



# **A Short Review on Dye-Wastewater Valorization Using Up-Flow Anaerobic Sludge Blanket Reactors**

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Abstract: Dye-containing effluent generated in textile industries is polluting and complex wastewater. It should be managed adequately before its final destination. The up-flow anaerobic blanket (UASB) reactor application is an ecofriendly and cost-competitive treatment. The present study briefly reviews the UASB application for dye-containing wastewater valorization. Bioenergy and cleanwater production potential during dye-containing wastewater treatment are emphasized to promote resource recovery in textile industries. Hydraulic retention time (HRT), organic loading rate (OLR), pH, temperature, and hydraulic mixing influence sludge granulation, microbial activity, and dye removal. HRT and OLR ranges of 6–24 h and 1–12 kg m<sup>-3</sup> d<sup>-1</sup> of chemical oxygen demand (COD) at a mesophilic temperature (30–40 °C) are recommended for efficient treatment. In these conditions, efficiencies of color and COD of 50-97% and 60-90% are reported in bench-scale UASB studies. Complex dye structures can hinder biomineralization. Pretreatment may be necessary to reduce dye concentration. Carbon-source and redox mediators are added to the UASB reactor to expedite kinetic reactions. A biogas yield of  $1.48-2.70 \text{ L} \text{ d}^{-1}$  in UASB, which treats dye-containing effluents, is documented. Cotreatment of dye wastewater and locally available substrate could increase biogas productivity in UASB reactors. Organic waste generated in the textile industry, such as dye sludge, cotton, and starch, is recommended to make cotreatment cost competitive. Bioenergy production and water reuse allow environmental and economic benefits. Studies on combined systems integrating UASB and membrane processes, such as ultrafiltration and nanofiltration, for the production of reusable water and pretreatment of wastewater and sludge for improvements in biogas production might realize the complete potential for resource recovery of UASB technology. UASB bioenergy usage for integrated treatment trains can reduce operating costs and assist process sustainability in the textile industry.

Keywords: biogas; dye-containing wastewater; resource recovery; sludge; UASB reactors; water reuse

# 1. Introduction

Dye-containing wastewater discharged from textile industries poses a significant environmental challenge. Among the several concerns, colored effluents impair plant photosynthesis and reduce light penetration and oxygen levels in aquatic ecosystems. It may also be lethal for marine life due to the presence of metals and chlorine in synthetic dyes [1]. In textile wastewater, metal ions, dyes, and color are of the first concern due to their harmfulness to public health and the environment. Discharge standards vary according to the local regulatory agency and municipalities; thus, it should be checked in each situation [2]. The recognition of the health hazards of dyes has highlighted the need to develop rapid and reliable analytical methods for detection and forced regulatory permissible limits in this respect. Twenty pharmacologically active dyes were quantified in water and industrial textile effluent samples. Dyes were found in two treated effluents.



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**Copyright:** © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). In one, rhodamine B was found at a concentration of 0.043  $\mu$ g L<sup>-1</sup>, and the other one contained crystal violet, methyl violet 2B, and rhodamine B in 0.023, 0.017, and 0.027  $\mu$ g L<sup>-1</sup>, respectively [3].

Dye wastewater should be preferentially treated using ecofriendly technologies. In this context, biotreatments are cost competitive, give total mineralization or nonhazardous byproducts, and consume less water than physical and oxidative methods [1]. Biotreatments occur under aerobic or anaerobic conditions, as the products of aerobic treatment are biomass,  $CO_2$ , and  $H_2O$ . In contrast, the main product of anaerobic treatment is biogas (composed of  $CH_4$  and  $CO_2$  in varying compositions). Combinations of anaerobic and aerobic systems are implemented on a full scale for dye-wastewater purification. The up-flow anaerobic sludge blanket (UASB) reactor is a promising anaerobic wastewater-treatment technology for high-strength wastewater like dye-containing effluents [4].

'UASB's compact design and low cost are useful for several applications, such as brewing and beverage, distilleries, food, pulp and paper, food processing, chemical industries, landfill leachate, and textile effluents [5,6]. A full-scale 1800 m<sup>3</sup> d<sup>-1</sup> UASB-treating sewage wastewater was monitored for 35 weeks. Organic matter removal was higher than 90%, and the biogas yield was estimated at 0.2 m<sup>3</sup> per kg of chemical oxygen demand (COD) removed [7]. For textile wastewater, a two-phase pilot UASB reactor was tested. A maximum COD removal of 88.5% was recorded in the methanogenic reactor with a biogas production of 0.312 m<sup>3</sup> d<sup>-1</sup> [8].

Recently, investigators have examined the factors affecting the UASB reactor's performance, conventional configuration, and derivatives [4]. Some parts of our previous work discussed treatability findings of UASB in textile-wastewater purification [9]. However, research still needs to analyze this cost-effective technology, focusing on energy and water recovery. Given the global energy crisis and rising water demand, bioenergy production and water reuse during wastewater treatment are fundamental to achieving sustainability [10].

