Rapid hydrogen generation from cotton wastes by mean of dark fermentation

Gaweł Sołowski¹ · Izabela Konkol¹ · Marwa Shalaby² · Adam Cenian¹

Received: 9 January 2020 / Accepted: 23 July 2020 / Published online: 28 July 2020

© The Author(s) 2020 **OPEN**

Abstract

Dark fermentation of textile wastes is discussed in the paper. In the experiment cotton wastes were fermented. Before fermentation the cotton was hydrolyzed using 0.1 M HCl acidic solution. The inoculum was pretreated by means of heat shock for 0.5 h at 105 °C. The fermentation was carried out under mesophilic conditions at a load of 5 g VSS/L, and pH 5. Oxygen was added in small quantities during fermentation. The oxygen flow rates (OFR) were between 0.3 and 1.0 mL/h. The fermentation was carried out for a few days at temperatures between 40 and 43 °C. Hydrogenesis prevailed at the lower temperature (40 °C) and methanogenesis at the higher (43 °C). Conversion of cotton waste to methane (3.4%) was slightly higher than conversion to hydrogen (2.6%). The highest hydrogen production was obtained for OFR 0.8 mL/h and the percentage of hydrogen in biogas was 43%. At higher temperatures (43 °C) no hydrogen production was observed

Keywords Dark fermentation · Hydrogen · Methane · Cotton

1 Introduction

Cotton waste constitute a high proportion of textile wastes, which account for 50% of world biomass waste [1]. Its utilization, complying with demands of circular economy, can solve a problem especially in the Middle East, including Syria [2] but also in Europe [3]. Poland generates around 236 661 Mg of cotton waste annually, which is clearly a huge amount to process [4]. On the other hand, there exists a high demand for energy [5] and industrial raw materials [6], that increasingly will not be easily met by fossil resources [7]. So, the role of renewable fuels like hydrogen produced from biomass will grow continuously [8]. Cotton plantations can contribute to global world sustainability [9], oil from its seeds may serve as biofuel or lubricant [10, 11], and cotton wastes may be transformed into biodiesel [12, 13] or as an additive to other biofuels [14, 15] with properties similar to FAME [16]. Cotton wastes

are used for PET or insulating material production [17] or serve as important substrate for pyrolysis [16, 17].

An interesting issue, undertaken in this research, is related to the possibility of using cotton waste, including cotton stalk [18] as a source of hydrogen. Fast depletion of fossil fuels [19] means that there is a growing need to seek renewable ways of hydrogen production, an important resource for chemistry [20] and carbon free fuel [8]. Taherzadeh works [2, 21] proved that cotton wastes are a good source of methane generation, but the process includes expensive chemical pretreatment using NMMO, highly concentrated sulfuric acid or sodium hydroxide [2, 22]. But the alternative dark fermentation (DF) process may lead to biohydrogen production [23]. DF process, a truncated anaerobic digestion (without final methanisation), converts substrates into gaseous products like hydrogen, carbon dioxide and volatile organic acids (e.g. butyric, acetic or propionic) [24].

✉ Gaweł Sołowski, gsolowski@imp.gda.pl | ¹Institute of Fluid-Flow Machinery of Polish Academy of Sciences, Gdańsk, Poland. ²Department of Chemical Engineering and Pilot Plant, National Research Center in Cairo, Cairo, Egypt.

According to initial assessments, cotton waste could be a potential source of hydrogen [25]. The aim of this research is to determine the optimal conditions for hydrogen production in the DF process, and its industrial scale viability, with a focus on the effect of temperature variation in the mesophilic range on hydrogen production from cotton waste. Similar studies were performed by Chandra et al. [26] for rice straw anaerobic fermentation. As in earlier research [27], related to sour cabbage fermentation, microaeration (which stimulates hydrogen production) was applied.

