Characterization of Effluent from Textile Wet Finishing Operations

Freeman Ntuli, *Member*, *IAENG*, Daniel Ikhu-Omoregbe, Pardon K. Kuipa, Edison Muzenda and Mohamed Belaid

Abstract— **The physico-chemical characteristics of effluent from textile wet finishing operations processing denim and other textile fabrics were evaluated. The effluent was characterized in terms of its organic and inorganic pollutant loading and amenability to biodegradation. The major pollution indicator parameters were the chemical oxygen demand (COD), biochemical oxygen demand (BOD), total dissolved solids (TDS), suspended solids (SS), colour and heavy metals levels. The effluent was highly turbid and coloured with average organic and inorganic loadings of 1766 kg COD.day^{-1} and 964 kg TDS.day^{-1} respectively. Overall mean COD, TDS and SS levels of the effluent were 5849 mgl^{-1}, 3193 mgl^{-1} and 521 mgl^{-1} respectively. Cu, Fe and Cr (VI) levels were less than 4 mgL^{-1}. Analysis of the BOD curves revealed an initial lag in microbial activity of between 1-2 days indicating the presence of non-persistent toxic substances that affect microbial activity. After the initial lag an average BOD rate constant (K_L) value of 0.55 day^{-1} was obtained, which was above the acceptable sewer discharge limit of 0.17 day^{-1}. BOD_5:COD ratios ranged from 0.2-0.5 indicating that the effluent contained a large proportion of non-biodegradable organic matter. The COD and TDS levels were above the generally accepted levels for discharge into municipality sewers. Even though the concentrations of these pollutants were not far above accepted limits, high pollutant mass loadings were obtained due to high discharge volumes. Based on its overall characteristics, the effluent was considered not to be suitable for discharge into municipality sewers without pretreatment. Thus, a pre-treatment step involving coagulation and flocculation coupled with carbon adsorption was recommended.**

Index Terms*— *textile, wet processing, effluent characterization, effluent pre-treatment

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F. Ntuli is with the Chemical Engineering Department, University of Johannesburg, P.O. Box 17011, Doornfontein 2028, Johannesburg, RSA (corresponding author phone: +27-11-559 6003; fax: +27-11-559 6430; e-mail: fntuli@uj.ac.za).

D. Ikhu-Omoregbe is with the Chemical Engineering Department, Cape Peninsular University of Technology, Cape Town, RSA (e-mail: ikhuomoregbed@cput.ac.za).

P. K. Kuipa is with the Chemical Engineering Department, National University of Science and Technology, Bulawayo, Zimbabwe (e-mail: pkkuipa@nust.ac.za).

E. Muzenda is with the Chemical Engineering Department, University of Johannesburg, P.O. Box 17011, Doornfontein 2028, Johannesburg, RSA (e-mail: emuzenda@uj.ac.za).

M. Belaid is with the Chemical Engineering Department, University of Johannesburg, P.O. Box 17011, Doornfontein 2028, Johannesburg, RSA (e-mail: mbelaid@uj.ac.za).

I. INTRODUCTION

Textile finishing processes involve a series of wash treatments designed to remove impurities and impart to the material desired properties of aesthetic appeal and touch. The textile wet finishing processes considered were denim wet processing, garment wash and fabric dyeing.

The major processing steps on the wet processing of denim garments involve desizing, stone-washing, bleaching and neutralization, and fabric softening. Desizing involves the removal of starch based sizes added during fibre processing by treating the denim with commercial α-amylase enzymes. Stone-washing is a more severe form of cellulase treatment which is essentially a degradative mechanism resulting in the loss of both the weight and strength of the fabric giving the material a worn-out appearance. Stone-washing with enzymes generally referred to as bio-stoning has gained popularity due to the reduced ecological problems normally encountered when stones are used [1]. Bleaching and neutralization is normally conducted to remove unwanted colour in preparation for dyeing.