This paper provides a short overview of UASB reactors for the valorization of dye wastewater. It introduces the aspects of UASB reactors and the operating conditions employed for effective dye removal. Next, it delves into the potential of bioenergy and clean-water production, emphasizing their role in promoting resource recovery in textile industries. In this context, knowledge gaps and research opportunities are identified.

# 2. Up-Flow Anaerobic Sludge Blanket Reactors

The UASB reactor, also known as a three-phase separator, allows the reactor to separate mixtures of gas, water, and sludge under conditions of high turbulence. During the UASB treatment, the wastewater passes through a bed of expanded sludge containing a high biomass concentration (up to 80 g L<sup>-1</sup>) [11]. The peristaltic pump pumps the influent into the UASB reactor from the bottom. It moves upwards, coming into contact with the biomass in the sludge bed and then moving upwards [12,13]. The typical height–diameter ratio of UASB reactors ranges from 0.2 to 0.5 [14]. A three-phase separator (Gas–Liquid–Solid, GLS) above the sludge blanket separates the GLS mixture. It, therefore, allows fluid and gas to exit the UASB reactor [15]. The GLS separator must have a designed height to avoid flotation effects and, consequently, floating layers. After treatment, the treated water is collected by the collection system through several drains distributed throughout the discharge area up to the main drain provided on the periphery of the reactor. The biogas generated is drained, and it contains mainly CH<sub>4</sub>, followed by CO<sub>2</sub> and traces of other compounds [16]. Figure 1 presents a 3D-designed UASB reactor for wastewater treatment and biogas production.



Figure 1. UASB Reactor in 3D designed for effluent treatment and biogas production.

The UASB performance is influenced by hydraulic retention time (HRT), temperature, organic loading rate (OLR), hydraulic mixing, and sludge granulation. HRT affects the treatment time and removal performance of pollution parameters. It is also linked with the up-flow velocity chosen for UASB operation. When the up-flow velocity is higher than 1.5 m/h, sludge disintegration and biomass washout may occur, reducing the removal efficiency of chemical oxygen demand (COD) [17]. In addition, OLR impacts microbial activity and biodegradation performance. An HRT range of 3–10 and an OLR of 4–15 is recommended to achieve COD removal of 60–85% [9,11]. The thermophilic temperature (50–65 °C) assures higher process stability and biogas production [18]. Still, a temperature range of 30–40 °C effectively maintained methanogen activity and reactor stability [17]. The following section presents a comprehensive summary of influence parameters impacting dye removal in UASB.

Likewise, the successful adoption of this technology depended on establishing a dense granular sludge bed within the UASB reactors. The efficacy of these reactors in wastewater treatment is ascribed to forming a compact sludge bed in the lower region of the bioreactor. Anaerobic granules comprise microbial clusters that are densely organized and highly structured, requiring no carrier media for support. This granular biomass presents as a densely aggregated microbial consortium characterized by its condensed architecture and expansive specific surface area, thereby facilitating the adsorption and biotransformation of contaminants [14].

In contrast, developing anaerobic granular sludge requires 2 to 8 months, leading to an extended initiation phase for the bioreactor—a notable challenge inherent to UASB technology [19]. Hulshoff Pol et al. [20] thoroughly examined theories on sludge granulation within UASB reactors, ultimately discerning the pivotal role of incorporating inert support particles in conjunction with operational conditions in the genesis of granular sludge. Likewise, a hypothesis suggesting that granulation is an inherent defensive response of microorganisms against external stresses is presented in the literature [20]. Such stresses could be manipulated by regulating reactor operational conditions to stimulate the development of granules. It was reported that the rapid growth of granules could be achieved through particle agglomeration of the flocculant sludge induced by hydraulic stress. In UASB, the up-flow liquid provides a selection pressure by washing out light and dispersed particles while retaining denser biomasses. Thus, controlling up-flow liquid velocity could be critical for granule formation [21].

As mentioned, the issue of sludge granulation relies on the extensive reactor's startup time to develop granules. In this sense, one effective method for a rapid start up is acquiring healthy granules from other reactors and using them as the inoculum. However, the availability of granular sludge may be limited, and the expenses for acquiring and transporting the granules can hamper it. Other possible ways to accelerate the start up include supplementing chemicals and polymers or stressing the loading rate [21]. It was recently demonstrated that chemical addition could stimulate sludge granulation. Calcium sulfate (CaSO<sub>4</sub>) and polymers were used to enhance granulation during the treatment of phenolic wastewater in UASB reactors. The CaSO<sub>4</sub> improved the granulation rate as nuclei, and the subsequent dissolution of CaSO<sub>4</sub> improved methanogen activity. The utilization of CaSO<sub>4</sub> and polymers enhanced the microbial diversity. The formed granules had a large particle size (>0.25 mm), great settleability, and high methanogenic activity [22].

Despite substantial investigative efforts, the mechanisms governing the formation of anaerobic granules still need to be discovered. Anaerobic granulation has become a central focus of both engineering and scientific research, making the need for efficient methods to expedite granule development desirable. In addition to long reactor start up, gas leakage and corrosion-related issues require periodic monitoring and maintenance for effective treatment outcomes [23].

