



Biochemical Treatment of Aqueous Wastewater Effluents from a Local Textile Industry

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ABSTRACT

This work is concerned with the finding of the optimum conditions for biochemical aqueous wastewater effluents treatment for a local textile mill. The water samples were taken from the Sunflag Textile Mill, Lagos, Nigeria (the aqueous wastewater effluents of the fabrics, leathers, dyes and other solid waste) in equal volumes and subjected to incubation, sedimentation, biological treatment, chemical treatment, and chemical and natural sedimentation treatment. The Box-Wilson method of experimental design was adopted to analyze the physio-chemical and biochemical effects on the wastewater effluents. The physio-chemical and biochemical analyses carried out on the sample revealed that the most favourable optimum operating conditions for the treatment variables are: Temperature 34.5°C, Contact period 40 hours, PH 7.3, Biological Oxygen Demand 28.77 mg/l, Chemical Oxygen Demand 11.71 mg/l and Total Suspended Solid 26.51 mg/l. The analyzed study showed a result that meets the Federal Environmental Protection Agency and Lagos State Environmental Protection Agency effluents treatment standard.

Keyword: Aqueous wastewater effluents, Biological Oxygen Demand, Chemical Oxygen Demand, Total Suspended Solid, Temperature, PH.

INTRODUCTION

In Textile Industry (fabrics) and for practical reasons, almost all textile mills are located on rivers to provide them with process water (30 to 80 m³ for 1 ton of processed raw skins) [1] and for the disposal of their effluents. The effluent from this industry is mainly waterborne [2]. The components of the effluent arise from purification of raw fibers, dyes from colouring, hides and skins before processing as well as from residual chemicals from the production processes. Hence, the textile industry had been recognized as a major contributor to water pollution problems. Biodegradable organic matter consumes oxygen and nutrients in complex biochemical reactions until rendered inert [3]. This exerts a biochemical oxygen demand as one of the fundamental parameters used to regulate the quality of the effluent [4]. The contaminants in industrial wastewater are removed by physical, chemical and biological means [5]. Facilities for handling wastewater are usually considered to have three major parts: collection, treatment and disposal [6]. Pre-aeration is used to improve the wastewater and to assist the removal of oil and grease [7]. Secondary treatment processes commonly consist of biological processes. This means that living organisms which control the environment of the process are used to partially stabilize (oxidize) organic matter not previously removed by treatment processes and to convert it into a form which is easier to remove from the wastewater [8]. Sedimentation or primary treatment makes the wastewater much clearer. Two clarifiers are used [9] to provide detention time (3 hours) where, almost 60% of the suspended solids (SS) will either settle to the bottom

or float to the surface and be removed. Removal of these solids will usually reduce the BOD of the waste approximately 35%.

The next step is the biological treatment which can typically be divided into aerobic and anaerobic. Anaerobic biological treatment is an oxygen-devoid process. Aerobic biological treatment is done in the presence of oxygen. It is applicable to wastewater containing bio-degradable organic constituents and some non-metallic inorganic constituents [2]. The bacterium, *Bacillus Subtilis* using the Activated Sludge Process (ASP) is the currently used biological treatment process for wastewater in most textile and tanning factory in Nigeria. The system consists of an equalization basin, a settling tank, an aeration basin, a clarifier, and a sludge line. The recirculation of the biomass, which is an integral part of the process, allow microorganisms to adapt changes in wastewater composition with relatively short acclimation time and also allow a greater degree of control over acclimated bacterial population [10]. For a proper control of the ASP, the growth of the micro-organisms should be controlled. Bacteria make up about 95% of the activated sludge biomass. These single celled organisms grow in the wastewater by consuming (eating) biodegradable materials such as proteins, carbohydrates, fats and many other compounds. Some important factors acting on growth and activity of bacteria in biochemical wastewater treatment are: food-to-microorganism ratio (F/M); use of oxygen [11]; formation of Floc [12]; mixing [12]; P^H [13]; temperature [6] and the effect of nutrients [14]. Biological oxygen demand (BOD) is a measure of the oxygen used by

