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Review

Effluent treatment process in molasses-based distillery industries: A review

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Abstract

Distillery effluent is a contaminated stream with high chemical oxygen demand (COD) varying from 45,000 to 75,000 mg/l and low pH values of between 4.3 and 5.3. Different processes covering aerobic, anaerobic as well as physico-chemical methods which have been employed to this effluent has been given in this review paper. Among the different methods available, it was found that "An Inverse Anaerobic Fluidization" to be a better choice for treating effluent from molasses-based distillery industries using an inverse anaerobic fluidized-bed reactor (IAFBR). This technology has been widely applied as an effective step in removing 80–85% of the COD in the effluent stream. Therefore, in this review, attention has been paid to highlight in respect of fluidization phenomena, process performance, stability of the system, operating parameters, configuration of inverse anaerobic fluidization and suitable carrier material employed in an inverse anaerobic fluidized-bed reactor especially for treating this effluent.

Keywords: Molasses; Spentwash; Treatment Methods; Anaerobic fluidized-bed reactor; Perlite; Inverse anaerobic fluidized-bed reactor

Contents

1. Introduction

In India, there are a number of large-scale distilleries integrated with sugar mills. The waste products from sugar mill comprise bagasse (residue from sugarcane crushing), pressmud (mud

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and dirt residue from juice clarification) and molasses (final residue from sugar crystallization). Bagasse is used in paper manufacturing and also as a fuel in boilers. Molasses is utilized as a raw material in distillery for ethanol production. Pressmud has no direct industrial application. Ethanol manufactured from molasses has large volumes of high strength wastewater that is of serious environmental concern. The effluent is characterized by extremely high COD (80,000–100,000 mg/l) and biochemical oxygen demand (BOD) (40,000–50,000 mg/l), apart from low pH, strong odor and dark brown color (Central Pollution Control Board (CPCB) 1994, 2003, India). India is the second largest producer of ethanol in Asia with a projected annual production of about 2300 million litres in 2006–2007 [1], alcohol distilleries have been rated as one of the 17 most polluting industries. Apart from high organic content, distillery wastewater also contains nutrients in the form of nitrogen (1660–4200 mg/l), phosphorous (225–3038 mg/l) and potassium (9600–17,475 mg/l) [2] that could lead to eutrophication of water bodies. Further, its dark color hinders photosynthesis by blocking sunlight and therefore deleterious to aquatic life [3]. The population equivalent of distillery wastewater based on BOD has been reported to be as high as 6.2 billion which means that the contribution of distillery waste in India to organic pollution is approximately seven times more than the entire Indian population. The wastewater from distilleries, major portion of which is spentwash, is nearly 15 times the total alcohol production. This massive quantity, approximately 40 billion liters of effluent, if disposed untreated would cause considerable stress on the watercourses leading to widespread damage to aquatic life. Adequate treatment is therefore imperative before the effluent is discharged. In the present day context stringent statutory requirements have made the distilleries to improve the quality level of their effluent by exploring viable alternatives. For instance, Indian distilleries were stipulated to achieve zero discharge of spentwash to inland surface water by December 2005 [4].

Production of ethyl alcohol in distilleries based on cane sugar molasses constitutes a major industry in Asia and South America. The world's total production of alcohol from cane molasses is more than 13 million m^3/annum. The aqueous distillery effluent stream known as spentwash is a dark brown, highly organic effluent and is approximately 12–15 times by volume of the product alcohol. It is one of the most complex, troublesome and strongest organic industrial effluents, having extremely high COD and BOD values. Because of the high concentration of organic load, distillery spentwash is a potential source of renewable energy.

The wastewater from distilleries, major portion of which is spentwash, is nearly 15 times the total alcohol production. This massive quantity, approximately 40 billion liters of effluent, if disposed untreated could cause considerable stress on the watercourses leading to widespread damage to aquatic life.

In an earlier review on this subject [5], discussed treatment options practised in the 1970s and more recently [6], have described the characteristics and anaerobic treatment of effluent obtained from different feedstock used for ethanol production. The wastewater characterization and salient features of the existing treatment options and the recent advancement in the field of an inverse anaerobic fluidization [7] have been discussed in the following sections.