# 3. Mechanisms and Influencing Parameters in Textile Decolorization in UASB Reactors

#### 3.1. Mechanisms of Dye Removal

The dye-removal process in UASB reactors involves two main mechanisms: abiotic adsorption and biotic biodegradation. The adsorption mechanism, facilitated by sludge granules, plays a significant role in decolorization. On the other hand, biodegradation occurs under anaerobic conditions and primarily focuses on azo 'dyes' biochemistry [24]. The primary degradation mechanism involves the cleavage of the azo bond (–N=N–) by extracellular azoreductase enzymes, which transfer four electrons (reducing equivalents) (Equation (1)). The permeation of the azo dyes through the membrane of microbial cells acts as the principal rate-limiting factor for decolorization [25]. The generated hydrazo intermediates undergo reductive cleavage, resulting in uncolored aromatic amines as byproducts, as shown in Equation (2) [26].

$$R_1 - N = N - R_2 \xrightarrow{2e^- + 2H^+} R_1 - NH - NH - R_2$$
(1)

$$R_1 - NH - NH - R_2 \xrightarrow{2e^- + 2H^+} R_1 NH_2 + R_2 NH_2$$

$$(2)$$

where  $R_1$  and  $R_2$  are aryls or heteroaryl groups.

It is important to note that produced aromatic amines are generally anaerobically recalcitrant and have higher toxicity than dye precursors. Consequently, anaerobically treated effluent needs further treatment. Biological sequential anaerobic–aerobic treatment has been used to remove azo dyes completely. Under low oxygen concentrations, facultative bacteria consume oxygen and introduce hydroxyl groups into polyaromatic compounds, facilitating biodegradation pathways. However, aromatic amines have substituents with nitro and sulfonic groups; these are highly recalcitrant for aerobic microorganisms, which prevents efficient contaminant mineralization [9,25]. The decolorization of azo dyes under anaerobic conditions is thought to be a relatively simple and nonspecific process. Readers are guided toward the contribution of Saratale et al. [25] for further background information on dye decolorization using biological methods.

In an anaerobic environment, organic matter undergoes four steps: hydrolysis, acidogenesis, acetogenesis, and methanogenesis. In the three former acid fermentation steps, fermentative bacteria hydrolyzed and metabolized organic macromolecules and converted them to carbon dioxide, hydrogen, and acetic acid. Later, acetic acid, carbon dioxide, and hydrogen are converted to carbon dioxide and methane by methanogenic archaeans [27].

Biodecolorisation under anaerobic conditions necessitates supplementary organic C-sources, as dye-reducing microbial consortia cannot utilize the dye as a growth substrate. Fermentative bacteria and hydrogenotrophic methanogens primarily carry out dye reduction. Noteworthy among the microorganisms involved in anaerobic biodecolorisation are *Methanosarcina archaea*, *Clostridium*, *Enterococcus*, *Pseudomonas*, *Bacillus*, *Aeromonas*, *Enterococcus*, *Desulfovibrio*, and *Desulfomicrobium* bacteria. [9].

#### 3.2. Influence Parameters of Dye Removal

Dye structure and concentration, electron donors and redox mediators, pH, temperature regime, hydraulic retention time (HRT), and organic loading rate (OLR) are the primary influence parameters governing dye removal in UASB reactors (Table 1).

Table 1. Influencing parameters in dye decolorization in UASB reactors.

Influencing Parameters	Main Aspects	Main Findings	Reference
Dye structure and concentration	<ul> <li>High dye concentration might affect microorganism growth rate, enzymatic activity, and biodescolorization performance.</li> <li>High dye dosage is linked to high salinity and biotoxicity, which reduces microbial activity.</li> <li>Salinity decreases biomass size and hydrophobicity, affecting biodegradation and sludge settling.</li> <li>Complex dye structure might hamper the mineralization of the molecules by microorganisms.</li> </ul>	<ul> <li>450 mg dye L<sup>-1</sup> could decrease the granular sludge porosity and strength, reduce its settling ability, and inhibit methanogenic activity.</li> <li>&gt;300 mg L<sup>-1</sup> sulfate dosage might inhibit methanogens.</li> </ul>	[28,29]
Electron donors and redox mediators	<ul> <li>C sources are required in anaerobic dye removal.</li> <li>Redox mediators increase biodescolorization kinetic as they accelerate electron transfer from C-source to dye.</li> </ul>	<ul> <li>Riboflavin and sulfonated compounds, such as anthraquinone sulfonate and disulfonated anthraquinone, are usually employed as redox mediators.</li> <li>Riboflavin (0.00175 mg L<sup>-1</sup>) and yeast extract (500 mg L<sup>-1</sup>) increased as C sources increased dye decolorization in UASB reactors.</li> </ul>	[30–33]
рН	<ul> <li>It affects 'microorganisms' growth rate, enzymatic activity, and biodescolorization efficiency.</li> <li>In an anaerobic environment, methanogens grow efficiently in the pH range of 6.0–8.0 and are sensitive to pH fluctuation.</li> </ul>	• Azo dye Direct Black G biodescolorization of 97% at pH 8.0, 79% at pH 11.0, and 81% decolorization at pH 4.0 after 48 h of residence time.	[34,35]
Temperature	• It affects the microbial community and methanogen activity.	• The optimum temperature for biodecolourisation ranges from 30 to 55 °C and exceeding this range could harm the syntrophic relationship among anaerobic microorganisms.	[33,36]

