

Review

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Review

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 the final destination. The up-flow anaerobic blanket (UASB) reactor application is an eco-friendly and cost-competitive treatment. The present study briefly reviews the UASB application for dye wastewater valorization. Bioenergy and clean water production potential during dye-containing wastewater treatment are emphasized to promote resource recovery in textile industries. Efficiencies of color and chemical oxygen demand of 50–97% and 60–90% are reported in bench-scale UASB studies. A biogas yield of 0.36–36.04 L d⁻¹ in UASB, which treats dye-containing effluents, is documented. Bioenergy production and water reuse allow environmental and economic benefits. However, data on full-scale UASB treating dye wastewater are missing. Besides, studies on combined systems integrating membrane processes, such as ultrafiltration and nanofiltration, and pretreatment of wastewater and sludge for improvements in biogas production might realize the complete potential for resource recovery of UASB technology.

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]. Therefore, dye wastewater should be preferentially treated using eco-friendly technologies. In this context, biotreatments are cost-competitive, give total mineralization or non-hazardous byproducts, and consume less water than physical and oxidative methods [2].

Biotreatments occur under aerobic or anaerobic conditions, as the products of aerobic treatment are biomass, CO₂, and H₂O. In contrast, the main product of anaerobic treatment is biogas (composed of CH₄ and CO₂ 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 [3].

UASB's compact design and low cost own several applications, such as brewing and beverage, distilleries, food, pulp and paper, food processing, chemical industries, landfill leachate, and textile effluents [4,5]. A full-scale 1800 m³ d⁻¹ UASB treating sewage wastewater was monitored for 35 weeks. Organic matter removal was higher than 90%, and biogas yield was estimated at 0.2 m³ per kg of chemical oxygen demand (COD) removed [6]. 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 biogas production of 0.312 m³ d⁻¹ [7].

Recently, investigators have examined the factors affecting the UASB reactor's performance, conventional configuration, and derivatives [3]. Some parts of our previous work discussed treatability findings of UASB in textile wastewater purification [8]. However, research has not yet analyzed 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 [9].

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^{-1}) [10]. 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 [11,12]. The typical height-diameter ratio of UASB reactors ranges from 0.2 to 0.5 [13]. A 3-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 [14]. 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_4 , followed by CO_2 and traces of other compounds [15]. Figure 1 presents a 3D-designed UASB reactor for wastewater treatment and biogas production.

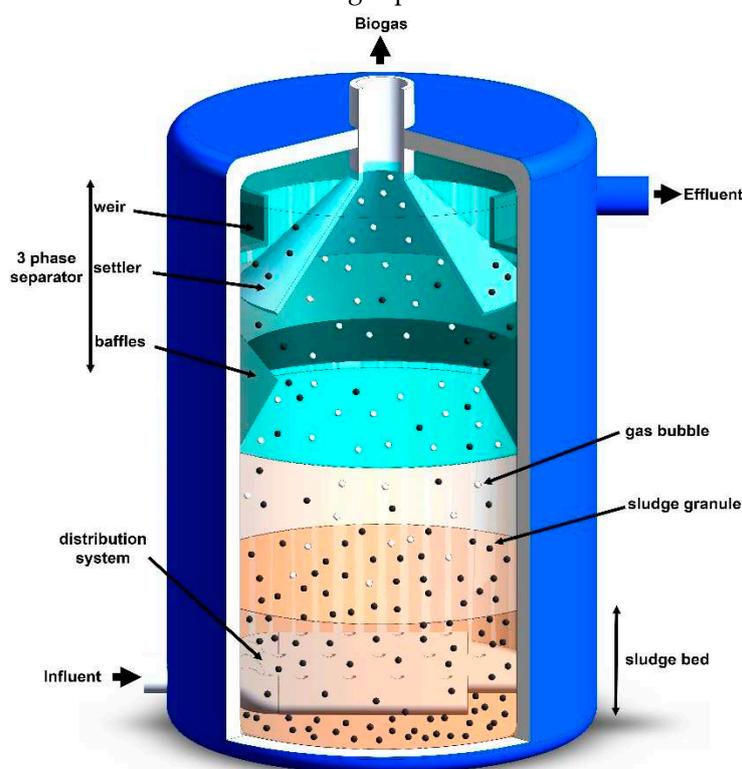


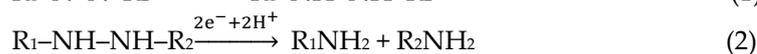
Figure 1. UASB Reactor in 3D designed for effluent treatment and biogas production.

The 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. 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. 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 [16]. Hulshoff Pol et al. [17] 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. Despite substantial investigative efforts, mechanisms governing the formation of anaerobic granules remain elusive. Besides long reactor start-up, gas leakage, and corrosion-related issues require periodic monitoring and maintenance for effective treatment outcomes [18].