2. Wastewater characterization

Table 1 lists out the major wastewater streams generated at different stages of the alcohol manufacturing process. Table 2 summarizes the typical characteristics of spentwash generated in Indian distilleries using sugarcane molasses. Table 3 describes the IS standards of distillery effluent. Values for beet molasses-based effluent have been given for comparison. The main source of wastewater generation was the distillation step wherein large volumes of dark brown effluent (termed as spentwash, stillage, slop or vinasse) were generated in the temperature range of 71–81 °C [8–10]. The characteristics of the spentwash depended on the raw material used [11]; also, it has been estimated that 88% of the molasses constituents end up as waste [12]. Molasses spentwash has had very high levels of BOD, COD, COD/BOD ratio as well as high potassium, phosphorus and sulfate content (Table 2). In addition, cane molasses spentwash contains low molecular weight compounds such as lactic acid, glycerol, ethanol and acetic acid [6]. Cane molasses also contained around 2% of a dark brown pigment called melanoidins that imparted color to the spentwash [13]. This reaction proceeded effectively at temperatures above 50 °C and pH 4–7. The structure of melanoidins was still not well known [14]. Only 6–7% degradation of the melanoidins has been achieved in the conventional anaerobic–aerobic effluent treatment process [15]. Due to their antioxidant properties, melanoidins were toxic to many microorganisms involved in wastewater treatment [16]. Apart from melanoidins, spentwash contained other colorants such as phenolics, caramel and melanin. Phenolics have been more pronounced in cane molasses wastewater whereas melanin was significant in beet molasses [17].

Table 1
Sample quantities and characteristics of wastewater streams generated in an Indian distillery (S. Majumdar, personal communication)

Parameter	Specific wastewater generation (kl/kl alcohol)	Color	pH	Suspended solids (mg/l)	BOD (mg/l)	COD (mg/l)
Spentwash	14.4	Dark brown	4.6	615	36,500	82,080
Fermenter cleaning	0.6	Yellow	3.5	3000	4000	16,500
Fermenter cooling	0.4	Colorless	6.3	220	105	750
Condenser cooling	2.88	Colorless	9.2	400	45	425
Floor wash	0.8	Colorless	7.3	175	100	200
Bottling plant	14	Hazy	7.6	150	10	250
Other	0.8	Pale yellow	8.1	100	30	250

Table 2
Characteristics of spentwash generated from various feedstock [18,6,2]

Characteristics	Feedstock		
	Cane molasses		Beet molasses
	Pathade [18]	Mahimairaja and Bolan [2]	Wilkie et al. [6]
COD (mg/l)	65,000–130,000	104,000–134,400	91,100
BOD (mg/l)	30,000–70,000	46,100–96,000	44,900
COD/BOD ratio	2.49		1.95
Total solids (mg/l)	30,000–100,000		
Total suspended solids (mg/l)	350		
Total dissolved solids (mg/l)	80,000	79,000–87,990	
Total nitrogen (mg/l)	1000–2000	1660–4200	3569
Total phosphorus (mg/l)	800–1200	225–3038	163
Potassium (mg/l)	8000–12,000	9600–17,475	10,030
Sulfur as SO_4 (mg/l)	2000–6000	3240–3425	3716
pH	3–5.4	3.9–4.3	5.35

3. Treatment methods

There are various technologies for treatment of raw spentwash which utilize resource recovery and disposal. The secondary and tertiary treatments on their own are not technically and economically viable options for mitigating the problems associated with treatment and disposal of high-strength spentwash. Post-anaerobic effluent has had high COD, BOD and suspended solids (SS). Solar evaporation required a large land area and was non-functional during the rainy seasons [9]. Two-stage aerobic treatment has high-energy requirements. Partially treated spentwash has had a high (carbon: nitrogen) C:N ratio (>20). It could reduce the availability of important mineral nutrients by trapping them into immobile organic forms, and might produce phytotoxic substances during decomposition. It has been unsuitable for irrigation. Tertiary treatment comprising physico-chemical methods, adsorption and advanced chemical oxidation processes, basically adopted for removal of color in addition to trace organics, involved high operational cost. Stringent regulations on the discharge of colored effluent impeded direct discharge of secondary treated effluent. Evaporation and incineration have not been widely adopted due to their being highly energy intensive. Distillery spentwash also contained high nitrogen, phosphorus and organic content. Its use has been further reported to increase sugarcane productivity; furthermore under controlled conditions, the effluent was capable of replacing application of inorganic fertilizers [19,20]. However, for the high strength molasses-based spentwash, the odor, putrefaction and unpleasant landscape due to unsystematic disposal are concerned in land application. In addition, this option was subject to land availability in the vicinity of the distillery; also, it was essential that the disposal site be located in a low-medium rainfall area [5]. More recent investigations have indicated that land disposal of distillery effluent could lead to groundwater contamination [21]. Deep well disposal was another option but limited underground storage and specific geological location limit this alternative. Other disposal methods like evaporation of spentwash to produce animal feed and incineration of spentwash for potash recovery have also been practised [5,6]. Considering the problems in the treatment and disposal of distillery spentwash on land and into surface water, waste management option with recourse to zero effluent discharge would appear to be more promising. Fig. 1 presents the options currently employed for molasses spentwash treatment.

