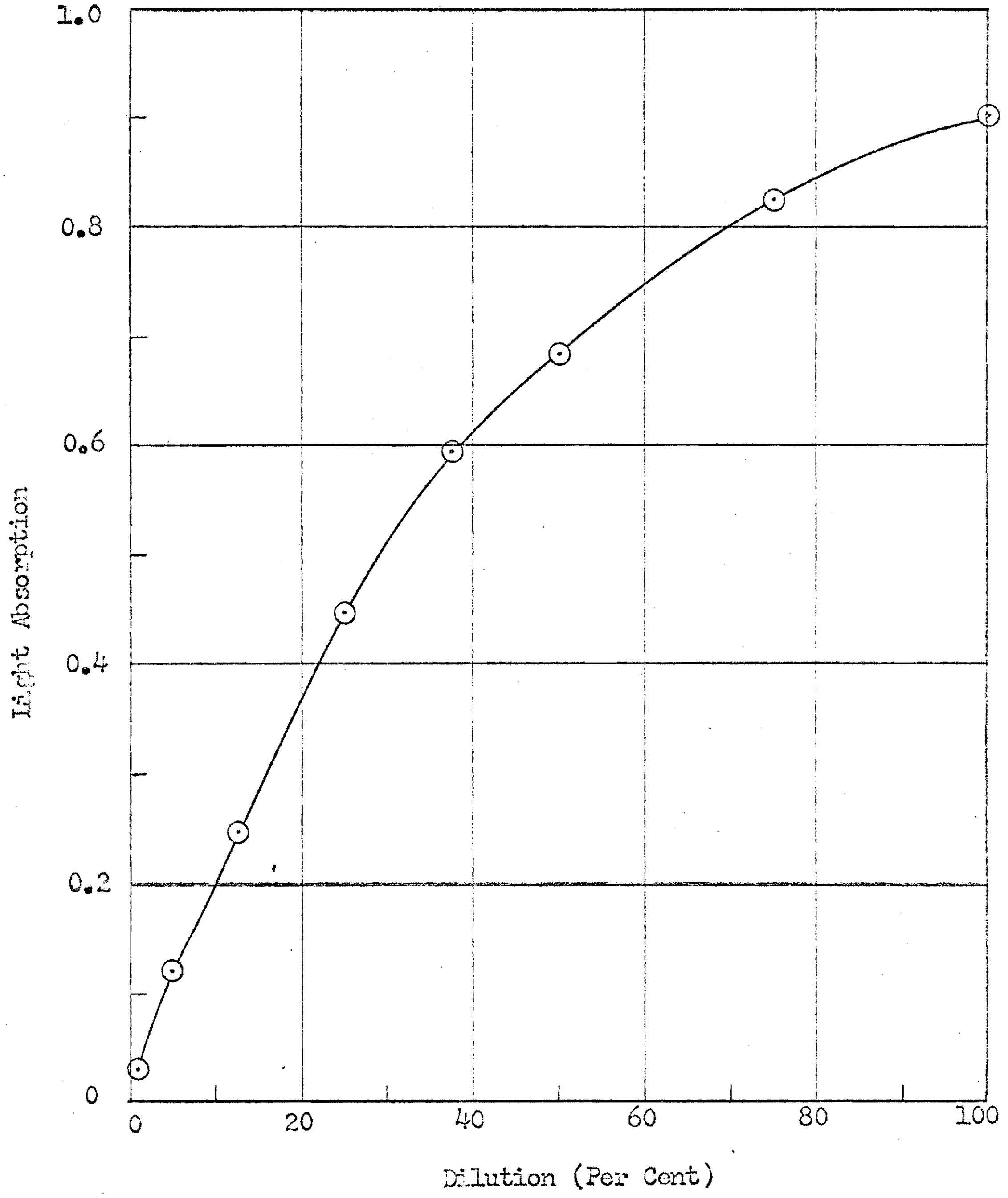

FIGURE 21. STANDARD CURVE FOR ALTERNATE COLOR REMOVAL ANALYSIS

absorption value above. This dilution value was subtracted from 100 per cent to obtain the per cent color removal.

Per Cent Dilution = 38.2 per cent

Per Cent Color Removal = 100.0 - 38.2 = 61.8 per cent

APPENDIX B

Tables for the Effect of Aeration Time on Chemical Treatment of Two Dispersed Dye Wastes

TABLE 3

THE EFFECT OF AERATION TIME ON CHEMICAL TREATMENT OF 701 BLACK DISPERSED DYE WASTE

Sample Aeration Time (Hours)	Dosage (P.P.M.)		Settling Time (Min.)	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum Coagulant Dosage (P.P.M.) at 95% Color Removal	
	$FeCl_3$	$Ca(OH)_2$						$FeCl_3$	$Ca(OH)_2$
0	2,160	888	45	6.20	6.00	72.1	46	4,680	1,920
0	3,600	1,480	45	6.20	6.00	92.8	81		
0	5,400	2,220	45	6.20	6.15	95.7	55		
0	7,200	2,960	45	6.20	6.20	97.0	48		
0	11,700	4,810	45	6.20	6.20	99.6	48		
0	12,600	5,180	45	6.20	6.20	99.8	48		
32.5	540	222	45	6.45	6.10	1.2	14	3,240	1,330
32.5	1,440	592	45	6.45	4.90	54.0	39		
32.5	2,160	888	45	6.45	5.90	58.5	40		
32.5	2,880	1,184	45	6.45	5.65	93.8	56		
32.5	6,300	2,590	45	6.45	3.80	98.4	46		
32.5	10,800	4,440	45	6.45	2.40	77.3	40		
49.5	1,800	740	45	6.55	5.70	72.9	31	3,510	1,440
49.5	3,600	1,480	45	6.55	4.90	96.4	69		
49.5	7,200	2,960	45	6.55	2.90	13.5	41		
49.5	10,800	4,440	45	6.55	3.10	64.3	39		
66.5	900	370	45	6.65	5.80	47.5	22	3,240	1,330
66.5	2,700	1,110	45	6.65	3.80	90.2	40		
66.5	3,600	1,480	45	6.65	2.70	96.6	52		
66.5	4,500	1,850	45	6.65	2.80	87.5	44		
66.5	6,300	2,590	45	6.65	2.80	0	35		
66.5	9,000	3,700	45	6.65	2.60	4.0	28		