microorganisms to decompose this waste. A large quantity of organic waste in the water requires large amount of bacteria to decompose it. Thus, the demand for oxygen will be high (high level of BOD). As the waste is consumed or dispersed through the water, BOD levels will begin to decline [15]. Nitrates and phosphates in a body of water can contribute to high BOD levels. Nitrates and phosphates are plant nutrients and can cause plant life and algae to grow quickly. When plants grow quickly, they also die quickly. This contributes to the organic waste in the water, which is then decomposed by bacteria. This results in a high BOD level [15]. When BOD levels are high, dissolved oxygen (DO) levels decrease because the oxygen that is available in the water is being consumed by the bacteria. Since less dissolved oxygen is available in the water, fish and other aquatic organisms may not survive [16].

Objective of this study

The present work is an attempt to shed more light on the pollution potential of Textile Industries in Nigeria using Sunflag Textile Mill Industry as a case study and to find optimum operating conditions for treating aqueous wastewater effluents from our local textile industries.

Parameters by which Wastewater is measured in Textile Industries

Urban waste water in our local textile industries is characterized in terms of its physical, chemical and biological constituents. The strength of waste water is normally measured using accurate analytical techniques. The more common analyses used to characterize waste water entering and leaving a plant are:

- BOD
- COD
- TSS
- P^H
- Total phosphorus
- Total nitrogen

Biological Oxygen Demand (BOD):

BOD is the amount of oxygen used by organisms while consuming organic matter in a waste water sample. It is possible to assess the performance of a waste water treatment plant by measuring the BOD of the inflow and the outflow. Many factors can influence this test, such as temperature of incubation, dilution rate, nitrification, toxic substances, and nature of bacterial seed and presence of anaerobic organisms. The method of measurement for the BOD test in the Urban Waste Water Treatment (UWWT) Regulations requires:

- that the sample is homogenized, unfiltered and undecanted; and
- that a nitrification inhibitor is added.

The UWWT Regulations allow for the BOD test to be replaced by another parameter, total organic carbon (TOC) or total oxygen demand (TOD) if a relationship can be established between BOD and the substitute parameter.

Chemical Oxygen Demand (COD):

The COD test uses the oxidizing agent potassium dichromate (specified in the UWWT Regulations) to oxidize organic matter in the sample. The test is extensively used because it takes less time (about 3 hours) than other tests such as the BOD, which takes 5 days. The COD test does not, however, differentiate between biodegradable and non-biodegradable organic matter. For municipal waste water it is generally possible to establish a relationship between COD and BOD. Once a correlation has been established, the COD test can be a very useful indicator for the operation and control of the plant.

Total suspended solids (TSS):

This is the sum of the organic and inorganic solids concentrations and can be subdivided into:

- Suspended Solids (SS): represent the solids that are in suspension in the water. Generally comprised of 70% organic and 30% inorganic solids and can be removed by physical or mechanical means.
- Organic Solids (OS): about 50% of solids present in urban waste water derive from the waste products of animal and vegetable life. Sometimes called the combustible fraction or volatile solids as these can be driven off by high temperature.
- Inorganic Solids (IS): these substances are inert and are not subject to decay. Include sand, gravel and silt.
- Settleable Solids (SS): this is a subset of suspended solids and represents that fraction of suspended solids that will settle in a given period.
- Colloidal Suspended Solids (CSS): these refer to solids that are not truly dissolved and yet do not settle readily. They tend to refer to organic and inorganic solids that rapidly decay.

P^H : This is the concentration of hydrogen ions in solution and indicates the level of acidity or alkalinity of an aqueous solution. If the P^H of the waste water is outside the range 5-10, there may be considerable interference with biological processes.

Total phosphorus: This parameter is normally divided into three fractions, namely:

- orthophosphate: dissolved inorganic phosphate (PO_4^{3-})
- polyphosphates: complex compounds generally derived from detergents
- organically bound phosphate: dissolved and suspended organic phosphates

Total phosphorus analysis requires two steps:

- conversion of polyphosphates and organically bound phosphorus to dissolved orthophosphate, and
- Colorimetric determination of the dissolved orthophosphate.