Fabric dyeing involves the following major steps; scouring, bleaching, dyeing, dye fixation and fabric softening. Scouring is performed to remove impurities through the use of alkaline baths prior to further fabric wet processing. Garment washing involves the use of detergents and softeners to remove dirt and improve the fabric texture before finished garments are sent to the market.

All the three textile finishing processes are water-intensive requiring large volumes of water for processing and rinsing [2]. Furthermore, a wide variety of chemicals, detergents and softeners are also employed to improve the efficiency of each process. Since the processing and rinsing steps are conducted as batch operations and there are stringent water quality requirements for each processing step, water used is normally used once for each processing step or rinse before being discharged. This greatly increases the discharge volume and fresh water requirements for the wet processes. Most textile finishing plants in Southern Africa discharge their effluent to municipality sewers for further treatment in public owned treatment works (POTW). Since most POTW were designed to handle domestic effluent, industrial dischargers are normally required to meet effluent discharge limits stipulated by the municipality. However, most municipalities use concentration based standards and rarely consider the pollutant loading of the effluent. Furthermore, only aggregate quality parameters e.g. COD, SS and pH are only considered thus the overall impact of the effluent on the treatment works is normally overlooked. As a result, incidence of water pollution by effluent discharges from POTW have been increasing in the Southern African region due to lower treatment efficiencies of POTW as a result of failure to adequately handle mixed domestic and industrial effluent. Corrosion of sewer lines conveying industrial waste has also been noted in a number of municipalities resulting in an increase in the incidence of groundwater pollution and large capital expenses incurred by municipalities in replacing and repairing the sewer lines. Thus, it is important to fully characterize the effluent from the major industrial dischargers in order to mitigate these problems and to enable recommendations for pre-treatment requirements.

Previous studies have shown that the major effluent pollution indicator parameters of concern in textile effluents were the COD, colour, toxicity and salinity [2]-[4]. Due to the semi-arid nature of the Southern African region, opportunities for water recycling and re-use need to be explored to reduce water consumption in this industrial sector if sustainable use of water resources is to be achieved.

II. EXPERIMENTAL

A. Sampling

The sampling programme was divided into two phases. Manual and systematic sampling was employed to collect the effluent samples. The first phase was conducted over a period of two months. Snap samples were collected over an 8-hour day shift after an interval of 8 days. Five snap samples were collected on each sampling day and these were combined to form a time-interval composite sample. The objective of this phase was to build an understanding of the variation of the effluent quality over a shift and thus provide data for the statistical design of the sampling programme.

Samples were collected in glass containers because of the presence of oils and grease in the effluent. 1.5 L of effluent was collected for each snap sample. Samples were collected from the plant outlet to the sewer line after going through a screen (mesh size 1 mm).

B. Sample preservation prior to analysis

Samples were preserved by refrigeration at 4^oC without chemical addition for all the parameters measured except for the heavy metals. In the case of heavy metals nitric acid was used to lower the pH to a value less than 2 before refrigeration. Parameters such as temperature, TDS, conductivity and pH were determined soon after sampling.

C. Analysis of samples

Samples were analysed within 24 hrs of collection. Standard methods as outlined in the Standard Methods for the Examination of Water and Wastewater [5] were used to analyse the samples. The pH of the effluent was measured using a Hach pH meter (Model 51935-00), while the conductivity, TDS, salinity and temperature were measured using a Hach conductivity meter (Model 51975-03) fitted with a temperature sensor. Chemical oxygen demand (COD) was determined by closed reflux method using a Hach COD reactor (Model 45600) followed by calorimetric determination of Cr^{3+} at a wavelength of 620 nm using a Hach spectrophotometer (Model DR 2010). Chloride interference was eliminated by the use of mercuric nitrate as a complexing agent. The respirometric method was used to determine the effluent 5 day-Biochemical Oxygen Demand (BOD) using a manometric respirometer (Hach BOD trak, Model 26197-01). Total phosphates were determined by cadmium and ascorbic acid reduction respectively, followed by calorimetric determination on a Hach spectrophotometer. Determination of chlorides was done by titration using mercuric chloride with a mixture of diphenylcarbazone and bromophenol blue as indicators. Sulphates were determined by measuring the barium sulphate turbidity at 450 nm using a Hach spectrophotometer.