TABLE 3 (Continued)

Sample Aeration Time (Hours)	Dosage (P.P.M.)		Settling Time (Min.)	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum Coagulant Dosage (P.P.M.) at 95% Color Removal	
	$FeCl_3$	$Ca(OH)_2$						$FeCl_3$	$Ca(OH)_2$
0*	3,600	1,480	45	6.45	4.90	81.7	69	• •	• •
112.5	1,800	740	45	6.80	5.10	76.6	27	3,550	1,460
112.5	2,700	1,110	45	6.80	4.90	67.3	30		
112.5	3,600	1,480	45	6.80	4.40	96.4	56		
112.5	4,500	1,850	45	6.80	4.40	96.4	53		
112.5	6,300	2,590	45	6.80	2.90	52.0	37		
112.5	9,000	3,700	45	6.80	2.80	77.6	33		
184.5	900	370	45	6.90	6.00	56.0	15	4,680	1,920
184.5	1,800	740	45	6.90	5.90	72.8	21		
184.5	2,700	1,110	45	6.90	5.70	78.4	36		
184.5	3,600	1,480	45	6.90	5.40	91.0	54		
184.5	4,500	1,850	45	6.90	5.60	96.2	55		
184.5	6,300	2,590	45	6.90	5.00	98.3	51		
184.5	8,100	3,330	45	6.90	6.60	99.1	59		

*Note: Raw sample stored for 66.5 hours.

TABLE 4

THE EFFECT OF AERATION TIME ON CHEMICAL TREATMENT OF 701 BLACK DISPERSED DYE WASTE

Sample Aeration Time (Hours)	Coagulant Dosage (P.P.M.)		pH Adjusting Reagent	pH	Settling Time	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum pH Range
	$FeCl_3$	$Ca(OH)_2$								
184.5	6,300	2,590	H_2SO_4	4.40	45 min.	6.70	4.30	98.7	43	4.40 to 8.70
184.5	6,300	2,590	H_2SO_4	5.40	45 min.	6.70	5.50	98.1	43	
184.5	6,300	2,590	. .	6.10	45 min.	6.70	6.10	99.5	39	
184.5	6,300	2,590	Lime	6.40	45 min.	6.70	6.30	99.5	40	
184.5	6,300	2,590	Lime	7.30	45 min.	6.70	7.20	99.5	38	
184.5	6,300	2,590	Lime	8.70	45 min.	6.70	8.50	98.6	38	
184.5	6,300	2,590	H_2SO_4	4.40	24 hrs.	6.70	4.40	98.7	36	4.40 to 8.70
184.5	6,300	2,590	H_2SO_4	5.40	24 hrs.	6.70	5.80	98.3	36	
184.5	6,300	2,590	. .	6.10	24 hrs.	6.70	6.30	98.8	31	
184.5	6,300	2,590	Lime	6.40	24 hrs.	6.70	6.60	98.4	28	
184.5	6,300	2,590	Lime	7.30	24 hrs.	6.70	7.20	98.8	26	
184.5	6,300	2,590	Lime	8.70	24 hrs.	6.70	8.10	98.7	31	

TABLE 5

THE EFFECT OF AERATION TIME ON CHEMICAL TREATMENT OF 701 BLACK DISPERSED DYE WASTE

Sample Aeration Time (Hours)	Dosage (P.P.M.)		Settling Time (Min.)	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum Coagulant Dosage (P.P.M.) at 89% Color Removal	
	$FeSO_4$	$Ca(OH)_2$						$FeSO_4$	$Ca(OH)_2$
0	2,780	740	45	6.20	5.50	68.7	15		
0	11,120	2,960	45	6.20	6.10	67.7	19	15,830	4,210
0	16,680	4,440	45	6.20	6.20	92.0	32		
0	22,240	5,920	45	6.20	6.30	91.5	32		
0	33,360	8,880	45	6.20	6.00	81.8	38		
0	38,920	10,360	45	6.20	5.90	81.3	39		
32.5	1,390	370	45	6.45	5.75	80.2	20		
32.5	2,780	740	45	6.45	5.70	80.2	17		
32.5	5,560	1,460	45	6.45	5.70	89.3	19		
32.5	11,120	2,960	45	6.45	6.10	92.6	21	11,120	2,960
32.5	19,460	5,180	45	6.45	6.20	90.3	34		
49.5	5,560	1,480	45	6.55	5.65	85.2	18		
49.5	11,120	2,960	45	6.55	6.10	86.0	19		
49.5	19,460	5,180	45	6.55	6.50	88.8	23	19,460	5,180
66.5	8,340	2,220	45	6.65	5.80	88.9	25	8,340	2,220
66.5	11,120	2,960	45	6.65	6.00	90.5	24		
66.5	13,900	3,700	45	6.65	6.40	92.1	30		
66.5	16,680	4,440	45	6.65	6.30	92.7	32		
66.5	22,240	5,920	45	6.65	6.30	89.5	38		

TABLE 5 (Continued)