Total Nitrogen: This refers to the sum of measurements of total oxidized nitrogen (nitrate and nitrite) and total Kjeldahl nitrogen (ammonia and organic nitrogen). This parameter is a

growth limiting nutrient in marine environments and a limit is specified in the UWWT Regulations for discharges to sensitive water bodies.

Bacteria Biodegradation

Wastewater from textile mill industry contains different varieties of dyes. Efforts were made during the 1970s to isolate bacterial cultures capable of degrading azo-dyes (*Bacillus subtilis*). Mixed bacteria cultures from a variety of habitats have also been shown to decolorize the diazotized chromophore of dye molecules in about 15 days. The ability of bacteria to metabolize azo-dyes had been investigated. Under aerobic conditions, azo-dyes are not readily metabolized, the intermediates formed by these degradative steps resulted in disruption of metabolic pathways and the dyes were not actually mineralized. Under aerobic conditions, many bacteria gratuitously reduce azo-dyes reportedly by the activity of unspecific, soluble, cytoplasmic redundancies known as azo-redundancies. These enzymes are reported to result in the production of colourless aromatic amines which may be toxic, mutagenic and possibly carcinogenic to animals.

EXPERIMENTAL PROCEDURE

Materials and Samples Collection

Wastewater samples were collected from a stream receiving wastewater effluents from Sunflag Textile Mill Plant, Surulere, Lagos, Nigeria. Material used for the collection of the sample were pre-heated by washing the sample container with dilute hydrochloric acid [HCL] and rinsed with distilled water. This was to ensure that the container did not react with the sample. The containers were later dried in an oven for about 60 minutes at a temperature of $105 \pm 5^\circ\text{C}$ and allowed to cool at ambient temperature. At the collection point, the containers were also rinsed with the sample twice and were filled with the sample, corked tightly and were taken to the laboratory for treatment and analyses. Samples were stored at a temperature of 34.5°C to avoid any change in the physio-chemical characteristics. The containers used for collecting the sample were also rinsed with the sample twice before being filled with the sample, after which the container were corked tightly before taken to the laboratory for treatment and analyses. The collected samples were stored at a temperature of 27°C to avoid any change in the physio-chemical characteristics. The samples were then analyzed for the following: P^{H} , BOD, COD, TSS and the Colour quality (absorbent) of the effluents [17].

MATERIALS/EQUIPMENT AND REAGENTS

The materials and equipment as well as the reagents used were of good analytical grade and were obtained from **Federal Industrial Institute of Research (FIIR) Oshodi, Lagos State, Nigeria**. All the analyses were also carried out at the laboratory of Water Technology Nigeria limited, No. 17 Murtala Mohammed International Airport Road, Ajao Estate, Oshodi, Lagos State, Nigeria.

The materials and Equipment were: P^{H} meter, P^{H} papers, BOD bottle (300ml), Incubator, Measuring cylinder, Heater, 20D model spectrometric spectrophotometer, Beakers, Test tubes,

Burettes, Pipette, Masking tapes, Test tube holders, Cell DX 325.

The Reagents were: Potassium permanganate, manganese sulphate, potassium iodide, concentrated tetraoxosulphate (VI) acid, sodium thiosulphates, and starch, de-ionized water/distilled water, potassium dichromate, ferrous ammonium sulphate, ferroin indicator, indicator, mercuric sulphate and silver sulphate.

Determining the effects of physio-chemical parameters in the sample

The water samples were taken from the Sunflag Textile Mill, Lagos, Nigeria (the aqueous wastewater effluents of the fabrics, leathers, dyes and other solid waste) in equal volumes and subjected to incubation, sedimentation, biological treatment, chemical treatment, and chemical and natural sedimentation treatment. The samples were first screened to remove fabrics, hair and skins pieces and then neutralized by adding sulfuric acid (50 wt. %) to a final P^{H} of (7-9). Sedimentation (settling) process was then carried out to reduce the solid content to about 65% within 2-3 hours. A specified volume of the sample (30 litres) was placed in the bath. The microorganism (*Bacillus subtilis*) were added to the water sample in the bath, and aeration was started.