3. Mechanisms and influencing parameters in textile decolorization in UASB reactors

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 [19]. 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 generated hydrazo intermediates undergo reductive cleavage, resulting in uncolored aromatic amines as byproducts, as shown in Equation 2 [20].



Where R_1 and R_2 are aryls or heteroaryl groups.

The anaerobic process is divided into 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 [21].

Biodecolourisation 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 biodecolourisation are *Methanosarcina archaea*, *Clostridium*, *Enterococcus*, *Pseudomonas*, *Bacillus*, *Aeromonas*, *Enterococcus*, *Desulfovibrio*, and *Desulfomicrobium* bacteria. [8].

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 style="list-style-type: none"> High dye concentration might affect microorganism growth rate, enzymatic activity, and biodescolorization performance. High dye dosage is linked to high salinity and biotoxicity, which 	<ul style="list-style-type: none"> 450 mg dye L⁻¹ could decrease the granular sludge porosity and strength, reduce its settling ability, and inhibit methanogenic activity. >300 mg L⁻¹ sulfate dosage might inhibit methanogens. 	[22,23]

	<ul style="list-style-type: none"> reduces microbial activity. Salinity decreases biomass size and hydrophobicity, affecting biodegradation and sludge settling. Complex dye structure might hamper the mineralization of the molecules by microorganisms. 	
Electron donors and redox mediators	<ul style="list-style-type: none"> C-sources are required in anaerobically dye removal. Redox mediators increase biodescolorization kinetic as they accelerate electron transfer from C-source to dye. 	<ul style="list-style-type: none"> Riboflavin and sulfonated compounds such as anthraquinone sulfonate and disulfonated anthraquinone are usually employed as redox mediators. Riboflavin (0.00175 mg L⁻¹) and yeast extract (500 mg L⁻¹) increased as C-sources increased dye decolorization in UASB reactors. [24–27]
pH	<ul style="list-style-type: none"> It affects microorganisms' growth rate, enzymatic activity, and biodescolorization efficiency. In an anaerobic environment, methanogens grow efficiently in the pH range of 6.0–8.0 and are sensitive to pH fluctuation. 	<ul style="list-style-type: none"> Azo dye Direct Black G biodescolorization of 97% at pH 8.0, 79% at pH 11.0; and 81% decolorisation at pH 4.0 after 48 h of residence time. [28,29]
Temperature	<ul style="list-style-type: none"> It affects the microbial community and methanogen activity. 	<ul style="list-style-type: none"> The optimum temperature for biodecolourisation ranges from 30 to 55°C, and exceeding this range could harm the syntrophic relationship among anaerobic microorganisms. [27,30]
Organic loading rate (OLR.)	<ul style="list-style-type: none"> High-OLR can affect methanogens and inhibit methane production in UASB reactors. 	<ul style="list-style-type: none"> It was reported that methane production efficiency was 75% at OLR of 2.4 kg COD m⁻³ d⁻¹. [31–33]

Hydraulic retention time (HRT.)	<ul style="list-style-type: none"> Lower than optimal HRT leads to the misdevelopment of granular sludge and acidification. Higher than optimal HRT results in low reactor components and biomass washout utilization. 	<ul style="list-style-type: none"> Temperature adjustment and effluent recirculation can alleviate the harmful effects of high OLR. Dye removal was reported at 67% at 16 h HRT and 55% at HRT of 96 h. Optimal HRT ranges from 5 to 20 h.
		¹ and 38% at 22.5 kg COD m ⁻³ d ⁻¹ . [34–36]

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 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 HRT ranging from 5 to 20 hours, and an OLR of 2 to 15 kg COD m⁻³ d⁻¹ is recommended. 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 [37].

4. Dye wastewater treatment in UASB reactors

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. The 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.

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

Scheme	Scale	UASB reactor conditions	Dye compounds			Treatability results		Reference
			Type	Name	Concentration/ Amount	Color	COD	
UASB reactor	Lab	Continuous mode, 27°C, HRT 24 h, OLR*	Azo dye	Reactive Red 2	50 mg L ⁻¹	51%	89%	[38]
UASB reactor + Activated sludge process	Lab	Continuous mode, 16°C – 29°C, HRT 24h, OLR*	Azo dye	Yellow Gold Remazol	50 mg L ⁻¹	85%	67–88%	[39]