Sample Aeration Time (Hours)	Dosage (P.P.M.)		Settling Time (Min.)	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum Coagulant Dosage (P.P.M.) at 89% Color Removal	
	$FeSO_4$	$Ca(OH)_2$						$FeSO_4$	$Ca(OH)_2$
112.5	8,340	2,220	45	6.80	6.00	88.7	22	8,900	2,365
112.5	11,120	2,960	45	6.80	6.30	90.5	26		
112.5	13,900	3,700	45	6.80	6.50	92.2	32		
112.5	16,680	4,440	45	6.80	6.30	90.5	34		
112.5	19,460	5,180	45	6.80	6.30	90.5	34		
112.5	22,240	5,920	45	6.80	6.40	87.9	42		
184.5	5,560	1,480	45	6.90	6.80	90.5	56	5,560	1,480
184.5	8,340	2,220	45	6.90	6.80	97.8	70		
184.5	11,120	2,960	45	6.90	6.70	97.8	71		
184.5	13,900	3,700	45	6.90	6.80	97.8	77		
184.5	16,600	4,440	45	6.90	6.60	97.8	74		

TABLE 6

THE EFFECT OF AERATION TIME ON CHEMICAL TREATMENT OF 803 BROWN DISPERSED DYE WASTE

Sample Aeration Time (Hours)	Dosage (P.P.M.)		Settling Time	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum Coagulant Dosage (P.P.M.) at 98% Color Removal	
	$FeCl_3$	$Ca(OH)_2$						$FeCl_3$	$Ca(OH)_2$
0	7,500	3,100	45 min.	6.35	. .	. .	. .	9,500	3,927
0	9,000	3,720	45 min.	6.35	5.70	97.8	57		
0	10,500	4,340	45 min.	6.35	5.90	98.4	57		
0	12,000	4,960	45 min.	6.35	6.50	99.0	55		
0	13,500	5,580	45 min.	6.35	6.50	99.4	55		
0	15,000	6,200	45 min.	6.35	6.80	98.9	53		
0	7,500	3,100	24 hrs.	6.35	. .	. .	. .	. .	. .
0	9,000	3,720	24 hrs.	6.35	6.20	98.7	42		
0	10,500	4,340	24 hrs.	6.35	6.50	98.6	43		
0	12,000	4,960	24 hrs.	6.35	6.70	97.5	45		
0	13,500	5,580	24 hrs.	6.35	6.90	97.8	45		
0	15,000	6,200	24 hrs.	6.35	6.70	99.0	41		
51.0	3,000	1,240	45 min.	6.35	5.80	95.2	50	7,500	3,100
51.0	6,000	2,480	45 min.	6.35	5.60	97.6	78		
51.0	9,000	3,720	45 min.	6.35	4.60	98.4	57		
51.0	12,000	4,960	45 min.	6.35	4.40	98.6	53		
51.0	15,000	6,200	45 min.	6.35	3.20	0	50		
51.0	18,000	7,440	45 min.	6.35	3.20	0	48		
51.0	3,000	1,240	24 hrs.	6.35	5.90	96.5	45		
51.0	6,000	2,480	24 hrs.	6.35	5.80	97.5	59		

TABLE 6 (Continued)

Sample Aeration Time (Hours)	Dosage (P.P.M.)		Settling Time	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum Coagulant Dosage (P.P.M.) at 98% Color Removal	
	$FeCl_3$	$Ca(OH)_2$						$FeCl_3$	$Ca(OH)_2$
51.0	9,000	3,720	24 hrs.	6.35	4.80	100.0	43	6,600	2,728
51.0	12,000	4,960	24 hrs.	6.35	4.10	99.0	41		
51.0	15,000	6,200	24 hrs.	6.35	3.10	76.1	40		
51.0	18,000	7,440	24 hrs.	6.35	2.90	0	37		
76.0	1,500	620	45 min.	6.50	6.55	62.3	55	5,000	2,067
76.0	3,000	1,240	45 min.	6.50	7.10	95.5	59		
76.0	4,500	1,860	45 min.	6.50	6.80	97.8	64		
76.0	6,000	2,480	45 min.	6.50	6.80	98.4	63		
76.0	7,500	3,100	45 min.	6.50	6.60	99.0	61		
76.0	10,500	4,340	45 min.	6.50	6.10	99.1	57		
76.0	12,000	4,960	45 min.	6.50	6.30	97.8	53		
76.0	13,500	5,580	45 min.	6.50	5.80	99.0	51		
76.0	1,500	620	24 hrs.	6.50	. .	. .	. .	4,500	1,860
76.0	3,000	1,240	24 hrs.	6.50	7.30	95.6	47		
76.0	4,500	1,860	24 hrs.	6.50	7.80	98.0	48		
76.0	6,000	2,480	24 hrs.	6.50	6.55	99.0	47		
76.0	7,500	3,100	24 hrs.	6.50	6.70	99.0	46		
76.0	10,500	4,340	24 hrs.	6.50	6.45	99.1	41		
76.0	12,000	4,960	24 hrs.	6.50	6.45	100.1	38		
76.0	13,500	5,580	24 hrs.	6.50	6.20	99.3	41		
103.5	4,500	1,860	45 min.	6.60	. .	. .	. .		

TABLE 6 (Continued)