3.1. Biological treatment methods

3.1.1. Aerobic process

Post-anaerobic effluent has had high BOD, COD and SS. Solar evaporation required a large land area and also needed to take into consideration the weather conditions prevailing in the region. It was also non-functional during the monsoon [9]. The other aerobic methods have been described below.

3.1.1.1. Biocomposting process. Biocomposting is a method of activated bioconversion through the aerobic route, whereby heterotrophic microorganisms act on carbonaceous materials depending on the availability of the organic source and the presence of inorganic material essential for their growth. Composting is particularly effective in converting the wet materials to a usable form thereby stabilizing the organics and destroy-

Table 3
ISI Standards (IS: 506–1980) for limits of composition of distillery effluent

Characteristics	For discharge into water course	For discharge into public sewers	For discharge on land
PH	5.5–9.0	5.5–9.0	5.5–9.0
BOD5 (mg/l)	100	500	500
Total suspended solids (mg/l)	100	600	–
Oil and grease (mg/l)	10	100	6–10
Temperature °C	Shall not exceed 40 °C in any section of the stream within 15 m from effluent outlet		

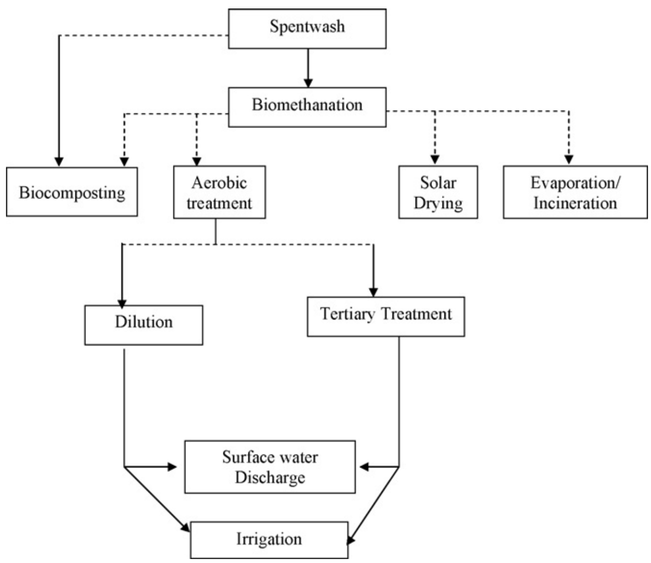

Fig. 1. Spentwash treatment options.

ing the pathogenic organisms in addition to significant drying of the wet substrate. In the composting process, under aerobic conditions, thermophilic biodegradation of organic wastes at 40 ± 60% moisture content occurs to form relatively stable, humus-like material [22]. The process is expressed as:

Organic matter + O_2 + microorganisms → CO_2 + H_2O + Heat

The process steps followed during active composting are as follows:

(1) Windrow making.
(2) Drying to reduce moisture content to [illegible] ± 60%.
(3) Trimming for breaking of lumps.
(4) Aerotilling to loosen the pressed substrate.
(5) Inoculating with microbial culture for faster decomposition and stabilization of organic materials.
(6) Aerotilling for proper mixing, distribution and oxygenation.
(7) Maintaining inoculum by addition of small doses at regular intervals for better performance.
(8) Efficient concentrated spentwash spraying.
(9) Temperature maintained between 50 and 70 °C.
(10) Moisture content maintained in the range of 40 ± 60%.
(11) Aeration carried out after each concentrated spentwash application in the following sequence: concentrated spraying → trimming → aerotilling to attain the oxygen concentration of more than 10%.
(12) Climatic conditions including temperature, wind velocity and rainfall taken into the consideration.
(13) Improper composting encourages breeding of flies, which became a nuisance. Fly control achieved through grinding, turning and heating effect in proper decomposing conditions.
(14) Nitrogen loss in the form of ammonia in anaerobic conditions prevented by maintaining proper initial C:N ratio (range 38 ± 40), pH, moisture content and oxygen concentration.

3.1.1.2. Activated sludge process. The most common post-biomethanation step is the activated sludge process wherein research efforts are targeted at improvements in the reactor configuration and performance. For instance, aerobic sequencing batch reactor (SBR) is reported to be a promising solution for the treatment of effluents originating from small wineries [23]. The treatment system consisted of a primary settling tank, an intermediate retention trough, two storage tanks and an aerobic treatment tank. A start up period of 7 days was given to the aerobic reactor and the system resulted in 93% COD and 97.5% BOD removal.