UASB reactor + shallow polishing pond	Lab	Continuous mode, 16°C – 29°C, HRT 24h, OLR*	Azo dye	Yellow Gold Remazol	50 mg L ⁻¹	85%	67–88%	[39]
UASB reactor	Lab	Continuous mode, temperature *, TRH 24h, OLR*	Azo dye	Red Bronze	40–325 mg L ⁻¹	75–94%	60–91%	[40]
UASB reactor + Aerated bioreactor	Lab	Continuous mode, 37±1 °C, H.R.T. 6h, OLR 12.97 kg C.O.D. m ⁻³ d ⁻¹	Azo dye	2-Naphthol Red	0.1 g L ⁻¹	96%	85.6%	[41]
UASB reactor + microaerated UASB reactor	Lab	Continuous mode, 25.0±1.4°C, HRT*, OLR 1.27–1.50 kg m ⁻³ d ⁻¹	Azo dye	Direct Black 22	0.6 mM	70–78%	67–72%	[42]
UASB reactor + shallow polishing pond	Lab	Continuous mode, 16–29°C, HRT 24 h, OLR*	–	Real textile wastewater	–	50%	80%	[43]
UASB reactor	Lab	Continuous mode, 27 – 29°C, HRT 24 h, OLR 6.20 kg COD m ⁻³ d ⁻¹		Simulated wastewater containing Remazol blue RSP	12.5 mg L ⁻¹	97.37±3.62%	76.69±2.83%	[44]
UASB reactor + SBR	Lab	Intermittent mode, 35°C, HRT 48 h, OLR 0.74 – 0.90 kg COD m ⁻³ d ⁻¹		Real textile wastewater	–	87.7%	90.4%	[45]

Note: *, Data not available. COD = Chemical oxygen demand, UASB = Up-flow anaerobic sludge blanket, HRT = Hydraulic retention time, OLR = Organic loading rate, and SBR = Sequencing batch reactor.

Bahia et al. [43] used an integrated UASB-shallow pond system in continuous feeding, achieving color and COD removal rates of 50% and 80%. Saleem et al. [45] 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 non-feeding 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 bio-transformation. This completion occurs through hydroxylation or cleavage of the ring using oxidative enzymes such as laccase, phenoxidase, and peroxidase [46].

In another work, Carvalho et al. [42] proposed using a microaerated UASB reactor to effectively 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%. Besides, 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

Add-value product extraction from dye industry wastes has been investigated, and a comprehensive review of resource recovery of colored effluents was recently published [47]. 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.

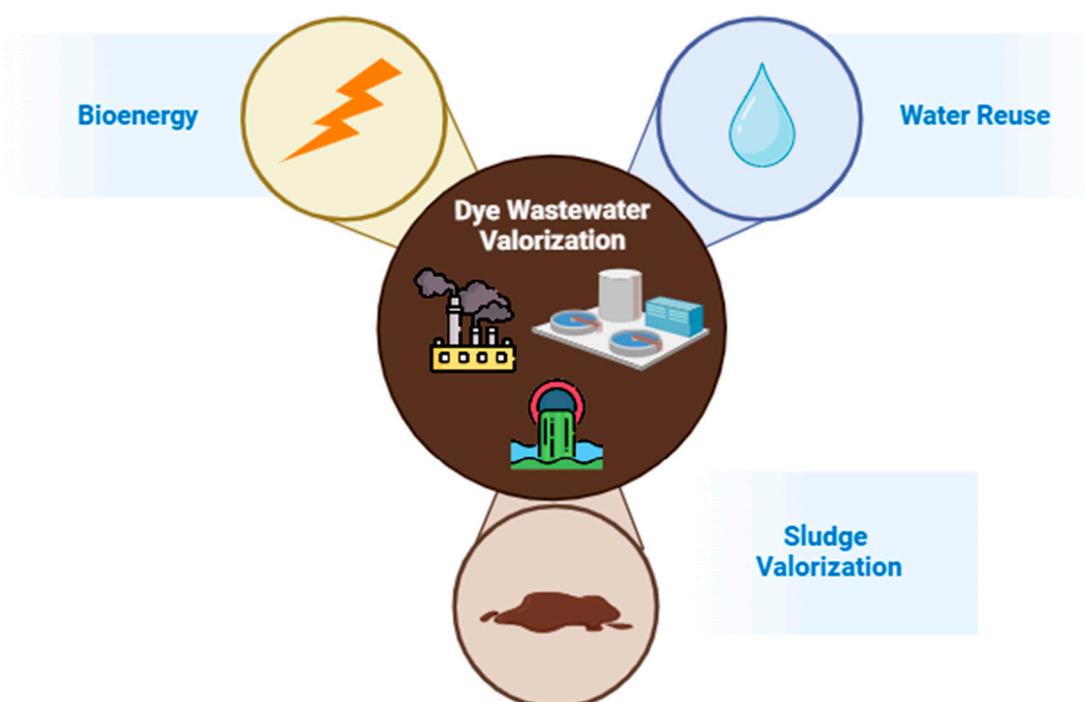


Figure 2. Dye wastewater valorization for sustainability in textile industries.

5.1. Bioenergy production

Anaerobic technology offers a 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 [48].