Lack of information on successful scaling up of DF processes [28] (except possibly [29] leads Carrillo-Reyes et al. [30] to state that even lower efficiency (15% of practical hydrogen yield) could be considered as feasible. In most cases the optimal dark fermentation process proceeds under acidic conditions (pH ~ 5.0), at mesophilic temperature and using stressed inoculum. However, for some substrates like potato waste [31] or cotton stalk, optimal conditions [18] are closer to pH neutral or basic. Therefore, it is important to clarify these differences and determine conditions (including pH, temperature, microaeration rate, etc.) that are optimal for hydrogen DF production for various available substrates and to scale up process into profitable range. Besides, it is also checked whether the upper temperature limit of mesophilic process (for various aeration rates) stimulates or inhibits hydrogen production [26]. At the same time we check how various considered conditions influence methane production. The studied issues as well as the investigated substrates are rarely discussed, or contradicting results are presented (e.g. pH level). The results obtained here were compared with data from earlier studies [32] for sour cabbage and cotton waste at higher pH value (7.54) and without inoculum stress.

2 Materials and methods

The fermentation process of cotton wastes was performed in batch reactors of volume 2 dm³ with working volume 1.2 dm³. As inoculum, sludge from an agricultural biogas plant (Pomerania Region) was used, and 5 g VSS/L (VSS = volatile suspended solids) were applied to each batch of cotton waste. A substrate (load 5 g VSS/L), obtained from 100% cotton lab coats, was milled and hydrolysed using 0.1 M HCl acidic solution for 2 h.

Initially, the inoculum was treated by heat shock for 0.5 h at 105 °C. Later, the initial pH 7.84 was lowered by HCl to pH 5.0 and applied to the DF process. The substrate was pretreated analogous to a Nasirian procedure for DF of wheat straw [33] but a 0.1 M solution of H₂SO₄, which was the most efficient in the case of hydrogen

production from their substrate [34], was replaced here by cheaper HCl (for pH 3).

Then cotton and inoculum samples were added to the reactors. The temperatures 40 and 43 °C (maximum temperature for mesophilic conditions [35] were maintained in the reactors. The oxygen flow rates (OFR) range from 0 to 1.0 mL/h on average was provided for the fermentation process in the reactors. The oxygen was added twice a day (for approximately 2 s) until the fermentation process was stopped.

All the experiments were carried out in triplicate, (see scheme of setup in Fig. 1). The biogas production was determined using the Owen method [36]. The qualitative and quantitative assessment of the gases produced was performed using a gas chromatograph (GC) with a thermal conductivity detector and argon as a carrier (gas flow rate was 0.6 mL/h). A Silco packed column Restek® with characteristics of 2 m/2 mm ID 3 mm OD Silica was used. In order to determine the right amount according to Standard Methods [37], of fresh matter (FM) of inoculum and the substrate total solids (TS) [%FM] and volatile solids (VSS) [%TS] (determined to the [38]. The results are presented in Table 1.

Cotton wastes characteristics (TS, VSS) were determined before and after hydrolysis and after fermentations and drying. Cotton wastes were sieved by net with mesh 2 mm after hydrolysis and after DF (taking it from bottom of reactors). The changes of VSS were used to determined degree of utilization of cotton waste in agreement with norms [37, 38].

Fig. 1 Fermentation setup used in experiment: 1. glass reactors, 2. cylindrical vessel collecting biogas, 3. water bath chamber enabling mesophilic conditions in reactors

Table 1 Characteristics of inoculums and substrate

Material	pH	TS	VSS
Boiled inoculum	7.84	1.5%±0.03%	37.91% TS±1.22%
Boiled inoculum after fermentation at 40 °C	5.5	1.8%±0.03%	42.96% TS±1.02%
Boiled inoculum after fermentation at 43 °C	5.9	1.2%±0.02%	42.22% TS±1.12%
Cotton wastes	–	98%±0.02%	98.29% TS±1.2%
Cotton waste after hydrolysis	–	95%±0.03%	96% TS±1.03%
Cotton wastes after fermentation at 43 °C and drying [38]	5.5	92.5%±0.02%	93.5% TS±1.02%
Cotton wastes after fermentation at 40 °C and drying [38]	5.9	91.4%±0.02%	92.6% TS±1.02%

Fig. 2 Total biogas and hydrogen production obtained during fermentation process stimulated by pretreated inoculum, at $T=40\text{ }^{\circ}\text{C}$ and OFR (oxygen flow rate) 0.3, 0.8 and 1 mL/h

3 Results

The GC spectra allowed determination of methane, hydrogen, carbon monoxide, carbon dioxide and nitrogen (from process initiation procedure) concentrations. The fermentation process with pretreated inoculum (initial pH ~ 5) was continued for 5 h (later DF process had stopped) at temperature $40\text{ }^{\circ}\text{C}$, while at $43\text{ }^{\circ}\text{C}$ the fermentation process was continued for 45 h.