The activated sludge process and its variations utilize mixed cultures. To enhance the efficiency of aerobics systems, several workers have focused on the treatment by pure cultures. Further, aerobic treatment has also been examined as a precursor to anaerobic treatment. In studies with both beet spentwash and molasses, aerobic pre-treatment of beet spentwash with *Penicillium decumbens* resulted in about 74% reduction in phenolics content and 4[illegible] reduction in color [[illegible]]. Anaerobic digestion without aerobic pretreatment resulted in a sharp drop in COD removal efficiencies with decreasing hydraulic retention time (HRT). The organic matter removal was marginally higher for beet molasses previously fermented with *P. decumbens*. The anaerobic reaction followed first-order kinetics and the rate constant decreased on increasing the organic loading with untreated molasses; however, it remained almost constant with pre-treated molasses [24,25]. *Geotrichum candidum* is another species that resulted in partial elimination of phenolic inhibitors such as gentisic acid, gallic acid, quercetin, *p*-coumaric acid, etc., thereby enhancing the effectiveness of anaerobic process [26].

3.1.1.3. Constructed wetlands (CWs). Billore et al. [27] have demonstrated a four-celled horizontal subsurface flow (HSF) CW for the treatment of distillery effluent after anaerobic treatment. The post-anaerobic treated effluent had a BOD of about 2500 mg/l and a COD of nearly14,000 mg/l. A pre-treatment chamber filled with gravel was used to capture the suspended solids. All the cells were filled with gravel up to varying heights and cells three and four supported the plants *Typha latipholia* and *Phragmites karka*, respectively. The overall retention time was 14.4 days and the treatment resulted in 64% COD, 85% BOD, 42% total solids and 79% phosphorus content reduction. In another study, a distillery in northern India is presently employing CW for polishing the effluent prior to land discharge for irrigation in the surrounding paddy fields. The effluent was initially subjected to primary treatment which included settling and anaerobic digestion in a structured media attached growth (SMAG)-type anaerobic reactor. The primary treated effluent, with a COD of 28,000–35,000 mg/l, is subjected to two-stage aeration to bring down the COD to 400 mg/l. Thereafter, it is directed to a CW before final discharge.

3.1.2. Anaerobic process

The high organic content of molasses spentwash makes anaerobic treatment attractive in comparison to direct aerobic treatment. Therefore, biomethanation is the primary treatment step and is often followed by two-stage aerobic treatment before

Table 4
Performance of various anaerobic fluidized-bed reactor for treating molasses distillery wastewater

Name of the Reactor	OLR (Kg COD/m^3/day)	HRT (days)	COD removal (%)	References
Down-flow anaerobic fluidized-bed reactor	3.27–5.75	1.35–O.87	70–81	[41]
Inverse turbulent bed reactor	15		75 – 85	[43]
Inverse fluidized-bed reactor	10–12	60–3	>90	[44]
Anaerobic fixed film reactor	10		60–70	[45]
Granular-bed anaerobic bed reactor	4.75		80	[46]
Anaerobic contact filer	14	4	73–97	[35]
Fluidized-bed reactor	2.55–0.37	5.88	96.5	[47]
Inverse anaerobic fluidized-bed reactor	34.05	0.19	84	[7]

discharge into a water body or on land for irrigation [9]. Aerobic treatment alone is not feasible due to the high-energy consumption for aeration, cooling, etc. Moreover, 50% of the COD is converted to sludge after aerobic treatment [28]. In contrast, anaerobic treatment converts over half of the effluent COD into biogas [6]. Anaerobic treatment could be successfully operated at high organic loading rates; also, the biogas thus generated can be utilized for steam generation in the boilers, thereby meeting the energy demands of the unit [9].

The performance and treatment efficiency of anaerobic process could be influenced both by inoculums source and feed pre-treatment. In particular, thermal treatment of wastewaters could result in rapid degradation of organic matter leading to lower HRT, higher loading rate and BOD reduction. Moreover, the methane content and calorific value of biogas produced from thermophilic systems were higher [29]. Significant improvement was observed using inoculums from anaerobic lagoon with 27.2% COD reduction with thermally pre-treated wastewater and 51% reduction with untreated wastewater. Further, the temperature of thermal pre-treatment was also to be considered as an important one. After 150 days adaptation period, wastewater treated at lower temperature (77 °C) showed 66% COD reduction [30]. This was nearly twice the removal obtained with treatment at 23 °C. Further, addition of micronutrients (iron, boron and molybdenum) eliminated the long adaptation periods. Anaerobic lagoons are the simplest option for the anaerobic treatment of distillery spentwash. Though anaerobic lagoons are still employed in Indian distilleries, high rate anaerobic reactors are more popular [31]. These reactors offer the advantage of separating the HRT from solid retention time (SRT) so that slow growing anaerobic microorganisms can remain in the reactor independent of wastewater flow.