Sample Aeration Time (Hours)	Dosage (P.P.M.)		Settling Time	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum Coagulant Dosage (P.P.M.) at 98% Color Removal	
	$FeCl_3$	$Ca(OH)_2$						$FeCl_3$	$Ca(OH)_2$
103.5	6,000	2,480	45 min.	6.60	6.10	98.4	57	. .	. .
103.5	7,500	3,100	45 min.	6.60	6.20	99.1	56		
103.5	9,000	3,720	45 min.	6.60	5.75	99.1	56		
103.5	10,500	4,340	45 min.	6.60	5.80	100.0	54		
103.5	12,000	4,960	45 min.	6.60	5.65	100.0	53		
103.5	4,500	1,860	24 hrs.	6.60	. .	. .	. .	. .	. .
103.5	6,000	2,480	24 hrs.	6.60	6.90	98.4	44		
103.5	7,500	3,100	24 hrs.	6.60	7.00	99.1	41		
103.5	9,000	3,720	24 hrs.	6.60	6.20	99.0	40		
103.5	10,500	4,340	24 hrs.	6.60	6.60	99.1	41		
103.5	12,000	4,960	24 hrs.	6.60	6.20	98.8	39		
124.5	4,500	1,860	45 min.	6.65	6.80	97.5	53	6,667	2,757
124.5	6,000	2,480	45 min.	6.65	6.50	97.6	54		
124.5	7,500	3,100	45 min.	6.65	6.70	98.5	54		
124.5	9,000	3,720	45 min.	6.65	6.00	99.0	54		
124.5	10,500	4,340	45 min.	6.65	6.30	99.1	54		
124.5	12,000	4,960	45 min.	6.65	5.90	99.3	54		
124.5	4,500	1,860	24 hrs.	6.65	. .	. .	. .	. .	. .
124.5	6,000	2,480	24 hrs.	6.65	7.10	98.7	41		
124.5	7,500	3,100	24 hrs.	6.65	7.00	98.3	41		

TABLE 6 (Continued)

Sample Aeration Time (Hours)	Dosage (P.P.M.)		Settling Time	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum Coagulant Dosage (P.P.M.) at 98% Color Removal	
	$FeCl_3$	$Ca(OH)_2$						$FeCl_3$	$Ca(OH)_2$
124.5	9,000	3,720	24 hrs.	6.65	6.70	99.0	40	. .	. .
124.5	10,500	4,340	24 hrs.	6.65	6.80	98.9	39		
124.5	12,000	4,960	24 hrs.	6.65	6.60	99.0	39		
172.0	3,000	1,240	45 min.	6.70	6.30	93.5	56	7,140	2,952
172.0	4,500	1,860	45 min.	6.70	6.20	96.8	59		
172.0	6,000	2,480	45 min.	6.70	5.60	96.1	57		
172.0	7,500	3,100	45 min.	6.70	5.80	98.6	53		
172.0	9,000	3,720	45 min.	6.70	5.50	99.0	54		
172.0	10,500	4,340	45 min.	6.70	5.50	99.3	53		
172.0	3,000	1,240	24 hrs.	6.70	6.60	95.5	44	4,250	1,756
172.0	4,500	1,860	24 hrs.	6.70	6.90	98.5	45		
172.0	6,000	2,480	24 hrs.	6.70	6.10	98.6	44		
172.0	7,500	3,100	24 hrs.	6.70	6.70	98.7	40		
172.0	9,000	3,720	24 hrs.	6.70	6.10	99.7	40		
172.0	10,500	4,340	24 hrs.	6.70	6.50	100.0	39		
244.0	3,000	1,240	45 min.	6.85	6.75	85.1	31	6,000	2,480
244.0	4,500	1,860	45 min.	6.85	7.60	97.6	46		
244.0	6,000	2,480	45 min.	6.85	7.60	98.0	47		
244.0	7,500	3,100	45 min.	6.85	7.90	99.3	47		
244.0	9,000	3,720	45 min.	6.85	7.90	99.1	53		

TABLE 6 (Continued)

Sample Aeration Time (Hours)	Dosage (P.P.M.)		Settling Time	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum Coagulant Dosage (P.P.M.) at 98% Color Removal	
	$FeCl_3$	$Ca(OH)_2$						$FeCl_3$	$Ca(OH)_2$
244.0	10,500	4,340	45 min.	6.85	8.00	100.0	53		
244.0	3,000	1,240	24 hrs.	6.85	7.00	92.0	22	4,452	1,840
244.0	4,500	1,860	24 hrs.	6.85	7.50	98.2	30		
244.0	6,000	2,480	24 hrs.	6.85	7.50	98.6	34		
244.0	7,500	3,100	24 hrs.	6.85	7.50	98.8	34		
244.0	9,000	3,720	24 hrs.	6.85	7.60	99.3	40		
244.0	10,500	4,340	24 hrs.	6.85	8.00	99.0	38		

TABLE 7

THE EFFECT OF AERATION TIME ON CHEMICAL TREATMENT OF 803 BROWN DISPERSED DYE WASTE

Sample Aeration Time (Hours)	Coagulant Dosage (P.P.M.)		pH Adjusting Reagent	pH	Settling Time	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum pH Range
	$FeCl_3$	$Ca(OH)_2$								
0	12,000	4,960	H_2SO_4	2.70	45 min.	6.35	2.70	0	60	3.00 to 6.90
0	12,000	4,960	H_2SO_4	3.00	45 min.	6.35	3.00	89.5	64	
0	12,000	4,960	. .	4.40	45 min.	6.35	4.40	96.8	61	
0	12,000	4,960	Lime	4.50	45 min.	6.35	4.50	97.5	63	
0	12,000	4,960	Lime	4.50	45 min.	6.35	4.50	99.0	62	
0	12,000	4,960	Lime	5.40	45 min.	6.35	5.30	98.6	51	
0	12,000	4,960	Lime	6.20	45 min.	6.35	6.20	99.0	58	
0	12,000	4,960	Lime	6.80	45 min.	6.35	6.80	99.1	57	
0	12,000	4,960	Lime	6.90	45 min.	6.35	6.80	97.6	57	
0	12,000	4,960	H_2SO_4	2.70	24 hrs.	6.35	3.10	77.2	51	3.00 to 6.90
0	12,000	4,960	H_2SO_4	3.00	24 hrs.	6.35	3.20	95.4	51	
0	12,000	4,960	. .	4.40	24 hrs.	6.35	4.80	98.0	45	
0	12,000	4,960	Lime	4.50	24 hrs.	6.35	4.80	98.0	50	
0	12,000	4,960	Lime	4.50	24 hrs.	6.35	4.80	98.2	46	
0	12,000	4,960	Lime	5.40	24 hrs.	6.35	5.40	98.0	41	
0	12,000	4,960	Lime	6.20	24 hrs.	6.35	6.30	98.4	45	
0	12,000	4,960	Lime	6.80	24 hrs.	6.35	6.90	98.5	43	
0	12,000	4,960	Lime	6.90	24 hrs.	6.35	6.90	97.4	45	
51.0	12,000	4,960	H_2SO_4	2.80	45 min.	6.35	2.80	86.0	64	2.80 to 6.60
51.0	12,000	4,960	H_2SO_4	4.10	45 min.	6.35	4.10	97.9	58	
51.0	12,000	4,960	. .	4.30	45 min.	6.35	4.30	98.5	55	
51.0	12,000	4,960	Lime	5.50	45 min.	6.35	5.60	99.3	56	