In the case of boiled inoculum (pH ~ 8.5) biogas wasn't generated, so process at temperature of $43\text{ }^{\circ}\text{C}$ was checked only for pretreated inoculum (pH ~ 5). In the case of process performed at temperature $40\text{ }^{\circ}\text{C}$ the biogas contained carbon dioxide, nitrogen and hydrogen (no methane generation was observed, in contrast to fermentation at $43\text{ }^{\circ}\text{C}$). The hydrogen production strongly depends on OFR, see Fig. 2 for oxygen flow rates: 0.3, 0.8 and 1 mL/h.

The total biogas production equaled:

Fig. 3 Total biogas and methane production fermentation process stimulated by pretreated inoculum, $T=43\text{ }^{\circ}\text{C}$ for OFR ~ 0.3 mL/h and anaerobic conditions

- 0.31 dm^3 containing 18.9% H_2 (0.06 dm^3) for OFR ~ 0.3 mL/h;
- 0.39 dm^3 containing 42% H_2 (0.168 dm^3) for OFR ~ 0.8 mL/h;
- 0.31 dm^3 containing 10.3% H_2 (0.03 dm^3) for OFR ~ 1 mL/h.

After 5 h DF process terminated at $40\text{ }^{\circ}\text{C}$ for all OFR rates. Thus the results are not presented in the form of cumulative hydrogen production versus time as is usual [39]. The optimum OFR value lies between 0.3 and 1 mL/h. Fast and effective hydrogen production has already been observed in dark fermentation but with lower rate e.g. in ref.[40], although it continued longer, even for 80 h.

At fermentation temperature $43\text{ }^{\circ}\text{C}$, biogas comprises: methane, carbon dioxide and nitrogen. No hydrogen was registered. The strictly anaerobic cf an oxygen flow rate ~ 0.3 mL/h conditions were compared—see Fig. 3. Under strict anaerobic conditions 0.92% of methane (0.003 dm^3) in 0.34 dm^3 of biogas was found after 45 h fermentation. In the case of OFR ~ 0.3 mL/h, higher concentration

of 1.7% of methane (0.006 dm^3) in biogas (0.34 dm^3) was measured.

The pH value in the case of fermentation at 40°C changed from 5.0 to 5.5, under condition of free pH evolution (no pH control measures applied—see e.g. [41]. In the case of fermentation at temperature 43°C the pH value increased to 5.9 after 45 h for all OFR values, i.e. pH value is higher in comparison to that at 40°C .

The presented results showed that fermentation process (after heat shock pretreatment of inoculum) led to higher hydrogen production than in [42] (and no methane generation) at the lower temperature 40°C . Actually at 43°C hydrogen was not generated in significant amount and methane at concentration below 2% of biogas.

4 Discussion of results

In this study DF proceeds under standard biogas inoculum condition in contrast to isolated bacterial consortium like in Li et al. [18]. It is worth adding that in the presented experiment there were not any nutrients like agar plates [43] or salts applied [44]. Moreover, the discussed process of hydrogen production is rather simple and not demanding; it proceeds without any bacteria isolation or hydrolysate separation. Thus, it is more economical than those already reported [45–48].

The process of cotton digestion slows down at higher temperature 43°C (the rate is 9 times lower) when compared with the case at 40°C . The observed at 40°C hydrogen yields per day were higher than those reported by Li et al. [18]. The hydrogen production measured here was 6 times higher than in [49]. On the other hand biomass conversion is low (2.6%), 0.9% lower than in production of methane from cotton waste at 43°C —see e.g. [22].