3.1.2.1. Fixed-bed reactor. This involves immobilization of microorganisms on some inert support to limit the loss of biomass and enhance the bacterial activity per unit of reactor volume. Moreover, it provides higher COD removal at low HRT and better tolerance to toxic and organic shock loadings. In anaerobic contact filters, various packing materials, viz. polyurethane, clay brick, granular activated carbon (GAC), polyvinyl chloride (PVC) plastic media have been employed resulting in 67–98% reduction in COD [32–35]. GAC as support media is relatively expensive, but because of its adsorptive properties, it contributes towards improved process stability. The interference by sulfate, unionized sulfate and total hydrogen sulfide is anaerobic. It was observed that the percentage sulfate removal increased with increasing HRT from 2 to 5 days. This might be due to the utilization of sulfate as a nutrient by microorganisms present in anaerobic contact and alter their conversion to sulfide by sulfate reducing bacteria (SRB) under anaerobic conditions. However, at higher sulfate concentration (426 mg/l), the removal decreased, possibly due to low SRB population in comparison to methanogens. Also, the removal of sulfide was explained by stripping of hydrogen sulfide from liquid to vapor phase by carbon dioxide and methane generated during the anaerobic process. In another study [36], anaerobic treatment of undiluted whisky pot ale using an upflow anaerobic filter (UAF) packed with special support type resulted in 76% COD removal. The pilot system also consisted of a decanter, dephosphatation or magnesium ammonium phosphate (MAP) ($MgNH_4PO_4$) reactor, denitrification reactor, nitrification reactor and sedimentation tank for the reduction of nitrogen and phosphate. Down-flow filter using plastic PVC as support material has been employed for the treatment of beet molasses wastewater [37]. The system resulted in 55–85% reduction in COD. Also, though high sulfide concentration (4250 mg) was inhibitory to the system, it was not toxic at higher loadings (44 kg COD/m^3/day) probably due to high stripping of H_2S.

3.1.2.2. Anaerobic fluidized-bed reactor. An anaerobic fluidized-bed reactor (AFBR) consists of a vertical vessel containing an inorganic media (e.g. rock, sand, activated carbon, anion and cation exchange resins) [38]. Anaerobic fluidized-bed reactor utilizes small fluidized media particles to induce extensive cell immobilization thereby achieving a high reactor biomass hold-up and a long mean cell residence time [39]. The fluidized-bed technology presented a series of advantages compared to other kinds of anaerobic processes [40], like high organic loading rates and short hydraulic retention times. Therefore, a number of design modifications have been tested or adopted in order to improve the performance of the system. In the classic case of fluidized systems, the solid particles have had a higher density than the fluid. Performance of various anaerobic fluidized-bed reactors for treating molasses distillery wastewater is given in Table 4.

3.1.2.3. Inverse anaerobic fluidized-bed reactor. In the field of anaerobic treatment process, an inverse anaerobic fluidized-bed reactor (IAFBR) has emerged as a good alternative for the treatment of spentwash from distilleries. In an inverse fluidization, the liquid specific density was found to be higher than the specific density of the particle, and the bed has been expanded downward by the liquid flow. The down-flow fluidized-bed reactor or inverse fluidized-bed reactor has been described for application in the anaerobic treatment of wastewater [41]. In their description, of down-flow fluidization, particles with a specific gravity smaller than the liquid are fluidized downward by a concurrent flow of liquid. The biofilm formation and its effect on hydrodynamics of the reverse fluidized-bed reactor have been described [41]. The application of inverse fluidization in wastewater treatment from laboratory to full-scale bioreactors has been described [42]. The biofilm, growing on the surface of support particles, increased the overall bioparticle (support particle plus biofilm) diameter. It resulted in bed expansion and very slow movement of the lower bed level downward until the lower bed level reached the lower draft tube opening and some of the bioparticles entered the draft tube with the liquid flow. The inverse fluidized-bed biofilm reactor designed in such a way that the biofilm thickness can be controlled to avoid the intrabiofilm diffusion limitations.

Perlite was an interesting carrier material when compared to others like cork, polyethylene or polypropylene [41]. The carrier particles chosen in a particular study [7] were perlite. Before starting up the reactor, physical properties of the carrier material were determined. 1 mm diameter perlite particle was found to have a wet specific density of 295 kg/m^3 with specific surface area of 7.010 m^2/g and low energy requirements for fluidization. It was used for the treatment of distillery waste and performance studies were carried out for 65 days. Once the down-flow anaerobic fluidized-bed system reached the steady state, the organic load was increased stepwise by reducing HRT from 2 to 0.19 days, while maintaining the constant feed of COD concentration. Most particles have been covered with a thin biofilm of uniform thickness. This system achieved [illegible]4% COD removal at an organic loading rate (OLR) of 35 kg COD/m^3/d [7].