D. Analysis of the BOD data

The BOD rate constant (K_L) was calculated from the respirometric data using the Thomas graphical method based on (1), where L is the BOD at time t [6]. The K_L (quoted to the base e) was calculated from the slope and intercept of the plot of $(t/y)^{1/3}$ against t using (2).

$$\left(\frac{t}{y}\right)^{1/3} = \left(2.3K_L L\right)^{-1/3} + \frac{K_L^{\ 2/3}}{3.43L^{1/3}}.t \quad (1)$$

$$K_L = 6.01\times\left(\frac{\text{slope}}{\text{intercept}}\right) \quad (2)$$

E. Flow measurements

The volumetric flow rate was measured on a rectangular channel discharging to the POTW using a Millitronics open channel monitor (OCM III, Model PL-505). The monitor was used in conjuction with a remote ultrasonic transducer (Model ST-25) with an inbuilt temperature sensor. The transducer was mounted over a sewer manhole and the volumetric flow rate was continuously monitored over a period of seven days. Flow rates (Q) were calculated by the monitor from the head measurements (h) using the ratiometric method (equation 3). Q_{cal} and h_{cal} were the flow rates and head at maximum flow which were 0.063 m^3/s and 0.25 m respectively.

$$Q = Q_{cal}\times f(h)/f(h_{cal}) \quad (3)$$

III. RESULTS AND DISCUSSIONS

A. Effluent characteristics from the three wet finishing operations

The quality of the effluent generated from each of the three major wet finishing operations namely; denim wet processing, garment wash and fabric dyeing, were characterized using pollution indicator parameters shown in Table I. The results were obtained from composite samples prepared by combining snap samples collected from each processing stage within each wet finishing operation. Thus, the results reflect the general composition of the mixed effluent stream from a particular wet finishing operation.

From an eco-toxicological point of view the major process streams of major concern in textile finishing processing are those from the scouring, bio-stoning and dyeing operations.

The effluent from all the three wet processing operations were characterized by high COD levels indicating the presence of a significant amount of organic material.

Table I.

Effluent characteristics from denim wet processing, garment washing and fabric dyeing.

Parameters	Wet processing operations		
(mgl^{-1} unless stated)	Denim wet processing	Garment wash	Fabric dyeing
COD	10,980	6470	14,480
SS	625	95	221
Total alkalinity	85	1585	3275
Chloride	680	4	11,900
Sulphate	70	30	140
Phosphates	13	2	32

The COD levels obtained from garment washing show that detergents, softeners and impurities on the fabrics contributes a significant portion of the COD. Highest COD levels were obtained on dyeing indicating that in addition to fabric impurities removed during scouring or desizing and the contribution of detergents and softeners, residual dyes contributed a large proportion of the COD. SS levels were highest on denim wet processing largely due to solid material removed during bio-stoning. Chloride levels were extremely high for fabric dyeing operations due high salt levels used to enhance dye exhaustion. Sulphate and phosphate levels were low in all the three washes. Since the samples were not digested prior to phosphate measurement, the result only indicates phosphates that are present as reactive ortho-phosphate. Total alkalinity was highest on the effluent from fabric dyeing indicating that the effluent was alkaline (average pH>10) and lowest for denim processing indicating an acidic effluent. High total alkalinity levels obtained for fabric dyeing and garment wash effluents are a result of the use of sodium carbonate as a scouring agent and caustic soda for the saponification of waxes, oils and soils during fabric washing operations. The low alkalinity obtained for denim wet processing effluent is largely due to acidic conditions employed during bio-stoning.