The experiments pointed to the importance of inoculum pretreatment (thermal shock) and process temperature for hydrogen production rate. When DF temperature is higher methane production prevails, bacterial consortium is stimulated to generate methane (although in low quantities if compared to [22] even at low pH [50]. Similar results were reported by Chandra et al. [26] for rice straw anaerobic fermentation. Also Del et al. [51] concludes that stable mesophilic condition with temperature from the range $36\text{--}40^\circ\text{C}$ is necessary to keep process of hydrogen production continuing.

It was also found that in some cases after termination of hydrogen generation, methanogenesis continues e.g. when food and garden waste mixture is fermented at 37°C [52] or xylose fermented at 30°C [53]. However, it was not the case for cotton fermented here at 40°C .

The results obtained here were compared with DF data for other substrates—see Table 2. The hydrogen yields obtained here were larger than from cow dung pretreated using 2% HCl solution [54], microalgal biomass [55] or sunflower pretreated by 4% HCl [56] but less than 50% of hydrogen obtained from cotton stalk after pretreatment in 4% H_2SO_4 and 121°C for 30 min. or 80% less than from aspen wood treated using 2% NaOH. Highest yields were obtained for cassava with addition of α -amylase and gluco-amylase after thermal pretreatment at 112°C for 15 min and using activated sludge by heat shock for 30 min.

5 Conclusions

Cotton waste at load VSS 5 g/L at pH 5.0 was found to be a good source of hydrogen by mean of dark fermentation, when inoculum is stressed. The process is fast (short) and efficient. The discussed process of hydrogen production is rather simple and not demanding; it proceeds without any bacteria isolation or hydrolysate separation. Thus it is more economical than those already reported.

However, rather low conversion of cotton waste was observed, e.g. conversion of cotton waste in methanogenesis 3.4% was higher than in hydrogenation process 2.6% and lower increase of bacterial biomass was registered. So, the process can be used for utilization of cotton wastes as fast preliminary stage of cotton waste utilization followed by methanisation.

The highest production was observed at OFR 0.8 mL/h and DF process temperature 40°C i.e. 0.168 dm^3 of hydrogen—42% of biogas content. Besides it was found that temperature is a relevant parameter of stimulating or inhibiting hydrogen production in DF. Methanogenesis can revive again after repeated heat shock treatment and temperature of fermentation increase to 43°C in the case of cotton.

Besides, it should be underlined that that the applied 5 g VSS/L should be probably increased in future studies to check the effects of load increase. The phenomena will be investigated further.

Table 2 Comparison of hydrogen production from different substrates (batch process)

Type of substrate/pretreatment of substrate	Hydrogen Yield (mL H ₂ /g subs.)	Type of inoculum	Process conditions (g/L/°C/pH)	Source
Cotton waste/in 0.1 M HCl for 1 h	34	Heat shocked at 105 °C for 30 min	(5/40/5.0 microaeration)	This study
Cotton stalk/in 4% H ₂ SO ₄ at 121 °C for 30 min	72.7	Bacteria from wild carp instentine/ isolation broth medium	40/37/8	[18]
Sour cabbage/no pretreatment	4.8	Untreated sludge	(10/38/7.9 microaeration)	[57]
Reed canary grass/in 3% HCl, 121 °C, 90 min	36	Untreated sludge	(5/38/3.4–6	[45]
Mixture of hydrothermally pretreated asbestos to glucose in proportion 1:6	1.3	Activated sludge with stressing using 2-Bromoethanesulfate	(5/35/5.45)	[46]
Grass-comminuted 10 g/l 20 mesh/in 0.5% NaOH, 105 °C for 3 h	4.39	Sludge from cracked cereal baked for 2 h and boiled for 30 min	(5/35/7.0)	[58]
Cassava/ α -amylase + gluco-amylase – /heat gelatinising/ at 112 °C for 15 min	240	Activated sludge heat shocked for 30 min	(10/35/7.0)	[54]
Cow dung compost/2%HCl and 8-min microwaves	0.5	Dung compost heat shocked at 130 °C for 2 h	(25/36/7.0)	[56]
Sunflower stalk /in 4% HCl at 170 °C for 1 h	2.3	Activated sludge heat-shocked at 90 °C for 15 min	(5/35/5.5)	[55]
Microalgal biomass/ centrifuged	20.9	Activated sewage sludge/by keeping at 4 °C in solution 15 g/L peptone from casein, 5 g/L peptone from soymeal, and 5 g/L sodium chloride for 3 day and then heat shocked for 15 min at 121 °C and pressure 1.4 bar	(10/30/7)	[59]
Aspen wood/in 2% NaOH	195	Hot spring culture, heat shocked at 60 °C in pH 6.0	(1.23/50/7)	[60]
Depackaging wastes with glucose 1:6 /heat treatment	1.4	Activated sludge with stressing using 2-Bromoethanesulfate 10 mM	(4/37/7)	[61]