Influencing Parameters	Main Aspects	Main Findings	Reference
OLR	• High OLR can affect methanogens and inhibit methane production in UASB reactors.	<ul> <li>It was reported that methane-production efficiency was 75% at OLR of 2.4 kg COD m<sup>-3</sup> d<sup>-1</sup> and 38% at 22.5 kg COD m<sup>-3</sup> d<sup>-1</sup>.</li> <li>Temperature adjustment and effluent recirculation can alleviate the harmful effects of high OLR.</li> </ul>	[37–39]
HRT	<ul> <li>Lower-than-optimal HRT leads to the misdevelopment of granular sludge and acidification.</li> <li>Higher-than-optimal HRT results in low reactor components and biomass washout utilization.</li> </ul>	<ul> <li>Dye removal was reported at 67% at 16 h HRT and 55% at HRT of 96 h.</li> <li>Optimal HRT ranges from 5 to 20 h.</li> </ul>	[40-42]

Table 1. Cont.

Note: COD: chemical oxygen demand. HRT: hydraulic retention rate.

In sum, complex dye structures can hinder their biomineralization. Therefore, monitoring the dye level in wastewater before initiating the anaerobic process is essential. Pretreatment may be necessary to reduce the dye concentration. C-source and redox mediators are commonly added to the UASB reactor to expedite kinetic reactions. Temperature, pH, OLR, and HRT influence microbial activity and UASB performance. For optimal results, operating the UASB reactor at 30 °C and 40 °C, with an HRT ranging from 5 to 20 h, and an OLR of 2 to 15 kg COD m<sup>-3</sup> d<sup>-1</sup> was demonstrated to be ideal [11,43]. Likewise, Mohan and Swathi [4] identified that optimal conditions for UASB for treating various types of wastewater are an HRT of 3–24 h, an OLR of 1–15 kg COD m<sup>-3</sup> d<sup>-1</sup>, and an operational temperature in the mesophilic range (30–40 °C). To mitigate the harmful impacts of high OLR, adopting a feed mode in an intermittent regime and employing internal effluent recirculation can be effective strategies for UASB operations [44].

# 4. UASB Reactor's Performance in Treating Dye-Containing Effluents

In decolorization studies, color and COD are commonly employed as monitoring parameters to evaluate the performance of UASB reactors. Table 2 presents the data on dye removal using UASB reactors, as reported in the recent literature from 2018 to 2022. Based on data from Table 2, HRT and OLR ranges of 6–24 h and 1–12 kg COD m<sup>-3</sup> d<sup>-1</sup> at a mesophilic temperature are recommended for efficient treatment. The operating conditions are similar to those previously discussed in the literature when treating diverse wastewaters. The treatability results demonstrate a range of color removal efficiencies from 50% to 97% and COD-removal efficiencies from 60% to 90%. All the reported findings are based on lab-scale investigations, necessitating further full-scale research to validate the outcomes in full-scale plants.

Scheme	Scale	<b>UASB</b> Reactor Conditions		Dye Compounds		Treatabili	ity Results	Reference
			Туре	Name	Concentration/Amount	t Color	COD	
UASB reactor	Lab	Continuous mode, 27 °C, HRT 24 h, OLR *	Azo dye	Reactive Red 2	$50~{ m mg~L^{-1}}$	51%	89%	[45]
<b>UASB reactor</b> + Activated sludge process	Lab	Continuous mode, 16 °C–29 °C, HRT 24 h, OLR *	Azo dye	Yellow Gold Remazol	$50 \text{ mg } \mathrm{L}^{-1}$	85%	67–88%	[46]
<b>UASB reactor</b> + shallow polishing pond	Lab	Continuous mode, 16 °C–29 °C, HRT 24 h, OLR *	Azo dye	Yellow Gold Remazol	$50 \text{ mg } \mathrm{L}^{-1}$	85%	67–88%	[46]
UASB reactor	Lab	Continuous mode, temperature *, TRH 24 h, OLR *	Azo dye	Red Bronze	40–325 mg $L^{-1}$	75–94%	60–91%	[47]
<b>UASB reactor</b> + Aerated bioreactor	Lab	Continuous mode, $37 \pm 1$ °C, HRT 6 h, OLR 12.97 kg C.O.D. m <sup>-3</sup> d <sup>-1</sup>	Azo dye	2-Naphthol Red	$0.1  \mathrm{g}  \mathrm{L}^{-1}$	96%	85.6%	[48]
<b>UASB reactor</b> + microaerated UASB reactor	Lab	Continuous mode, 25.0 ± 1.4 °C, HRT *, OLR 1.27–1.50 kg m <sup>-3</sup> d <sup>-1</sup>	Azo dye	Direct Black 22	0.6 mM	70–78%	67–72%	[49]
<b>UASB reactor</b> + shallow polishing pond	Lab	Continuous mode, 16–29 °C, HRT 24 h, OLR *	_	Real textile wastewater	_	50%	80%	[50]
<b>UASB reactor</b> + EC system	Lab	Temperature *, HRT 8–12 h, OLR *		Congo Red dye	$100~{ m mg}~{ m L}^{-1}$	>96%	>82%	[51]
UASB reactor	Lab	Continuous mode, 27–29 °C, HRT 24 h, OLR 6.20 kg COD m <sup>-3</sup> d <sup>-1</sup>		Simulated wastewater containing Remazol blue RSP	$12.5 { m mg} { m L}^{-1}$	97.37 ± 3.62%	$76.69 \pm 2.83\%$	[52]
<b>UASB reactor</b> + SBR	Lab	Intermitent mode, 35 °C, HRT 48 h, OLR 0.74–0.90 kg COD m <sup>-3</sup> d <sup>-1</sup>		Real textile wastewater	_	87.7%	90.4%	[53]