From the results above it can be concluded that aside from the COD levels, effluent from garment washing meets the generally accepted standards of discharge into most municipality sewers. For denim wet processing and fabric dyeing, pre-treatment is required to reduce the COD, SS (for denim wet processing) and chloride levels if such effluents are to be discharged to sewer. In addition since both effluents streams are coloured, colour removal will also be required.

The effluent characterization results presented thereafter are representative of the mixed effluent stream from all the three wet operations since the above processes are normally conducted concurrently.

B. *Effluent characterization results*

The characteristics of the effluent from the processing plant during the first phase of monitoring are shown in Table II. The wide variation in effluent quality reflects the wide variation in effluent characteristics obtained from each characteristic stage within a wet processing operation. Since the results were obtained by analyzing snap samples they are representative of the effluent quality at the time of sampling, thus they reflect effluent discharge from different wet processing stages.

Table II

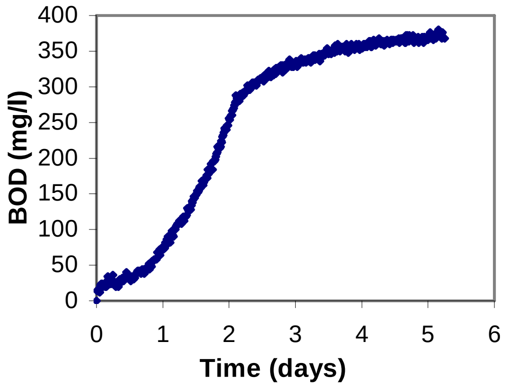
Effluent characteristics during the monitoring period

Parameters, mgl^{-1} unless stated	Range of values obtained	Average
pH (pH units)±0.20	5.2-11.8	8.8
Temperature (^{0}C) ±1.0	30.0-60.0	41.7
SS±10	40-3840	521
COD±26	3700-10,200	5849
BOD±10	770-5000	2040
Total Alkalinity±5	35-19,800	1590
Turbidity (FAU) ±49	40-4600	451
Conductivity (µS/cm) ±10	400-40,200	6385
TDS±5	170-20,100	3193
Phosphates±0.1	2.0-305.0	145.0
Chloride±1	20-11,800	879
Hardness±0.3	85-150	113
Iron±0.1	<4	2.6
Copper±0.1	<1	0.3
Chromium (VI) ±0.1	<1	0.1
Sulphates±1	20-180	133

The COD levels were in most instances above 4500 mgl^{-1} and generally well above the discharge limit of 2000 mgl^{-1}. SS levels were below the discharge limit of 600 mgl^{-1} for 73% of the snap samples collected, however, SS levels in excess of 1000 mgl^{-1} were noted in 18% of the snap samples collected. Chloride levels were above the discharge limit of 500 mgl^{-1} for 38% of the snap samples, however, in such cases the chloride levels were above 2000 mgl^{-1} and one snap sample had a chloride concentration of 11,800 mgl^{-1}. TDS levels were above the discharge limit of 2000 mgl^{-1} in 50% of the snap samples collected. The effluent temperature was above the discharge limit of 45 ^{o}C in 23% of the snap samples collected. Phosphate levels obtained were much higher than those shown in Table I, since the samples were digested prior to analysis. This indicates that most of the phosphates were present in their condensed inorganic form thus needed to be hydrolyzed under acidic conditions to convert them to orthophosphates, which are measured by the method of analysis. The phosphate levels were above the discharge limit of 25 mgl^{-1} in 77% of the snap samples collected. The phosphates are mainly from the detergents used during wash operations and act as water softeners or detergent builders.
The ratio of the BOD_5:COD obtained from the results ranged from 0.2-0.5. This indicated that the effluent contained a