P^{H} of the effluents

This was carried out by measuring 10 ml of the effluent sample as well as those of the treated sample into the containers separately. The P^{H} of each of the sample was determined with the aid of the P^{H} meter. The P^{H} meter was calibrated using P^{H} 4.0 and 9.0 buffers.

BOD of the effluents

The BOD bottles were filled with wastewater sample. The bottles were closed tightly. One of the bottles was placed in an incubator at 34.5°C . The incubation of the sample was for 5 days. Prior to incubation, the dissolved oxygen of the sample was measured and recorded as D_0 . The oxygen was also measured after incubation and was recorded as D_1 . 2 ml of manganese sulphate solution was added to the sample, followed by 2 ml of potassium iodide reagents well below the surface of the liquid. The solution was then mixed carefully and the precipitate was allowed to settle down. The 2 ml of concentrated tetraoxosulphate (VI) acid was added and mixed well until the precipitates dissolved. The bottle was then re-stoppered and mixed by gentle inversion until dissolution was complete. 100 ml of the sample was taken from the BOD bottle and titrated with 0.025N normality of sodium thiosulphate solution using 1.0 ml of starch solution as an indicator. Titration was stopped where there was an observed colour change from blue to colourless. The volume at which this change in colour occurs was recorded and the BOD value was then calculated.

COD of the effluents

15 ml of concentrated tetraoxosulphate (VI) acid with 0.3 g of mercuric sulphate and a pinch of silver sulphate along with 5 ml of 0.025N of potassium dichromate was taken into a test tube. 10 ml of the sample (thoroughly mixed) was pipetted out into the mixture and kept for about 90 minutes on a hot plate for digestion. 40 ml of distilled water was added to the cooked mixture to make up to 50 ml and titrated against 0.25N

normality FAS using ferroin indicator till the colour change from blue green to wine red indicating an end point.

TSS of the effluents

The TSS tests were done according to the **Nigerian Federal Environmental Protection Agency (FEPA)** and **Lagos State Environmental Protection Agency (LASEPA)** methods for pollution control, 2001 [18, 19]. The dissolved organic concentration was measured using a (Cell DX 325) type device, which consists of a gold-metal electrode (**Water Technology, Nigeria Limited, Lagos State, Nigeria**). The accuracy of this method is (0.1 mg/l). The readings were checked versus titration method with standard 0.025N normality sodium thiosulphate using starch as an indicator. The dilution, the BOD measurement and the determination of the phosphate concentration were carried out in accordance with standard methods [17]. The TSS of the effluents was then determined by subtracting total solids from total dissolved solids. The total solids refer to the solid residue in the vessel after evaporation of the sample while total dissolved solids refer to the solids that are dissolved in solution.

Colour Quality (Absorbency rate) of the effluents

A spectromic 20D model spectrophotometer was used to measure the effluents sample. The corvettes of the spectrophotometer were clear plastic and held one millimeter of the sample. The light was at a wavelength of 515nm due to the

colour of the sample. The spectrophotometer compared each sample to the blank containing de-ionized water. Each sample was read and the absorbency rate recorded.

Bacillus subtilis on effluent wastewater

Samples of the wastewater were divided into 5 portions of equal volumes. The total volume of the waste water sample was 750 ml. Each portion of the wastewater had volume of 150 ml. after which each of the volume was treated with varying concentrations of the bacterium, bacillus subtilis. The concentrations used were as follows: 0.5 ml, 1.5 ml, 2.5 ml, 3.5 ml and 4.5 ml. Each of the 5 volumes was mixed slowly using a stirrer for 20 minutes to ensure good sample of bacillus subtilis contact. After this the effluent were then collected and the parameters P^H, BOD, COD, and TSS are analyzed for. It should be noted that, in the case of colour quality determination, the same volume of the bacterium was used. The volume used was 3.5 ml. In this case, the period of biological contact between the sample and the bacterium was varied.

RESULTS AND DISCUSSION

This work deals with estimating the effect of these variables, temperature, contact period and bacillus subtilis concentration on the PH, BOD, COD, TSS and Colour quality (absorbency rate) of the aqueous wastewater effluents.