The dual potential of anaerobic technology helps in wastewater valorization and contributes to sustainable energy production [49]. Katal et al. [50] conducted experiments using a lab-scale UASB reactor to treat textile effluent and measure the biogas production yield. They achieved maximum biogas productivity of 36 L per day at an HRT of 50 hours, with a biomethane content of 79%. Other bench-scale studies reported biomethane production rates ranging from 0.36 to 2.7 L per day [36,51,52] (Table 3).

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

Scheme	UASB reactor conditions	Dye compound	Biogas production	Reference
UASB reactor	Temperature of 37°C, H.R.T. 20h, OLR 3.86 kg C.O.D. m ⁻³ d ⁻¹	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 ⁻¹ (70%CH ₄ , v/v)	[52]
UASB + CSTR reactor	Temperature of 37°C, HRT 3 – 30h, OLR 2 – 15 kg COD m ⁻³ d ⁻¹	Real textile wastewater	0.36 – 0.94 L d ⁻¹	[51]
UASB reactor	Temperature of 37°C, HRT 18.3h, OLR 0.286 kg m ⁻³ d ⁻¹	Red Congo azo dye (100 mg L ⁻¹)	2.0 – 2.7 L d ⁻¹	[36]
UASB reactor	Temperature of 33°C, H.R.T. 50h, O.L.R. 12 kg C.O.D. m ⁻³ d ⁻¹	Real textile wastewater	36.04 L d ⁻¹ (79%CH ₄ , v/v)	[50]
UASB reactor + aerobic system	Temperature of 37±1°C, H.R.T. 6h, OLR 12.97 kg C.O.D. m ⁻³ d ⁻¹	2-Naphthol Red (100 mg L ⁻¹)	3.86 L CH ₄ m ⁻³ d ⁻¹	[41]

Industrial treatment facilities have a high energy demand [53]; thus, UASB technology offers opportunities for reducing treatment costs while treating wastewater. Gadow and Li [41] 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 m³ of effluent. A maximum methane yield of 13.3 mmol CH₄ g⁻¹ COD d⁻¹ 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 CH₄ g⁻¹ COD was recorded during the treatment of synthetic dye wastewater [54]. Apart from bioenergy recovery and 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 [55].

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 [56,57]. UV pretreatment improved biogas production 2.7-fold compared with non-pretreated effluent and increased methane yield in anaerobic digestion (AD) of methylene blue [56].

A recent review analyzed landfill leachate pretreatment methods coupled with AD to enhance biogas production [58]. Landfill leachate, as dye effluent, is a complex and inhibitory wastewater for anaerobic processes [58–60]. 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. This 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 [61]. 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 [62]. This system could produce 0.97 tons of clean water at 24.7 kWh per m³ 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 [63,64]. Hybrid bio-oxidation and NF processes performed well in removing soluble dyes and surfactants. They could significantly reclaim water from textile wastewater [64]. In this work, the authors highlighted that integrating both treatments to produce recycled water is needed, corroborating the necessity of combining recovering technologies. In another research, Erkanli et al. [63] analyzed different configurations of the two-stage UF process for recovering water from real dye wastewater. Two-stage UF using membranes of molecular-weight cut off of 2 kDa produced high-quality water to an extent that allows reuse in fabric dyeing. As estimated 200 – 400 liters 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 [18]. Thus, it can be a potential candidate to be integrated with UASB technology aiming to produce clean water.

5.3. Sludge valorization

The excess sludge from UASB reactors requires dewatering, drying, stabilization, and/or disinfection for the final destination [65]. The dye sludge contains toxic chemicals, so its proper treatment must be guaranteed. Efforts have been made to recover add-value products from dye sludge (e.g., dyes, energy, salts, metals, and nutrients) [66], representing an exciting opportunity for economic savings and more sustainable operation in textile industries.

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 it was [67,68]. In recent work, anaerobic co-digestion (coAD) using food waste as a co-substrate was evaluated with thermally pretreated digestate [69]. The biomethane yield increased by 20 to 40%. Besides, 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. Yildirim and Ballice [70] treated textile biological sludges via hydrothermal gasification to produce fuel gas. The calorific value of the produced fuel gas was 24.3 MJ/Nm³ 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.

6. Conclusions

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 0.36–36.04 L d⁻¹ 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. Integrating UASB with membrane processes (e.g., UF and NF) and pretreatment methods of dye wastewater and sludge are the most promising routes for dye waste valorization. Future studies on these combined systems are recommended. Moreover, the techno-economic evaluation of biogas and water production while treating real dye-containing wastewater in full-scale applications is critical to promoting UASB technology in dye industries.

Author Contributions: Conceptualization, RA; methodology, RA; validation, RA and CSG; formal analysis, RA and CSG; investigation, RA and CSG; data curation, RA and CSG; writing—original draft preparation, RA; writing—review and editing, RA and CSG All authors have read and agreed to the published version of the manuscript.

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