large proportion of non-biodegradable organic matter. A BOD_5:COD ratio less than 0.5 indicates that the effluent contains a large proportion of non-biodegradable matter [7]. A typical BOD curve of the effluent from the textile wet processing plant is shown in Fig. 1(a). Analysis of the BOD curve shows an initial lag revealing that the microorganisms took time to be acclimatized to the waste. After the initial lag the curve reveals two distinct phases between day 1-2, and day 2-5 each with a different slope. This shows that the effluent consists of different types of organic constituents differing in the rate and ease of biodegradability. An analysis of the curve using the Thomas slope method (1) gave a negative slope for the first segment (day 1-2) of the curve indicating the presence of toxic material interfering with the BOD test. Analysis of the second segment of the curve (day 2-5) using the Thomas slope plot (Fig. 1(b)) gave a K_L value of 0.55 day^{-1}. This reveals that a certain fraction of the waste is readily biodegradable. However, the source of toxic contaminants was not identified. Identification of toxic components in textile processes is a lengthy process and has proved problematic due to the wide variety of factors involved, thus it was not considered at this stage of the work [8].

(a)



(b)

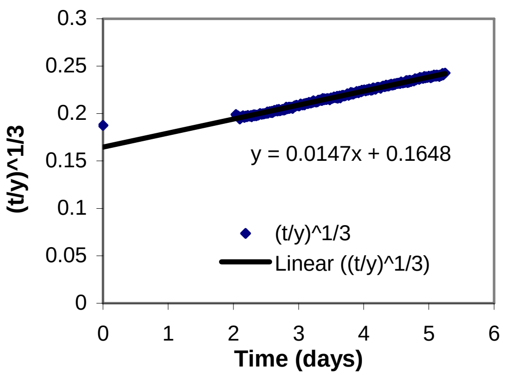


Fig. 1. BOD curve (a) and Thomas slope plot (b) for the textile effluent

The other remaining pollution indicator parameters were all below the discharge limit, with the exception of turbidity where no sewer discharge limit guidelines was available in the region.

Overall, the effluent was characterized by high phosphates, TDS and COD levels and thus was considered not to be suitable for discharge to sewer without pre-treatment. The presence of toxic substances that affect the amenability of the effluent to biological treatment and the high proportion of non-biodegradable substances will all have a negative impact on the performance of POTW. Though the effluent had high COD levels, sulphate levels were low, thus it is unlikely that such effluents can result in the corrosion of sewer lines even if the residence time was long. High organic levels in the presence of sulphate levels above 1400 mgl^{-1} coupled with long residence times in the sewer have been identified as the major cause of severe corrosion of concrete sewers made from ordinary Portland cement [9]. This phenomenon occurs due to the oxidation of sulphates to sulphides under anaerobic conditions, as a result of dissolved oxygen depletion by high oxygen demanding effluents.

C. Effluent quantification

The flow monitoring results obtained by measuring the flowrate over a period of seven days is shown in Fig. 2.

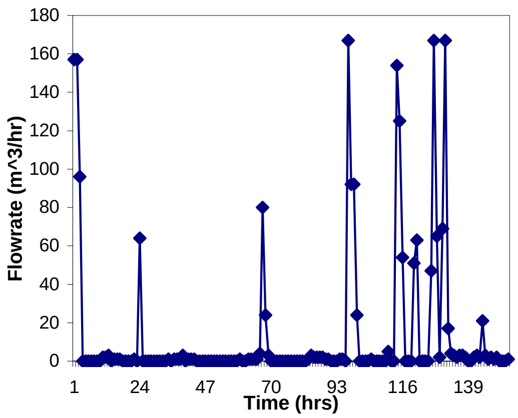


Fig. 2. Flow monitoring results

An analysis of Fig. 2 shows that the flow is intermittent and characteristic of batch processes. The high peak discharges are typical of the textile wet processing operations, which are normally characterized by the simultaneous discharge of the effluent from the tumble-washers. Since the textile plant operated for 24 hr, peak discharges seem to be randomly distributed with the majority of the peak discharges occurring towards or after midnight (i.e. between 11:00pm and 2am).