Table 2. Studies on UASB reactors on dye removal mapped from the last five years (2018–2022).

Note: \*, Data not available. COD: chemical oxygen demand, E: electrochemical, HRT: hydraulic retention time, OLR: organic loading rate, SBR: sequencing batch reactor, UASB: up-flow anaerobic sludge blanket.

Bahia et al. [50] used an integrated UASB–shallow pound system in continuous feeding, achieving color and COD removal rates of 50% and 80%. Saleem et al. [53] combined UASB with a sequencing batch reactor (SBR) in an intermittent regime, resulting in higher removal rates of 87.7% for color and 90.4% for COD. These studies highlight how the feeding mode can significantly impact UASB efficiency. Saleem et al. noted that, during nonfeeding periods, anaerobic microorganisms can better withstand dye toxicity and effectively handle changes in temperature, HRT, and OLR. This insight suggests that optimizing the feeding strategy can improve UASB performance in dye-wastewater treatment.

However, anaerobic treatment alone may not fully break down dye byproducts such as polyaromatic amines. As in those studies, aerobic systems were integrated with UASB to address this issue. Aerobes can utilize oxygen and introduce hydroxyl groups into polyaromatic compounds at aerobic conditions. This step is essential in facilitating subsequent biodegradation pathways. Consequently, the aerobic process acts as a polishing step, effectively completing the mineralization of intermediates that arise from the anaerobic biotransformation. This completion occurs through hydroxylation or cleavage of the ring using oxidative enzymes such as laccase, phenoloxidase, and peroxidase [54]. On the other hand, amine byproducts have substituents with nitro and sulfonic groups, hampering their mineralization in an aerobic environment. Romero-Soto et al. [51] investigated sequential UASB and electrochemical (EC) systems for Cong Red (CR) removal. COD and CR removals were >92% and >98% using UASB + electrocoagulation and >99% and >99% when UASB + electro-oxidation was employed. Results are promising to be used in dye-wastewater treatment for removing byproducts that arise from UASB treatment. Still, despite the widerange removal of pollutants, easy construction, and operating simplicity, technological developments of EC systems are needed to reduce energy consumption and electrode replacement in full-scale plants [55]. In another work, Carvalho et al. [49] proposed using a microaerated UASB reactor to remove Direct Black 22 azo dye. The UASB reactor was aerated in the upper part with a low oxygen concentration ( $0.18 \pm 0.05 \text{ mg O}_2 \text{ L}^{-1}$ ) to facilitate the mineralization of amines generated during the anaerobic process. As a result, the removal of COD and color ranged from 59% to 78%. In addition, the treated effluent from the microaerated reactor was 16 times less toxic than that of conventional UASB, indicating the effectiveness of the microaeration method in removing anaerobic metabolites.

#### 5. Dye-Wastewater Valorization

Added-value product extraction from dye-industry wastes has been investigated, and a comprehensive review of resource recovery of colored effluents was recently published [56]. Dye-wastewater management for bioenergy, water reuse, and sludge valorization is explored in the present section (Figure 2). We cover the UASB application for bioenergy and water reuse, which, to date, are the most realistic strategies for practical applications.

#### 5.1. Bioenergy Production

Anaerobic technology offers the dual advantage of degrading dye pollutants in wastewater while also serving as a significant source of clean energy. Dye-containing wastewaters are rich in organic chemicals. The organic load is converted into biogas in UASB reactors. Biogas consists of methane (up to 75%), carbon dioxide (up to 50%), and hydrogen (up to 5%) with small amounts of water vapor, dinitrogen, hydrogen sulfide, ammonia, and siloxanes. As a result, biogas possesses a high calorific value and can be directed for thermal and/or electrical energy production [57].

The dual potential of anaerobic technology helps in wastewater valorization and contributes to sustainable energy production [58]. Katal et al. [59] conducted experiments using a lab-scale UASB reactor to treat textile effluent and measure the biogas production yield. They achieved a maximum biogas productivity of 36 L per day at an HRT of 50 h, with a biomethane content of 79%. Other bench-scale studies reported biogas production rates ranging from 1.48 to 2.7 L per day [42,60–62] (Table 3).