Funding The research was funded from grant of Institute of Fluid-Flow Machinery, Polish Academy of Science in Gdansk FBW-44—Solowski and The National Centre for Research and Development 'New renewable energy technologies for sustainable development of rural areas and low-carbon agriculture' BIOSTRATEG3/344128/12/NCBR/2017.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

Open Access This article is licensed under a Creative Commons Attribution 4.0 International License, which permits use, sharing, adaptation, distribution and reproduction in any medium or format, as long as you give appropriate credit to the original author(s) and the source, provide a link to the Creative Commons licence, and indicate if changes were made. The images or other third party material in this article are included in the article's Creative Commons licence, unless indicated otherwise in a credit line to the material. If material is not included in the article's Creative Commons licence and your intended use is not permitted by statutory regulation or exceeds the permitted use, you will need to obtain permission directly from the copyright holder. To view a copy of this licence, visit <http://creativecommons.org/licenses/by/4.0/>.

References

1. Sasaki C, Kiyokawa A, Asada C, Nakamura Y (2019) Glucose and valuable chemicals production from cotton waste using hydro-thermal method. *Waste Biomass Valoriz* 10:599–607
2. Aslanzadeh S, Rajendran K, Jeihamipour A, Taherzadeh MJ (2013) The effect of effluent recirculation in a semi-continuous two-stage anaerobic digestion system. *Energies* 6:2966–2981
3. Halimi MT, Hassen MB, Sakli F (2008) Cotton waste recycling: Quantitative and qualitative assessment. *Resour Conserv Recycl* 52:785–791
4. Solowski G (2016) Theoretical potential of hydrogen production from textiles wastes in pomeranian region by means of dark fermentation. In: Noch T, Saczuk J, Wesołowska A (eds) *Globalizacja a regionalna ochrona srodowiska*, 1st edn. Wydawnictwo GSW, Gdańsk, pp 313–317 (ISBN 9788389762795)
5. Byrne E, Kovacs K, Van Niel EWJ, Willquist K, Svensson SE, Kreuger E (2018) Reduced use of phosphorus and water in sequential dark fermentation and anaerobic digestion of wheat straw and the application of ensiled steam-pretreated lucerne as a macronutrient provider in anaerobic digestion. *Biotechnol Biofuels* 11:1–16
6. Méndez-Vázquez MA, Gómez-Castro FI, Ponce-Ortega JM, Serafin-Muñoz AH, Santibañez-Aguilar JE, El-Halwagi MM (2017) Mathematical optimization of a supply chain for the production of fuel pellets from residual biomass. *Clean Technol Environ Policy* 19:721–734