The total volume of effluent discharged per day is shown in Table III. There was a wide between-day variation (i.e. ±313) in the daily total volume discharged. The volume discharged depends on the number of wet operations conducted and varies according to the number of batches being processed.

Table III

Total volume of effluent discharged per day

Day	1	2	3	4	5	6	7
Effluent volume (m^3)	442	27	124	83	764	651	21

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The average, maximum and minimum inorganic and organic pollution loads for the textile wet processing plant are shown in Table IV. There was a wide degree of variation in the pollutant load and the variation in the load has been shown to resemble that of the flowrate [10]. Even though the concentration of the pollutants were not extremely high (Table II), the mass of pollutants leaving the plant were high largely because of the high volumes of discharge (Table III). Since the effluent was characterised by intermittent flows the discharge of this effluent to sewer without any pre-treatment is likely to result in shock loads. This is likely to interfere with the treatment processes and cause pass-through of pollutants especially colour and chloride since biological processes employed in POTW do not remove these pollutants [11], [12]. However, since dilution and equalisation of effluent occurs naturally in larger collection systems these effects on the POTW are likely to be minimised.

Table IV
Maximum, minimum and average pollution loads from the textile wet finishing plant

Parameter	Pollutant load		
	Maximum	Minimum	Average
Inorganic load ($kg.day^{-1}$)	15,356	5	964
Organic load ($kg\ COD.day^{-1}$)	7793	100	1766

D. Pre-treatment requirements

Based on the quality characteristics of the effluent the pre-treatment requirements should seek to reduce the COD, TDS, phosphates levels and remove colour. However, the best practicable technology (BPT) should be adopted for this sector of industrial if their economic viability is not to be compromised. This approach has been found to be more suitable for developing nations than the best available technology (BAT) approach [13]. Taking the above factors into consideration, a typical textile plant for such operations is shown in Fig. 3. The effluent is normally passed through a bar screen before sampling in order to remove large pieces of cloth that might clog the sewer line, however, the fine SS pass through the screen. Since these fine SS are organic in nature they greatly contribute to the COD of the effluent. Thus, the use of fine screens is also recommended to improve the SS removal efficiency and reduce the COD load. These screens have been successfully employed in some textile industries, however, their solid removal efficiency is low (10-20%) [14]. Thus, the use of sedimentation coupled with the use of coagulants and flocculants which has a removal efficiency of around 60% is recommended [14]. In addition, current research work by the authors revealed that the use of coagulation and flocculation can significantly reduce the total phosphate content of the effluent. This also further reduces the COD and colour of the effluent by removing colloidal organic material. Coagulation and flocculation coupled with adsorption on activated powdered carbon has been shown to result in COD removal of more than 80% and colour removal of 50% [3]. Since the flow is intermittent, equalization is recommended to reduce the effect of shock loads to the treatment unit and POTW. For further colour removal, the use of activated carbon adsorption is recommended on economic grounds. Other methods of colour removal e.g. ozonation, fenton oxidation and membrane techniques are more effective but more expensive [15]-[17]. The use of adsoption besides removing the colour has an added advantage of removing some of the dissolved organic material and toxic compounds since these can also be adsorbed on the carbon particles. Previous studies have shown that biological treatment employed in POTW effectively removes the particulate COD fraction and 73% of the COD remaining after biological treatment is the soluble COD fraction [18]. The soluble COD fraction was also found to constitute 40% of the colour, thus reducing the soluble COD fraction is essentially before discharging to sewer. Reduction of TDS levels can not be effectively achieved using the proposed treatment plant, only advanced treatment methods e.g. membrane techniques can adequately reduce the TDS levels. Since most of the dissolved salts are generated from the dyeing process, the use of cleaner technology to reduce the amount of salt used in the dyeing process is recommended. Four main ways of reducing the salt load in the effluent that have been suggested in literature include lowering the liquor ratio, avoiding higher temperatures, optimising the dye recipe and the development of novel dyes [19]. All these approaches seek to minimise the salt load by adjusting dyeing parameters so that the lowest possible concentration of salt is added during the dyeing stage.