Table 1: Effluents limitation standard and guideline in Nigeria (2001)

Parameters	FEPA	LASEPA
Temperature	40	30-40
P ^H	6.9	6.5-8.0
BOD	20	30
TSS	30	20
COD	10	12

Table 2: Quality of effluent wastewater before treatment with the bacterium bacillus subtilis

P ^H	BOD (mg/L)	COD (mg/L)	TSS (mg/L)	Absorbency (abs.)
11.10	255.48	268.72	166.31	0.272

Table 3: Effect of temperature on BOD, COD and TSS after treatment

Bacillus Subtilis concentration (ml)	Temperature, T (°C)	BOD (mg/l)	COD (mg/l)	TSS (mg/l)
	20	200.21	158.32	150.09

0.5	25	180.09	153.21	133.91
	34.5	151.32	149.33	121.43
	40	190.34	155.77	140.11
	45	250.01	160.05	155.62
1.5	20	180.08	144.23	132.11
	25	120.43	120.08	100.19
	34.5	103.44	98.76	88.92
	40	150.31	131.09	111.44
	45	170.11	148.23	140.05
2.5	20	145.24	100.09	101.32
	25	121.04	88.34	88.34
	34.5	75.82	64.52	65.43
	40	110.88	90.78	90.08
	45	130.21	109.32	110.44
3.5	20	105.22	88.98	93.56
	25	87.03	56.84	77.98
	34.5	49.21	34.33	41.22
	40	77.65	63.68	80.08
	45	110.66	105.53	97.76
4.5	20	90.45	65.34	77.09
	25	67.89	34.21	46.87
	34.5	28.77	11.71	26.51
	40	55.44	37.54	50.78
	45	88.43	70.22	88.06

Table 4: Effects temperature on effluents obtained after treatment

Concentration (ml)	P ^H	BOD (mg/l)	COD (mg/l)	TSS (mg/l)
20	10.	151.32	149.33	121.43
	7			

25	10.1	103.44	98.76	88.92
	1			
34.5	7.3	28.77	11.71	26.51
40	8.1	49.21	34.33	41.22
45	9.4	75.82	64.52	65.43

Table 5: Absorbency rate of the treated effluent with varying bio-treated time (Contact period)

Incubation Period (hour)	Absorbency (515nm) abs.
0	0.272
1	0.202
7	0.112
24	0.039
40	0.002

Table 6: Percent reduction of effluent obtained after treatment with bacillus subtilis

Concentration (ml)	p ^H	% BOD	% COD	% TSS
0.5	10.7	37.33	40.12	34.32
1.5	10.1	44.08	47.82	40.08
2.5	9.4	54.73	57.39	51.23
3.5	8.0	69.83	74.52	65.09
4.5	7.3	81.08	84.85	75.20

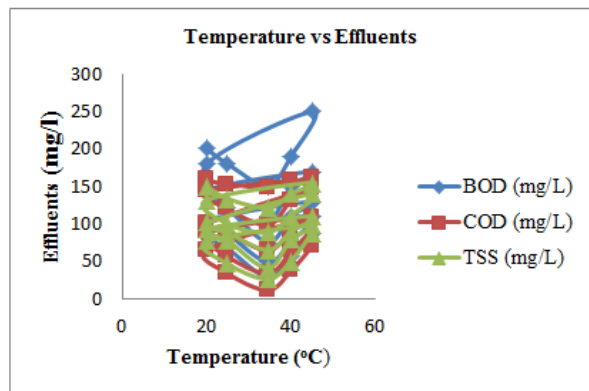


Figure 1: Effect of temperature on the treated effluents with different amounts of Bacillus Subtilis