TABLE 7 (Continued)

Sample Aeration Time (Hours)	Coagulant Dosage (P.P.M.)		pH Adjusting Reagent	pH	Settling Time	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum pH Range
	$FeCl_3$	$Ca(OH)_2$								
51.0	12,000	4,960	Lime	5.90	45 min.	6.35	5.90	99.3	54	4.10 to 6.60
51.0	12,000	4,960	Lime	6.60	45 min.	6.35	6.60	99.0	52	
51.0	12,000	4,960	H_2SO_4	2.80	24 hrs.	6.35	2.50	96.2	52	2.80 to 6.60
51.0	12,000	4,960	H_2SO_4	4.10	24 hrs.	6.35	3.90	99.0	42	
51.0	12,000	4,960	• •	4.30	24 hrs.	6.35	3.90	100.0	42	
51.0	12,000	4,960	Lime	5.50	24 hrs.	6.35	5.60	99.1	42	
51.0	12,000	4,960	Lime	5.90	24 hrs.	6.35	6.10	99.3	40	
51.0	12,000	4,960	Lime	6.60	24 hrs.	6.35	6.70	99.7	41	
76.0	12,000	4,960	H_2SO_4	4.60	45 min.	6.50	4.60	94.8	55	4.60 to 9.00
76.0	12,000	4,960	H_2SO_4	5.70	45 min.	6.50	5.70	98.0	54	
76.0	12,000	4,960	• •	6.20	45 min.	6.50	6.20	98.2	54	
76.0	12,000	4,960	Lime	7.30	45 min.	6.50	6.80	98.4	52	
76.0	12,000	4,960	Lime	8.30	45 min.	6.50	7.80	97.5	52	
76.0	12,000	4,960	Lime	9.00	45 min.	6.50	8.70	98.4	51	
76.0	12,000	4,960	H_2SO_4	4.60	24 hrs.	6.50	4.60	98.4	42	4.60 to 9.00
76.0	12,000	4,960	H_2SO_4	5.70	24 hrs.	6.50	5.90	98.4	39	
76.0	12,000	4,960	• •	6.20	24 hrs.	6.50	6.40	99.4	39	
76.0	12,000	4,960	Lime	7.30	24 hrs.	6.50	6.70	99.3	39	
76.0	12,000	4,960	Lime	8.30	24 hrs.	6.50	7.30	99.3	40	
76.0	12,000	4,960	Lime	9.00	24 hrs.	6.50	8.20	99.1	40	

TABLE 7 (Continued)

Sample Aeration Time (Hours)	Coagulant Dosage (P.P.M.)		pH Adjusting Reagent	pH	Settling Time	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum pH Range
	$FeCl_3$	$Ca(OH)_2$								
103.5	10,500	4,340	H_2SO_4	4.00	45 min.	6.60	4.10	96.1	57	4.00 to 9.60
103.5	10,500	4,340	H_2SO_4	5.50	45 min.	6.60	5.60	97.0	56	
103.5	10,500	4,340	. .	6.20	45 min.	6.60	6.20	98.4	55	
103.5	10,500	4,340	Lime	6.90	45 min.	6.60	6.70	99.7	55	
103.5	10,500	4,340	Lime	8.40	45 min.	6.60	8.40	98.4	54	
103.5	10,500	4,340	Lime	9.60	45 min.	6.60	9.40	98.2	56	
103.5	10,500	4,340	H_2SO_4	4.00	24 hrs.	6.60	4.00	99.0	44	4.00 to 9.60
103.5	10,500	4,340	H_2SO_4	5.50	24 hrs.	6.60	5.80	99.0	40	
103.5	10,500	4,340	. .	6.20	24 hrs.	6.60	6.55	99.3	41	
103.5	10,500	4,340	Lime	6.90	24 hrs.	6.60	7.30	98.7	40	
103.5	10,500	4,340	Lime	8.40	24 hrs.	6.60	8.85	98.5	40	
103.5	10,500	4,340	Lime	9.60	24 hrs.	6.60	10.20	99.9	42	
124.5	9,000	3,720	H_2SO_4	4.50	45 min.	6.65	4.70	96.8	62	4.50 to 8.80
124.5	9,000	3,720	H_2SO_4	5.90	45 min.	6.65	5.80	98.2	54	
124.5	9,000	3,720	. .	6.20	45 min.	6.65	6.10	99.3	56	
124.5	9,000	3,720	Lime	6.90	45 min.	6.65	6.80	100.0	52	
124.5	9,000	3,720	Lime	7.50	45 min.	6.65	7.20	98.8	52	
124.5	9,000	3,720	Lime	8.80	45 min.	6.65	8.50	98.6	52	
124.5	9,000	3,720	H_2SO_4	4.50	24 hrs.	6.65	4.90	98.4	44	4.50 to 8.80
124.5	9,000	3,720	H_2SO_4	5.90	24 hrs.	6.65	6.60	99.1	39	
124.5	9,000	3,720	. .	6.20	24 hrs.	6.65	6.60	99.4	42	