Figure 2. Dye-wastewater valorization for sustainability in textile industries.

The cotreatment of actual dye wastewater and starch effluent indicated higher biogas production than a solely dye-containing treatment in UASB. The literature reports a maximum biogas production range of 24.5–355 L d<sup>-1</sup>, cotreating dye and starch effluents [8,63,64]. Cotreatment using UASB reactors could be promising to increase biogas productivity; still, a technoeconomic analysis should be performed before adopting such a strategy since cosubstrate availability and logistics can hamper implementation on a full scale [65]. Cotreatment is the most cost competitive when the cosubstrate is locally available and implemented on a large scale [66]. Based on this, organic waste generated in the textile industry, such as dye sludge, cotton, and starch, is suggested to increase biogas production outcomes.

Industrial treatment facilities have a high energy demand [67]; thus, UASB technology offers opportunities for reducing treatment costs while treating wastewater. Gadow and Li [48] showed that the UASB technology could be extended to full-scale applications for 2-Naphthol red removal with a bioenergy recovery of 139.6 MJ per m<sup>3</sup> of effluent. A maximum methane yield of 13.3 mmol CH<sub>4</sub> g<sup>-1</sup> COD d<sup>-1</sup> was obtained at an HRT of 6 h. In another work from the same research group, a similar methane yield of 13.18 ± 0.64 CH<sub>4</sub> g<sup>-1</sup> COD was recorded during the treatment of synthetic dye wastewater [68]. Apart from bioenergy recovery and the related economic benefits, reducing greenhouse-gas emissions is expected and could help boost the C-neutrality of wastewater-treatment plants. Moreover, lower excess sludge is discharged from UASB reactors [69].

A recent study compared a pilot-scale UASB and anaerobic membrane bioreactor (AnMBR) treating domestic wastewater [70]. The UASB reactor produced  $230 \pm 35$  L of biogas daily (73 ± 3% CH<sub>4</sub>) at an HRT of 15 h. The UASB pilot plant demonstrated high stability and fewer technological requirements than AnMBR. Thus, it is a better candidate for decentralized treatment. It could also be integrated with other renewable energy alternatives for heat and electricity production.

Scheme	<b>UASB</b> Reactor Conditions	Dye Compound	<b>Biogas Production</b>	Reference
UASB reactor	Temperature of 37 °C, HRT 20 h, OLR 3.86 kg COD m <sup>-3</sup> d <sup>-1</sup>	Azo dye mixture: Reactive Black 5, Direct Red 28, Direct Black 38, Direct Brown 2, and Direct Yellow 12 (250 mg $L^{-1}$ )	2.26 L d <sup>-1</sup> (70%CH <sub>4</sub> , <i>v/v</i> )	[61]
<b>UASB reactor</b> + CSTR reactor	Temperature of 37 °C. HRT 3–30 h, OLR 2–15 kg COD m <sup>-3</sup> d <sup>-1</sup>	Real textile wastewater	$0.36-0.94 L d^{-1}$	[60]
UASB reactor	Temperature of 37 °C, HRT 18.3 h, OLR 0.286 kg m <sup><math>-3</math></sup> d <sup><math>-1</math></sup>	Red Congo azo dye (100 mg $L^{-1}$ )	$2.0$ – $2.7 L d^{-1}$	[42]
Two-phase UASB reactor	Ambient temperature, HRT 12 h, OLR 8 kg COD m <sup><math>-3</math></sup> d <sup><math>-1</math></sup>	Real dye wastewater + starch effluent (40:60% $v/v$ )	$24.5 L d^{-1}$	[64]
UASB reactor	Ambient temperature, HRT 24 h, OLR *	Real dye wastewater + starch effluent ( $30:70\% v/v$ )	$355 L d^{-1}$	[63]
Two-phase UASB reactor	Ambient temperature, HRT 24 h, OLR *	Real textile wastewater + sago effluent ( $30:70\% v/v$ )	$312 L d^{-1}$	[8]
UASB reactor	Temperature of 33 °C, HRT 50 h, OLR 12 kg COD m $^{-3}$ d $^{-1}$	Real textile wastewater	36.04 L d <sup>-1</sup> (79%CH <sub>4</sub> , $v/v$ )	[59]
UASB reactor	Temperature of 45 °C, HRT of 24 h, OLR *	Textile sludge	$1.48 \pm 0.89 \text{ L d}^{-1}$ (36.7% CH <sub>4</sub> , $v/v$ )	[62]
<b>UASB reactor</b> + aerobic system	Temperature of $37 \pm 1$ °C, HRT 6 h, OLR 12.97 kg COD m <sup>-3</sup> d <sup>-1</sup>	2-Naphthol Red (100 mg $L^{-1}$ )	$3.86 \text{ L CH}_4 \text{ m}^{-3} \text{ d}^{-1}$	[48]

 Table 3. Biogas production treating dye wastewater in UASB reactors.