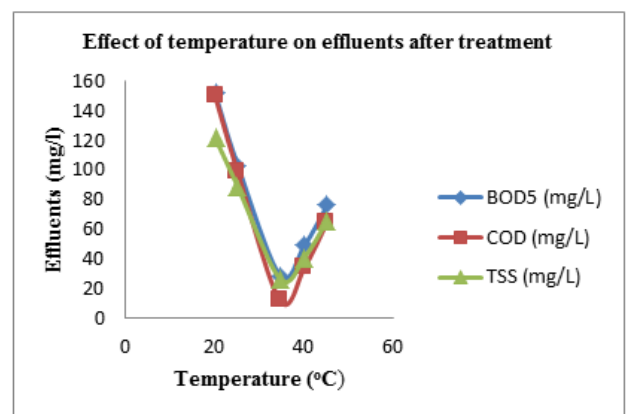


Figure 2: Optimum operating condition of the treated effluents with temperature

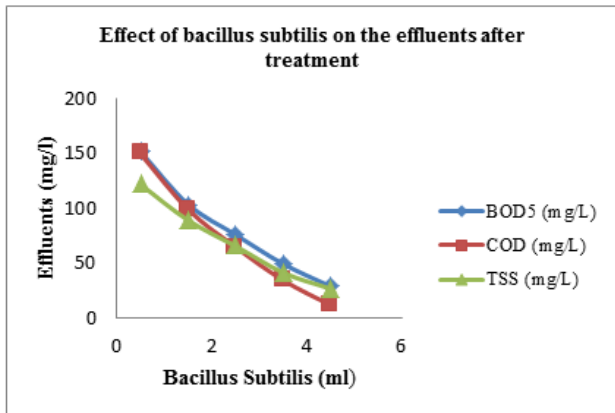


Figure 3: Effect of the bacterium (Bacillus Subtilis) on the treated effluents.

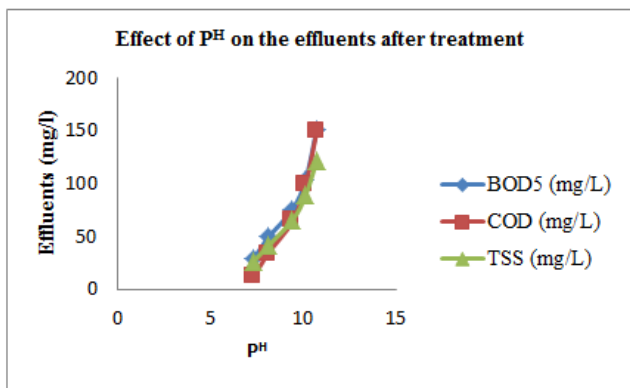


Figure 4: Effect of PH on the treated effluents with Bacillus Subtilis

DISCUSSION

Effect of Different Operation Variables on BOD, COD, TSS, PH Colour Quality and Temperature

Table 2 shows the result of the characterization of effluent wastewater. The BOD load obtained was 255.48 mg/l; COD was 268.72 mg/l; TSS was 166.31 mg/l and the P^H was 10.7 which were very high compared to FEPA and LASEPA standards. Absorbency rate of 0.272 Abs. obtained before treatment was also very high due to the presence of high concentration of organic matter in the wastewater. In Table 3 and Table 4, the optimum operating treatment variables were obtained as thus:

P^H of 7.3,

BOD of 28.77 mg/l,

COD of 11.71 mg/l,

TSS of 26.51 mg/l,

Temperature of 34.5°C,

Contact period of 40 hours.

Table 5 shows the absorbency rate of the treated effluent with varying bio-treated period (contact time). The increase in the contact time between the microorganism and the wastewater sample leads to a corresponding decrease in the absorbency rate which implies that more light passes through the wastewater as bio-treated period increases. Table 6 shows the quality of

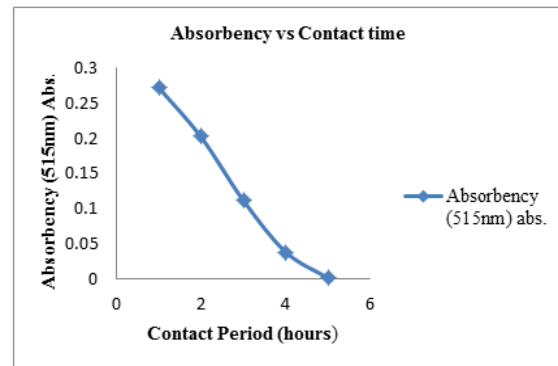


Figure 5: Absorbency rate as a function of Incubation Period.