TABLE 7 (Continued)

Sample Aeration Time (Hours)	Coagulant Dosage (P.P.M.)		pH Adjusting Reagent	pH	Settling Time	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum pH Range
	$FeCl_3$	$Ca(OH)_2$								
124.5	9,000	3,720	Lime	6.90	24 hrs.	6.65	7.60	99.1	40	4.50 to 8.80
124.5	9,000	3,720	Lime	7.50	24 hrs.	6.65	8.10	99.3	40	
124.5	9,000	3,720	Lime	8.80	24 hrs.	6.65	8.90	99.4	41	
244.0	7,500	3,100	H_2SO_4	5.10	45 min.	6.85	6.00	98.2	44	5.10 to 10.60
244.0	7,500	3,100	H_2SO_4	6.30	45 min.	6.85	7.10	98.7	46	
244.0	7,500	3,100	• •	6.90	45 min.	6.85	7.80	99.7	60	
244.0	7,500	3,100	Lime	8.30	45 min.	6.85	8.80	99.1	69	
244.0	7,500	3,100	Lime	9.60	45 min.	6.85	9.90	99.1	62	
244.0	7,500	3,100	Lime	10.60	45 min.	6.85	10.60	98.9	61	
244.0	7,500	3,100	H_2SO_4	5.10	24 hrs.	6.85	6.70	97.8	30	5.10 to 10.60
244.0	7,500	3,100	H_2SO_4	6.30	24 hrs.	6.85	7.40	98.6	34	
244.0	7,500	3,100	• •	6.90	24 hrs.	6.85	7.50	99.4	37	
244.0	7,500	3,100	Lime	8.30	24 hrs.	6.85	8.40	99.4	46	
244.0	7,500	3,100	Lime	9.60	24 hrs.	6.85	8.90	100.0	44	
244.0	7,500	3,100	Lime	10.60	24 hrs.	6.85	10.00	98.0	43	

TABLE 8

THE EFFECT OF AERATION TIME ON CHEMICAL TREATMENT OF 803 BROWN DISPERSED DYE WASTE

Sample Aeration Time (Hours)	Dosage (P.P.M.)	Settling Time	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum Coagulant Dosage (P.P.M.) at 93% Color Removal
	Purifloc C31						Purifloc C31
0	667	45 min.	6.35	6.60	0	19	1,277
0	1,000	45 min.	6.35	6.75	65.5	45	
0	1,333	45 min.	6.35	6.80	98.5	41	
0	1,667	45 min.	6.35	6.90	96.3	31	
0	2,000	45 min.	6.35	6.90	98.4	25	
0	2,333	45 min.	6.35	7.00	98.2	16	
0	3,333	45 min.	6.35	7.20	97.6	25	
0	667	24 hrs.	6.35	. .	. .	. .	. .
0	1,000	24 hrs.	6.35	. .	. .	. .	
0	1,333	24 hrs.	6.35	6.90	99.0	31	
0	1,667	24 hrs.	6.35	7.00	98.6	24	
0	2,000	24 hrs.	6.35	7.00	99.4	18	
0	2,333	24 hrs.	6.35	7.10	98.2	18	
0	3,333	24 hrs.	6.35	7.25	97.4	18	
51.0	667	45 min.	6.35	6.70	0	14	1,235
51.0	1,000	45 min.	6.35	6.80	78.0	48	
51.0	1,333	45 min.	6.35	7.00	99.3	43	
51.0	1,667	45 min.	6.35	7.00	99.0	29	
51.0	2,000	45 min.	6.35	7.10	99.1	28	
51.0	2,333	45 min.	6.35	7.20	98.7	16	
51.0	667	24 hrs.	6.35	6.60	0	13	

TABLE 8 (Continued)

Sample Aeration Time (Hours)	Dosage (P.P.M.) Purifloc C31	Settling Time	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum Coagulant Dosage (P.P.M.) at 93% Color Removal Purifloc C31
51.0	1,000	24 hrs.	6.35	6.60	85.9	45	1,189
51.0	1,333	24 hrs.	6.35	6.80	98.4	32	
51.0	1,667	24 hrs.	6.35	6.90	96.6	31	
51.0	2,000	24 hrs.	6.35	7.00	97.6	19	
51.0	2,333	24 hrs.	6.35	7.10	97.9	24	
76.0	833	45 min.	6.50	. .	. .	. .	. .
76.0	1,000	45 min.	6.50	6.95	98.7	42	
76.0	1,167	45 min.	6.50	6.95	98.4	43	
76.0	1,333	45 min.	6.50	7.00	98.9	40	
76.0	1,500	45 min.	6.50	7.05	98.6	30	
76.0	1,667	45 min.	6.50	7.05	99.1	26	
76.0	833	24 hrs.	6.50	. .	. .	. .	. .
76.0	1,000	24 hrs.	6.50	6.90	99.3	38	
76.0	1,167	24 hrs.	6.50	6.80	98.9	37	
76.0	1,333	24 hrs.	6.50	6.90	98.8	37	
76.0	1,500	24 hrs.	6.50	6.80	99.7	22	
76.0	1,667	24 hrs.	6.50	6.90	99.0	17	
103.5	833	45 min.	6.60	6.90	40.3	31	1,000
103.5	1,000	45 min.	6.60	7.00	93.8	45	
103.5	1,167	45 min.	6.60	7.00	98.4	45	
103.5	1,333	45 min.	6.60	7.00	97.6	38	

TABLE 8 (Continued)