Note: \*, Data not available. COD: chemical oxygen demand, CSTR: continuous stirred tank reactor, HRT: hydraulic retention time, OLR: organic loading time, UASB: up-flow anaerobic sludge blanket reactor.

A full-scale UASB reactor was operated for seven years for brewery-effluent treatment in Korea. COD removal of the UASB reactor averaged over 80% throughout the period, incurring operating costs of 0.20–0.31 USD m<sup>-3</sup> [71]. In Brazil, the energy potential of biogas from sewage treatment using UASB reactors for wastewater and/or sludge valorization was estimated at 1.53–3.50 MJ m<sup>-3</sup>. However, the energetic advantages of UASB have not been fully explored in the country [72]. In the Brazilian industry, biogas production was estimated at 0.7 billion Nm<sup>3</sup> y<sup>-1</sup> in 2022, amounting to only 126 plants [73]. The data show much room for growth in the Brazilian market, and industries should further explore the technoeconomic benefits of UASB technology.

On the other hand, energy recovery from dye effluents can be hampered, given the dye's low biodegradability and/or high effluent salinity. Pretreatments like advanced oxidation processes, ultraviolet (UV) photodegradation, and chemical coagulation were investigated to improve dye biodegradability [74,75]. UV pretreatment improved biogas production 2.7-fold compared with nonpretreated effluent and increased methane yield in the anaerobic digestion (AD) of methylene blue [74].

A recent review analyzed landfill-leachate pretreatment methods coupled with AD to enhance biogas production [76]. Landfill leachate, as a dye effluent, is a complex and inhibitory wastewater for anaerobic processes [76–79]. Because of its recalcitrance, biotreatments necessitate employing other techniques to complement and support the AD. The work concluded that electrochemical systems and photocatalysis are promising due to their performance and cost effectiveness. Studies on dye-wastewater pretreatments are scarce, and research is necessary to close existing knowledge gaps in this area. Sludge and dye-wastewater pretreatments might foster AD and UASB utilization for dye-wastewater valorization in full-scale applications.

# 5.2. Reclaimed Water

The dye industries consume a high amount of water, and, consequently, a high waste volume is discharged [80]. To solve such issues, water recovery for reuse in textile industries might allow environmental and economic benefits. However, the UASB technology must

be integrated to produce clean water for recycling. Therefore, a treatment train is required. An integrated system comprising reverse osmosis (RO), electrochemical oxidation, and electrodialysis was investigated. It demonstrated feasibility for large applications [81]. This system could produce 0.97 tons of clean water at 24.7 kWh per m<sup>3</sup> of dye wastewater. However, high energy demand can make this integrated process less competitive.

Recent studies have analyzed driven-pressure membrane processes, such as ultrafiltration (NF) and nanofiltration (UF), demonstrating the ability of these techniques to produce reclaimed water [82,83]. Hybrid bio-oxidation and NF processes performed well in removing soluble dyes and surfactants. They could significantly reclaim water from textile wastewater [83]. In this work, the authors highlighted that integrating both treatments to produce recycled water is needed, corroborating the necessity of combining recovering technologies. Membrane-based methods like RO, NF, and UF have been used for treating several kinds of wastewater for effective pollutant removal, making effluents reusable for industrial, agricultural, or domestic purposes [84–86].

Erkanli et al. [82] analyzed different configurations of the two-stage UF process for recovering water from actual dye wastewater. A two-stage UF using membranes with a molecular weight cut off of 2 kDa produced high-quality water to an extent that allows for reuse in fabric dyeing. At an estimated 200–400 L of water per kg of fabric, water recovery could promote significant economic savings. Also, the UF method is economical and less energy intensive than other membranes like NF and RO [23,87]. Thus, it can be a potential candidate to be integrated with UASB technology, with an aim to produce clean water. Table 4 summarizes the relevant studies on membrane-based methods for dye-containing effluent treatment for water reuse.

Treatment Scheme	Features	Main Findings	Reference
SBR + NF	Dye: raw textile wastewater; Membrane: Alfa Laval (Alfa Laval, Sweden); Operating conditions: TPM = 5 bars, 20 °C.	COD and color removal of >80% and >96%; Water flux of 23.71 LMH; Combined SBR and NF treatment cost estimated at 0.97 USD m <sup>-3</sup> .	[88]
UF	Dye: raw textile wastewater; Membrane: UF-GH 2 kDa GE (Water and Process Technologies); Operating conditions: TPM = 10 bars, 25 °C.	COD and color removal of 56% and >95% Water flux of 20–30 LMH; Treated water was suitable for dyed knitted cotton fabric washing.	[89]
SBR + NF	Dyes: Reactive Blue 21 and Sodium Dodecyl Sulfate; Membrane: NP010 (Microdyn Nadir); Operating conditions: TPM = 10 bars, 25 °C.	COD and dye removal of 97% and 96%; Water flux of 15.4 LMH for 1 h; NF process could produce reclaimed water.	[83]
RO	Dye: Biologically treated textile wastewater; Membrane: 8-inch DOW FILMTEC <sup>™</sup> FORTILIFE <sup>™</sup> CR100 RO element; Operating conditions: TPM = 8–20 bars, recovery of 70%, 30–40 °C.	Water flux of 19 LMH; COD, color, and conductivity parameters within required limits for reuse in the dyeing process.	[90]
Two-step UF	Dye: raw textile wastewater; Membranes: UF-GH 2 kDa and UF-PT 5 kDa (GE Osmonics); Operating conditions: TPM = 2–4 bars, volume reduction factor of 2.5–10, 25 °C.	TOC removal of >70%; Water flux of 4.5–16 LMH; The proposed treatment produced salty water for reuse.	[82]

Table 4. Membrane-based methods for dye-containing effluent treatment for water reuse.