3.2. Physico-chemical treatment

Sugarcane molasses spentwash after biological treatment by both anaerobic and aerobic method can still have a BOD of 250–500 mg/l [11]. Also, even though biological treatment results in significant COD removal, the effluent still retains the dark color [[illegible]]. The color imparting melanoidins are barely affected by conventional biological treatment such as methane fermentation and the activated sludge process [49]. Further, multistage biological treatment reduces the organic load but intensifies the color due to re-polymerization of colored compounds [50]. In this context, various physico-chemical treatment options have been explored.

3.2.1. Adsorption

Activated carbon is a widely used adsorbent for the removal of organic pollutants from wastewater but the relatively high cost restricts its usage. Decolorization of synthetic melanoidin using commercially available activated carbon as well as activated carbon produced from sugarcane bagasse was investigated by [51]. The adsorptive capacity of the different activated carbons was found to be quite comparable. Chemically modified bagasse using 2-diethylaminoethyl-chloride hydrochloride and 3-chloro-2-hydroxypropyltrimethylammonium chloride was capable of decolorizing diluted spentwash [52]. Significant decolorization was observed in packed bed studies on anaerobically treated spentwash using commercial activated charcoal with a surface area of 1400 m^2/g [53]. Almost complete decolorization (>99%) was obtained with 70% of the diluted sample, which also displayed over 90% BOD and COD removal. In contrast, other workers have reported adsorption by activated carbon to be ineffective in the treatment of distillery effluent [54,55]. Adsorption by commercially available powdered activated carbons resulted in only 18% color removal, however combined treatment using coagulation–flocculation with polyelectrolyte followed by adsorption resulted in almost complete decolorization [54,56] reported color removal up to [illegible]% with pyorchar. Yet another adsorbent that has been examined is the natural carbohydrate polymer chitosan derived from the exoskeleton of crustaceans [[illegible]] studied during the treatment of distillery wastewater using chitosan as an anion exchanger. At an optimum dosage of 10 g/l and 30 min contact time, 98% color and 99% COD removal was observed.

3.2.2. Coagulation and flocculation

[illegible]anc et al. [[illegible]] reported that coagulation with alum and iron salts was not effective for color removal. They explored lime and ozone treatment with anaerobically digested effluent. The optimum dosage of lime was found to be 10 g/l resulting in 82.5% COD removal and 67.6% reduction in color in a 30 min period. These findings have been in disagreement with those of [49] who used a commercial inorganic flocculent, a polymer of ferric hydroxysulfate with a chemical formula $[Fe_2(OH)n\cdot(SO4)_{3-n/2}]m$ for the treatment of molasses wastewater. The treatment resulted in around 87% decolorization for biodigested effluents; however an excess of flocculent hindered the process due to an increase in turbidity and TOC content. $FeCl_3$ and $AlCl_3$ were also tested for decolorization of biodigested effluent and showed similar removal efficiencies. About 93% reduction in color and 76% reduction in Total Organic Carbon (TOC) were achieved when either $FeCl_3$ or $AlCl_3$ was used alone. The process was independent of chloride and sulfate ion concentration but was adversely affected by high fluoride concentration. However, in the presence of high flocculent concentration (40 g/l), addition of 30 g/l CaO enhanced the decolorization process resulting in 93% color removal. This was attributed to the ability of calcium ions to destabilize the negatively charged melanoidins; further, formation of calcium fluoride (CaF_2) also precipitated the fluoride ions. Almost complete color removal (98%) of biologically treated distillery effluent has been reported with conventional coagulants such as ferrous sulfate, ferric sulfate and alum under alkaline conditions [58]. The best results were obtained using Percol 47, a commercial organic anionic polyelectrolyte, in combination with ferrous sulfate and lime. The combination resulted in 99% reduction in color and 87 and 92% reduction in COD and BOD, respectively. Similar findings have also been reported by [55]. Coagulation studies on spentwash after anaerobic–aerobic treatment have also been conducted using bleaching powder followed by aluminum sulfate [59]. The optimum dosage was 5 g/l bleaching powder followed by 3 g/l of aluminum sulfate that resulted in 96% removal in color, accompanied by up to 97% reduction in BOD and COD. Non-conventional coagulants namely wastewater from an iron pickling industry which is rich in iron and chloride ions and titanium ore processing industry containing significant amounts of iron and sulfate ions have also been examined [58]. The iron pickling wastewater gave better results with 92% COD removal, combined with over 98% color removal. Though the titanium processing wastewater exhibited similar color removal levels, the COD and BOD reductions were perceptibly lower.