Sample Aeration Time (Hours)	Dosage (P.P.M.) Purifloc C31	Settling Time	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum Coagulant Dosage (P.P.M.) at 93% Color Removal Purifloc C31
103.5	1,500	45 min.	6.60	7.10	98.4	29	
103.5	1,667	45 min.	6.60	7.10	98.4	24	
103.5	833	24 hrs.	6.60	6.80	41.9	23	992
103.5	1,000	24 hrs.	6.60	6.90	95.5	42	
103.5	1,167	24 hrs.	6.60	6.90	98.6	33	
103.5	1,333	24 hrs.	6.60	7.00	99.9	29	
103.5	1,500	24 hrs.	6.60	7.05	99.9	23	
103.5	1,667	24 hrs.	6.60	7.10	100.0	20	
124.5	833	45 min.	6.65	6.90	36.6	43	996
124.5	1,000	45 min.	6.65	6.90	94.2	45	
124.5	1,167	45 min.	6.65	6.95	99.0	45	
124.5	1,333	45 min.	6.65	7.00	98.6	35	
124.5	1,500	45 min.	6.65	7.10	98.6	33	
124.5	1,667	45 min.	6.65	7.10	97.7	26	
124.5	833	24 hrs.	6.65	7.00	37.5	32	1,029
124.5	1,000	24 hrs.	6.65	7.00	92.2	35	
124.5	1,167	24 hrs.	6.65	7.05	96.8	31	
124.5	1,333	24 hrs.	6.65	7.00	97.4	28	
124.5	1,500	24 hrs.	6.65	7.10	98.0	23	
124.5	1,667	24 hrs.	6.65	7.10	97.8	14	

TABLE 8 (Continued)

Sample Aeration Time (Hours)	Dosage (P.P.M.)	Settling Time	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum Coagulant Dosage (P.P.M.) at 93% Color Removal
	Purifloc C31						Purifloc C31
244.0	667	45 min.	6.85	6.60	0	14	1,148
244.0	833	45 min.	6.85	6.70	28.6	28	
244.0	1,000	45 min.	6.85	6.80	73.0	30	
244.0	1,000	45 min.	6.85	6.85	75.9	29	
244.0	1,167	45 min.	6.85	7.00	97.4	31	
244.0	1,333	45 min.	6.85	7.10	97.2	32	
244.0	1,500	45 min.	6.85	7.20	96.8	26	
244.0	667	24 hrs.	6.85	6.80	0	13	1,000
244.0	833	24 hrs.	6.85	6.80	47.3	15	
244.0	1,000	24 hrs.	6.85	6.90	84.8	15	
244.0	1,000	24 hrs.	6.85	6.90	100.0	19	
244.0	1,167	24 hrs.	6.85	7.05	98.9	22	
244.0	1,333	24 hrs.	6.85	7.05	100.0	15	
244.0	1,500	24 hrs.	6.85	6.90	84.4	15	

TABLE 9

THE EFFECT OF AERATION TIME ON CHEMICAL TREATMENT OF 803 BROWN DISPERSED DYE WASTE

Sample Aeration Time (Hours)	Coagulant Dosage (P.P.M.) Purifloc C31	pH Adjusting Reagent	pH	Settling Time	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum pH Range
0	1,333	H_2SO_4	3.30	45 min.	6.35	3.30	98.6	32	3.30 to 6.90
0	1,333	H_2SO_4	4.30	45 min.	6.35	4.40	98.5	41	
0	1,333	. .	6.90	45 min.	6.35	6.90	98.6	44	
0	1,333	Lime	8.50	45 min.	6.35	8.50	36.9	29	
0	1,333	Lime	10.30	45 min.	6.35	10.20	0	10	
0	1,333	H_2SO_4	3.30	24 hrs.	6.35	3.60	98.7	31	3.30 to 6.90
0	1,333	H_2SO_4	4.30	24 hrs.	6.35	4.70	99.3	41	
0	1,333	. .	6.90	24 hrs.	6.35	6.90	99.0	41	
0	1,333	Lime	8.50	24 hrs.	6.35	8.30	40.0	26	
0	1,333	Lime	10.30	24 hrs.	6.35	9.90	0	12	
51.0	1,333	H_2SO_4	3.60	45 min.	6.35	3.70	99.2	31	3.60 to 8.65
51.0	1,333	H_2SO_4	4.80	45 min.	6.35	4.80	98.9	35	
51.0	1,333	. .	6.90	45 min.	6.35	6.80	98.3	35	
51.0	1,333	Lime	7.55	45 min.	6.35	7.40	99.2	35	
51.0	1,333	Lime	8.65	45 min.	6.35	8.30	91.8	26	
51.0	1,333	Lime	9.90	45 min.	6.35	9.60	0	12	
51.0	1,333	H_2SO_4	3.60	24 hrs.	6.35	3.80	98.4	32	3.60 to 8.65
51.0	1,333	H_2SO_4	4.80	24 hrs.	6.35	5.80	98.4	41	
51.0	1,333	. .	6.90	24 hrs.	6.35	6.90	98.5	37	
51.0	1,333	Lime	7.55	24 hrs.	6.35	7.55	97.6	31	
51.0	1,333	Lime	8.65	24 hrs.	6.35	8.20	96.0	29	
51.0	1,333	Lime	9.90	24 hrs.	6.35	9.40	0	12	

TABLE 9 (Continued)