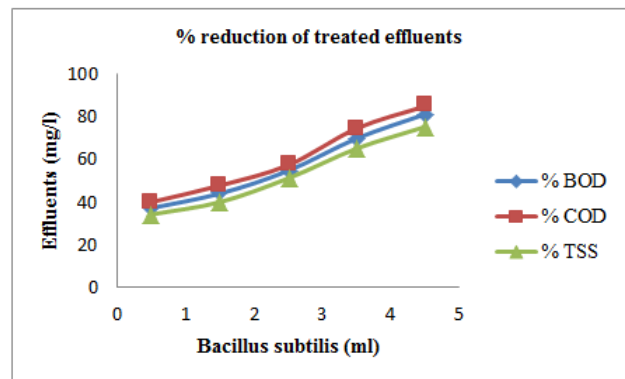


Figure 6: Percent reduction of the treated effluents with Bacillus Subtilis

effluents obtained after treating the sample with the bacterium bacillus subtilis. The percent reduction of the operating variables gives the efficiency of the treatment. The greatest efficiency was obtained at a concentration of 4.5 ml of the microorganism at a contact time of 40 hours.

Figure 1 shows the effect of temperature on the treated effluents with different amounts of Bacillus Subtilis. In Figure 2, the optimum operating conditions of the treated effluents with temperature were attained. It is clear that, the BOD, COD and TSS decreases with increasing temperature down to a value of 28.77 mg/l, 11.71 mg/l, and 26.51 mg/l respectively at 34.5°C and bio-treated period of 40 hours. Beyond this temperature, the BOD, COD and TSS values increases. Figure 3 shows the influence of the bacterium (Bacillus Subtilis) on the treated effluents. The introduction of microorganism helps to reduce the BOD, COD and TSS load, thereby reducing the amount of oxygen consumed by the bacterium (Bacillus Subtilis). The increased in the concentrations of the microorganism used for the treatment leads to a corresponding decrease in the measured operating variables. The effect of P^H on the treated effluents with Bacillus Subtilis was shown in Figure 4. Though the microorganism has little effect on the P^H of the treated effluents, but the P^H tends to decrease with decrease in the concentrations of the operating variables load.

The optimum operating P^H for the treated effluents occurred at 7.3 at an operating temperature of 34.5°C. A study of the graph in **Figure 5** obtained from **Table 5** shows the relationship between absorbency rate and contact period. The increased in the contact time between the microorganism and the wastewater effluents led to a corresponding decreased in the amount of light absorbed by the wastewater. The exponential decreased in the absorbency rate with respect to an increased contact time was due to the reductions in the concentrations of the organic waste in the treated effluents. Figure 4.6 shows that at a concentration of 4.5 ml of bacillus subtilis the most effective treatment of the wastewater was achieved and the lowest efficiency was achieved when the wastewater was treated with 0.5 ml of the bacterium (Bacillus Subtilis). Thus, at higher concentration of the microorganism, a greater percentage reduction of the aqueous wastewater effluents will be achieved.

CONCLUSION

The generated wastes and effluents from various textile industries can be best minimized by: avoiding wastes, reducing waste at the source of generation, recycling waste, and treating waste or effluents with bacillus subtilis to the acceptable discharge permits. From this study, the optimum conditions for biochemical treatment of aqueous wastewater effluent from our textile mill for the case under study were: Temperature, 34.5°C; bio-treated period, 40 hours; and effluents concentrations of 28.77 mg/l BOD, 11.71 mg/l COD and 26.51 mg/l TSS. Only bacteria and protozoa were found as microorganisms in the aqueous wastewater, because the alkaline condition and un-aeration are not suitable for the living of fungi.

NOMENCLATURE

BOD	Biological Oxygen Demand (mg/l)
COD	Chemical Oxygen Demand (mg/l)
TSS	Total Suspended Solids (mg/l)
%BOD	Percent Biological Oxygen Demand
%COD	Percent Chemical Oxygen Demand
%TSS	Percent Total Suspended Solids
FAS	Ferrous Ammonium Sulphate
P^H	Hydrogen ion concentration
FEPA	Federal Environmental Protection Agency
LASEPA	Lagos State Environmental Protection Agency
ASP	Activated Sludge Process
UWWT	Urban Waste Water Treatment

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