3.2.3. Oxidation process

Ozone destroys hazardous organic contaminants and that have been applied for the treatment of dyes, phenolics, pesticides, etc. [50]. Oxidation by ozone could achieve 80% decolorization for biologically treated spentwash with simultaneous 15–25% COD reduction. It also resulted in improved biodegradability of the effluent. However, ozone only transforms the chromophore groups but does not degrade the dark colored polymeric compounds in the effluent [60,50]. Similarly, oxidation of the effluent with chlorine resulted in >97% color removal but the color reappeared after a few days [55]. Ozone in combination with UV radiation enhanced spentwash degradation in terms of COD; however, ozone with hydrogen peroxide showed only marginal reduction even on a very dilute effluent [61]. Samples exposed to 2 h ultrasound pre-treatment displayed 44% COD removal after 72 h of aerobic oxidation compared to 25% COD reduction shown by untreated samples. These results are contrary to those of [55] who concluded ultrasonic treatment to be ineffective for distillery spentwash treatment. A combination of wet air oxidation and adsorption has been successfully used to demonstrate the removal of sulfates from distillery wastewater. Studies were done in a counter-current reactor containing 25 cm base of small crushed stones supporting a 20 cm column of bagasse ash as an adsorbent [62]. The wastewater was applied from the top of the reactor and air was supplied at the rate of 1.0 l/min. The treatment removed 57% COD, 72% BOD, 83% TOC and 94% sulfates. Wet air oxidation has been recommended as part of a combined process scheme for treating anaerobically digested spentwash [63]. The post-anaerobic effluent was thermally pre-treated at 150 °C under pressure in the absence of air. This was followed by some enzyme treatment, after which the effluent underwent a 2 h wet oxidation at 225 °C. 95% color removal was obtained in this scheme. Another option is photo catalytic oxidation that has been studied using solar radiation and TiO_2 as the photo catalyst [64]. Use of TiO_2 was found to be very effective as the destructive oxidation process leads to complete mineralization of effluent to CO_2 and H_2O. Up to 97% degradation of organic contaminants was achieved in 90 min [65] studied combined electron beam and coagulation treatment of distillery slops from distilleries processing grain, potato, beet and some other plant materials. Humic compounds and lignin derivatives constitute the major portion of this dark brown wastewater. The distillery wastewater was diluted with municipal wastewater in the ratio of 3:4, irradiated with electron beam and then coagulated with $Fe_2(SO_4)_3$. The optical absorption in UV region was decreased by 65–70% after this treatment.

3.2.4. Membrane treatment

Pre-treatment of spentwash with ceramic membranes prior to anaerobic digestion is reported to halve the COD from 36,000 to 18,000 mg/l [66]. The total membrane area was 0.2 m^2 and the system was operated at a fluid velocity of 6.08 m/s and 0.5 bar transmembrane pressure. In addition to COD reduction, the pre-treatment also improved the efficiency of the anaerobic process possibly due to the removal of inhibiting substances [67] employed emulsion liquid membrane (ELM) technique in a batch process for spentwash treatment. Water–oil–water type of emulsion was used to separate and concentrate the solutes resulting in 86% and 97% decrease in COD and BOD, respectively. Electro dialysis has been explored for desalting spentwash using cation and anion exchange membranes resulting in 50–60% reduction in potassium content [68]. In another study [69], reported the treatment of vinasse from beet molasses by electro dialysis using a stainless steel cathode, titanium alloy anode and 4% w/v NaCl as electrolytic agent. Up to 88% COD reduction at pH 9.5 was obtained, however, the COD removal percentage decreased at higher wastewater feeding rates. In addition, reverse osmosis (RO) has also been employed for distillery wastewater treatment. A unit in western India is currently processing effluent obtained after anaerobic digestion, followed by hold-up in a tank maintained under aerobic conditions, in a RO system (S.R. Agrawal, personal communication). 290 m^3/day of RO treated effluent is mixed with 300 m^3/day of fresh water and used in the process for various operations like molasses dilution (290 m^3/day), make-up water for cooling tower (178 m^3/day), fermenter washing (45 m^3/day), etc. Yet another unit in southern India is employing disc and tube RO modules for direct treatment of the anaerobically digested spentwash (M. Prabhakar Rao, personal communication). The permeate is discharged while the concentrate is used for biocomposting with sugarcane press mud. In a recent study [70], reported pilot trials on distillery spentwash using a hybrid nano filtration (NF) and RO process. Both the NF and RO stages employed thin film composite (TFC) membranes in spiral wound configuration with module dimensions of 2.5 in. diameter and 21 in. length. NF was primarily effective in removing the color and colloidal particles accompanied by 80, 95 and 45% reduction in total dissolved solids (TDS), conductivity and chloride concentration, respectively, at an optimum feed pressure of 30–50 bar. The subsequent RO operation at a feed pressure of 50 bar resulted in 99% reduction each in COD, potassium and residual TDS.