Sample Aeration Time (Hours)	Coagulant Dosage (P.P.M.)	pH Adjusting Reagent	pH	Settling Time	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum pH Range
	Purifloc C31								
76.0	1,000	H_2SO_4	2.70	45 min.	6.50	2.90	98.7	31	2.70 to 6.85
76.0	1,000	H_2SO_4	4.40	45 min.	6.50	4.60	99.0	44	
76.0	1,000	. .	6.85	45 min.	6.50	6.85	81.8	47	
76.0	1,000	Lime	7.70	45 min.	6.50	7.60	57.4	30	
76.0	1,000	Lime	10.40	45 min.	6.50	10.40	8.8	10	
76.0	1,000	Lime	11.30	45 min.	6.50	11.30	19.5	12	
76.0	1,000	H_2SO_4	2.70	24 hrs.	6.50	2.80	99.1	28	2.70 to 6.85
76.0	1,000	H_2SO_4	4.40	24 hrs.	6.50	4.80	99.1	32	
76.0	1,000	. .	6.85	24 hrs.	6.50	6.90	96.2	45	
76.0	1,000	Lime	7.70	24 hrs.	6.50	7.60	57.5	27	
76.0	1,000	Lime	10.40	24 hrs.	6.50	9.90	0	7	
76.0	1,000	Lime	11.30	24 hrs.	6.50	10.30	26.2	12	
103.5	1,000	H_2SO_4	2.90	45 min.	6.60	3.00	96.7	31	2.90 to 6.90
103.5	1,000	H_2SO_4	4.60	45 min.	6.60	4.60	97.8	39	
103.5	1,000	. .	6.90	45 min.	6.60	6.95	92.6	47	
103.5	1,000	Lime	7.80	45 min.	6.60	7.80	69.4	36	
103.5	1,000	Lime	10.30	45 min.	6.60	10.30	3.0	7	
103.5	1,000	Lime	11.45	45 min.	6.60	11.45	19.6	12	
103.5	1,000	H_2SO_4	2.90	24 hrs.	6.60	2.85	99.3	21	2.90 to 6.90
103.5	1,000	H_2SO_4	4.60	24 hrs.	6.60	4.80	99.3	27	
103.5	1,000	. .	6.90	24 hrs.	6.60	6.80	94.6	42	

TABLE 9 (Continued)

Sample Aeration Time (Hours)	Coagulant Dosage (P.P.M.) Purifloc C31	pH Adjusting Reagent	pH	Settling Time	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum pH Range
103.5	1,000	Lime	7.80	24 hrs.	6.60	7.50	70.5	30	2.90 to 6.90
103.5	1,000	Lime	10.30	24 hrs.	6.60	9.60	0	7	
103.5	1,000	Lime	11.45	24 hrs.	6.60	10.40	23.8	12	
124.5	1,000	H_2SO_4	3.10	45 min.	6.65	3.10	97.0	30	3.10 to 6.95
124.5	1,000	H_2SO_4	4.80	45 min.	6.65	4.90	98.4	41	
124.5	1,000	. .	6.95	45 min.	6.65	6.95	92.2	44	
124.5	1,000	Lime	7.80	45 min.	6.65	7.75	80.9	33	
124.5	1,000	Lime	10.45	45 min.	6.65	10.45	6.5	9	
124.5	1,000	Lime	11.10	45 min.	6.65	11.00	12.2	12	
124.5	1,000	H_2SO_4	3.10	24 hrs.	6.65	3.40	97.4	24	3.10 to 6.95
124.5	1,000	H_2SO_4	4.80	24 hrs.	6.65	5.30	98.2	32	
124.5	1,000	. .	6.95	24 hrs.	6.65	6.95	92.8	35	
124.5	1,000	Lime	7.80	24 hrs.	6.65	7.50	81.0	29	
124.5	1,000	Lime	10.45	24 hrs.	6.65	9.90	0	7	
124.5	1,000	Lime	11.10	24 hrs.	6.65	10.10	17.4	13	
244.0	1,167	H_2SO_4	3.30	45 min.	6.85	3.80	97.2	29	3.30 to 6.80
244.0	1,167	H_2SO_4	5.80	45 min.	6.85	6.00	98.0	43	
244.0	1,167	. .	6.80	45 min.	6.85	7.00	97.8	39	
244.0	1,167	Lime	8.70	45 min.	6.85	8.90	74.8	18	
244.0	1,167	NaOH	10.70	45 min.	6.85	10.50	9.6	0	
244.0	1,167	H_2SO_4	3.30	24 hrs.	6.85	4.00	100.0	18	3.30 to 6.80

TABLE 9 (Continued)

Sample Aeration Time (Hours)	Coagulant Dosage (P.P.M.)	pH Adjusting Reagent	pH	Settling Time	Initial pH of Waste	Final pH of Waste	Per Cent Color Removal	Per Cent Sludge Volume	Optimum pH Range
	Purifloc C31								
244.0	1,167	H_2SO_4	5.80	24 hrs.	6.85	6.40	100.0	21	3.30 to 6.80
244.0	1,167	• •	6.80	24 hrs.	6.85	7.50	99.0	18	
244.0	1,167	Lime	8.70	24 hrs.	6.85	9.00	80.9	13	
244.0	1,167	NaOH	10.70	24 hrs.	6.85	10.50	14.0	0	

ABSTRACT

THE EFFECT OF BIOOXIDATION ON THE COAGULATION OF DISPERSED COLOR

by

Gary Thomas Laing

The objective of this investigation was to determine the effect of aerated lagoon biological treatment on color removal by chemical coagulation of two dark dispersed dye wastes. Chemical coagulation was desirable because it effectively reduced the color of the dispersed dye waste. The aerated lagoon was thought to be a possible means of reducing the chemical coagulant demand of dye wastes.

A simulated, aerated lagoon operated on a batch basis was used to treat samples of dye wastes, for various periods of time. Samples withdrawn from the lagoon were periodically analyzed for B.O.D., C.O.D., optimum coagulant dosage, suspended solids, and color removal. Reductions in C.O.D. up to 34 per cent were effected by the aerated lagoon. The aerated lagoon also removed 3.5 to 25 per cent of the color of the original waste. Chemical coagulation effected 90 to 100 per cent color removal and about 68 per cent C.O.D. removal. Reductions in the chemical coagulant dosage caused by the aerated lagoon ranged from 21 to 41 per cent. The per cent sludge volume after settling for 45 minutes and 24 hours ranged from 45 to 64 per cent and from 41 to 48 per cent, respectively. Although settling

was complete in 45 minutes, the sludge continued to compact for 24 hours.