Treatment Scheme	Features	Main Findings	Reference
Ozonation + <b>UF + RO</b>	Dye: Biologically treated textile wastewater; Membranes *; Operating conditions: UF, TPM *; RO, TPM = 15–25 bars.	COD and color removal of >99% in RO; The reuse rate of reclaimed water is equal to 86.6%; UF treatment cost = 0.04 USD m <sup>-3</sup> and RO treatment cost = 0.14 USD m <sup>-3</sup> ; The proposed treatment produced high-quality water for reuse.	[91]
<b>RO</b> + EO + BMED	Dye: raw textile wastewater; Membrane: SG1812 (GE Power and Water Technologies) Operating conditions: TPM = 12 bars, recovery of 70%, 25 °C.	COD and color removal of >70 and 100%; Water flux of 19 LMH; The energy demand of combined RO-EO-BMED is equal to 24.6 kWh m <sup>-3</sup> RO permeate meets the requirements	[81]

Table 4. Cont.

Note: \*, Data not available. BMED: bipolar membrane electrodialysis. COD: chemical oxygen demand. EO: electrochemical oxidation. LMH: L m<sup>-2</sup> h<sup>-1</sup>. NF: nanofiltration. RO: reverse osmosis. SBR: sequencing batch reactor. TPM: transmembrane pressure. TOC: total organic carbon. UF: ultrafiltration.

for water reuse.

Likewise, manufacturing membranes with enhanced proprieties aiming for higher dye rejection and water flux during wastewater purification is a hot topic for research [92–94]. Gnanasekaran et al. [95] fabricated NF membranes incorporating MIL-100 (Fe) into chitosan (CS) using a film-casting technique. The prepared CS/MIL-100 (Fe) composite membrane attained improved water flux from 5.2 to 52.5 LMH with a 99% rejection of Methylene Blue and Methyl Orange dyes.

#### 5.3. Sludge Valorization

The excess sludge from UASB reactors requires dewatering, drying, stabilization, and/or disinfection for the final destination [96]. The dye sludge contains toxic chemicals, so its proper treatment must be guaranteed. Efforts have been made to recover added-value products from dye sludge (e.g., dyes, energy, salts, metals, and nutrients) [97], representing an exciting opportunity for economic savings and more sustainable operation in textile industries.

The AD of textile dye sludge has been extensively studied. In this case, sludge pretreatment to enhance organics solubilization and maximize biogas production is particularly important. Some pretreatments, such as thermal and alkaline, showed improvements in the AD performance of textile dyeing sludge. However, pretreated sludge did not perform as well in biomethane potential tests as expected [98,99]. In recent work, anaerobic codigestion (coAD) using food waste as a cosubstrate was evaluated with thermally pretreated digestate [100]. The biomethane yield increased by 20 to 40%. In addition, this work performed an energy balance. It showed that the electricity produced by biogas could satisfy the electric consumption of the wastewater-treatment facility and the coAD system with 57.69% and 41.78%, respectively.

Apart from using AD for sludge valorization, thermochemical processes were investigated. Yildirir and Ballice [101] treated textile biological sludges via hydrothermal gasification to produce fuel gas. The calorific value of the produced fuel gas was 24.3 MJ/Nm<sup>3</sup> after gasification (30 min of time reaction). Hydrothermal gasification is promising to convert wet sludge into clean fuel gas with high caloric value without any drying process. More research in thermochemical methods, including pyrolysis and torrefaction, might contribute to dye-sludge valorization. This work reviewed studies on UASB reactors for dye-wastewater valorization. UASB reactors offer a dual advantage of degrading dye pollutants in wastewater while also serving as a significant source of bioenergy. Color and COD removal efficiencies of 50–97% and 60–90% are reported in bench-scale studies. A biogas yield of 1.48–2.70 L d<sup>-1</sup> in UASB, which treats dye-containing effluents, is reported. The successful adoption of this technology depended on establishing a dense granular sludge bed. Therefore, mechanisms of sludge granulation and control methods to reduce the start-up of UASB reactors should be developed. Cotreatment of dye wastewater and locally available substrate could increase biogas productivity in UASB reactors. In addition, integrating UASB with membrane processes (e.g., UF and NF) and pretreatment methods of dye wastewater and sludge are promising routes for dye-waste valorization. Future studies on these combined systems are recommended. Moreover, the technoeconomic evaluation of biogas and water production while treating real dye-containing wastewater in full-scale applications is critical to promoting UASB technology in textile industries.

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