3.2.5. Evaporation/combustion

Molasses spentwash containing 4% solids can be concentrated to a maximum of 40% solids in a quintuple-effect evaporation system with thermal vapor recompression [71,72]. The condensate with a COD of 280 mg/l can be used in fermenters. The concentrated mother liquor is spray dried using hot air at 180 °C to obtain a desiccated powder with a calorific value of around 3200 kcal/kg. The powder is typically mixed with 20% agricultural waste and burnt in a boiler. The use of recirculating fluidized-bed (RCFB) incinerator is recommended to overcome the constraints due to stickiness of spentwash and its high sulfate content [73]. Combustion is also an effective method of on-site vinasse disposal as it is accompanied by production of potassium-rich ash [19] that can be used for land application.

4. Discussion

The various biological and physico-chemical treatment methods have been identified for the treatment of wastewater from molasses-based distilleries. Due to the high COD of raw spentwash, application of anaerobic treatment technology (involving biogas recovery) has been reported to be highly effective which is considered as the first step. [74–77,33]. The treatment with anaerobic lagoon and conventional digesters are applied to some extent in the field. However, most distilleries in India employ high rate digesters wherein the HRT is decoupled from the biological/solids retention time (BRT/SRT) thereby retaining the slow growing anaerobic microbes in the reactor even at high wastewater flow.

Biological treatment using aerobic processes like activated sludge, biocomposting, etc. is presently practised by various molasses-based distilleries. Due to the large volumes generated, only a part of the total spentwash gets consumed in biocomposting. Biocomposting utilizes sugarcane pressmud as the filler material; thus it is typically employed by distilleries attached to sugar mills. Since sugar manufacturing is a seasonal operation, pressmud availability is often a constraint. Further, biocomposting requires a large amount of land; also, it cannot be carried out during the rainy season. Though aerobic treatment like the conventional activated sludge process leads to significant reduction in COD, the process is energy intensive and the color removal is still inadequate. Anaerobic reactor, particularly an inverse anaerobic fluidized-bed reactor [7] appears to be the only choice for the reduction in pollutional strength of distilleries when compared to other reactors. However, process design and control criteria for full exploitation of these reactors are yet to be developed.

In general physico-chemical treatment has met with little success. The techniques such as adsorption, coagulation/flocculation, oxidation processes and membrane treatment are more effective in removal of color as well as reduction in organic loading, sludge production and disposal is a constraint in coagulation/flocculation and adsorption. Settling is found to be unsatisfactory even with the addition of coagulants and other additives such as alum, lime, ferric chloride, other aluminum salts, iron salts, etc. in the coagulation/flocculation process. Also, the cost of chemicals, adsorbents and membranes is deterrent to the adoption of these methods [78]. Membrane operations like microfiltration/ultrafiltration for spentwash treatment are characterized by significant membrane fouling that limits its applicability [79].

5. Conclusion

This review article examined the extent of pollution created by distilleries and the different methods available for the treatment and disposal of distillery wastewater. Physico-chemical methods are capable of reducing organic load, consequently, in spite of cost; the advanced methods like membrane filtration, oxidation by ozone are being field trials. Biological treatment methods appear to be only choice for the removal of color and organic content; however, some of the questions are yet to be answered on its process efficiency. This is because of the lack of information on various aspects such as desirable influent COD, optimal level of volatile fatty acids (VFA) concentration in the reactor, the reliable estimates of the bio kinetics constants and their dependence on the substrate level (COD or VFA).

This review has showed, the reactor namely an inverse anaerobic fluidized-bed technology has been found to be more effective than anaerobic filter technology as it favors the transport of microbial cells from the bulk to the surface and thus enhanced the contact between microorganisms and the substrate for the treatment of high strength wastewater from molasses-based distilleries. The review also indicated the real evidence about excellent physical characteristics and adequate environment of perlite as an anaerobic microorganism immobilizer in an inverse anaerobic fluidized-bed reactor and presented series of advantages of an inverse anaerobic fluidized-bed technology compared to other kinds of anaerobic processes interms of high organic loading rates and short hydraulic retention times. The application of this reactor to the treatment of molasses-based distillery wastewater would give satisfactory results compared to other biological reactors.

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