IV. CONCLUSION

Effluents from textile finishing plants conducting three major operations namely; denim wet processing, garment wash and fabric dyeing were characterized by average COD and TDS levels of 5849 mgl^{-1} and 3193 mgl^{-1} respectively, which were above generally accepted sewer discharge limits. A large proportion of the organic material was generated from the dyeing and denim wet processing operations while a large proportion of dissolved salts came from fabric dyeing operations. SS and phosphate levels were above the sewer discharge limits of 600 mgl^{-1} and 25 mgl^{-1} for 73 % and 77 % of the snap samples collected respectively. The BOD_5:COD ratio ranged from 0.2-0.5 indicating that the effluent contained a large proportion of non-biodegradable organic matter. Analysis of the BOD curves using the Thomas slope graphical method revealed an initial lag of 1-2 days, thereafter an average BOD rate constant (K_L) value of 0.55 day^{-1} was obtained. This value was above the acceptable sewer discharge limit of 0.17 day^{-1} [7]. After the initial lag the first segment of the Thomas slope plot gave a negative slope indicating the presence of toxic material interfering with the BOD test.

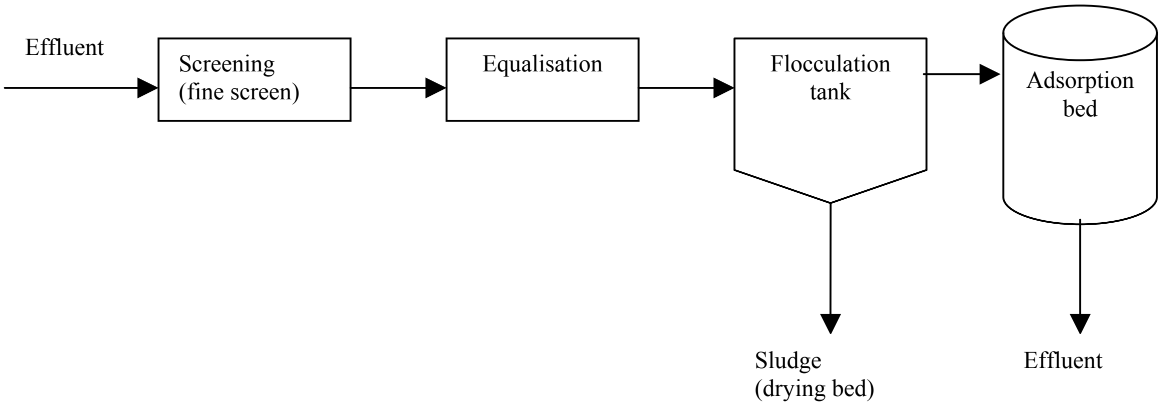

Fig. 3. Proposed effluent treatment plant for textile wet finishing operations discharging to sewers

Flow monitoring results revealed that the flow was intermittent and the average organic and inorganic pollutant loads were 1766 kg COD.day^{-1} and 964 kg TDS.day^{-1} respectively. Even though the concentrations of the pollutants were not extremely high the mass of pollutants leaving the plant were high largely because of the high volumes of discharge.

Overall, the effluent was considered not suitable for discharge into POTW without pretreatment to reduce the TDS and COD levels, removal of toxic material and colour. Though the effluent had high COD levels, sulphate levels were below 208 mgl^{-1}, thus it is unlikely that such effluents can result in the corrosion of sewer lines even if the sewer residence time was long. Based on the effluent quality and economic factors, the best practicable pre-treatment option recommended was coagulation and flocculation coupled with carbon adsorption. This treatment option has been demonstrated to achieve COD and colour removal efficiencies of around 80 % and 50 % respectively.

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