7. Nikolaidis P, Poullikkas A (2017) A comparative overview of hydrogen production processes. *Renew Sustain Energy Rev* 67:597–611
8. Kumari D, Singh R (2018) Pretreatment of lignocellulosic wastes for biofuel production: a critical review. *Renew Sustain Energy Rev* 90:877–891
9. Prabakar D, Suvetha KS, Manimudi VT, Mathimani T, Kumar G, Rene ER, Pugazhendhi A (2018) Pretreatment technologies for industrial effluents: critical review on bioenergy production and environmental concerns. *J Environ Manage* 218:165–180
10. Sarıdemir S, Ağbulut Ü (2019) Combustion performance vibration and noise characteristics of cottonseed methyl ester–diesel blends fuelled engine. *Biofuels* 10(1080/17597269):1667658
11. Ağbulut Ü, Sarıdemir S, Albayrak S (2019) Experimental investigation of combustion, performance and emission characteristics of a diesel engine fuelled with diesel–biodiesel–alcohol blends. *J Brazilian Soc Mech Sci Eng* 41:1–12
12. Ağbulut Ü, Sarıdemir S, Karagöz M (2020) Experimental investigation of fusel oil (isoamyl alcohol) and diesel blends in a CI engine. *Fuel* 267:117042
13. Ağbulut Ü, Sarıdemir S (2018) A general view to converting fossil fuels to cleaner energy source by adding nanoparticles. *Int J Ambient Energy*. <https://doi.org/10.1080/01430750.2018.1563822>
14. Ağbulut Ü, Karagöz M, Sarıdemir S, Öztürk A (2020) Impact of various metal-oxide based nanoparticles and biodiesel blends on the combustion, performance, emission, vibration and noise characteristics of a CI engine. *Fuel*. <https://doi.org/10.1016/j.fuel.2020.117521>
15. Ağbulut Ü, Bakır H (2019) *Düzce University Journal of Science & Technology*. Düzce Univ J Sci Technol 4:25–36
16. Guo M, Song W, Buhain J (2015) Bioenergy and biofuels: history, status, and perspective. *Renew Sustain Energy Rev* 42:712–725
17. Sandin G, Peters GM (2018) Environmental impact of textile reuse and recycling e A review. *J Clean Prod* 184:353–365
18. Li Y, Zhang Q, Deng L, Liu Z, Jiang H, Wang F (2018) Biohydrogen production from fermentation of cotton stalk hydrolysate by *Klebsiella* sp. WL1316 newly isolated from wild carp (*Cyprinus carpio* L.) of the Tarim River basin. *Appl Microbiol Biotechnol* 102:4231–4242
19. Koniuszewska I, Korzeniewska E, Harnisz M, Czatkwowska M (2020) Intensification of biogas production using various technologies: a review. *Int J Energy Res*. <https://doi.org/10.1002/er.5338>
20. Kumar V, Longhurst P (2018) Recycling of food waste into chemical building blocks. *Curr Opin Green Sustain Chem* 13:118–122
21. Patinvoth RJ, Taherzadeh MJ (2019) Fermentation processes for second-generation biofuels. Elsevier Inc., Amsterdam (**ISBN 9780128151624**)
22. Jeihanipour A, Aslanzadeh S, Rajendran K, Balasubramanian G, Taherzadeh MJ (2013) High-rate biogas production from waste textiles using a two-stage process. *Renew Energy* 52:128–135
23. Sołowski G, Konkol I, Cenian A (2019) Perspectives of hydrogen production from corn wastes in Poland by means of dark fermentation. *Ecol Chem Eng S* 26:255–263
24. Detman A, Mielecki D, Pleśniak Ł, Bucha M, Janiga M, Matyasik I, Chojnacka A, Jędrysek MO, Błaszczak MK, Sikora A (2018) Methane-yielding microbial communities processing lactate-rich substrates: a piece of the anaerobic digestion puzzle. *Biotechnol Biofuels* 11:116
25. Hallenbeck PC, Abo-Hashesh M, Ghosh D (2012) Strategies for improving biological hydrogen production. *Bioresour Technol* 110:1–9
26. Chandra R, Takeuchi H, Hasegawa T (2012) Hydrothermal pretreatment of rice straw biomass: a potential and promising method for enhanced methane production. *Appl Energy* 94:129–140
27. Sołowski G, Hrycak B, Czyłkowski D, Konkol I, Pastuszek K, Cenian A (2019) Hydrogen and methane production under conditions of dark fermentation process with low oxygen concentration. In: Jibin K, Kalarikkal N, Thomas S, Nzihou A (eds) *Re-Use and recycling of materials solid waste management and water treatment*. River Publisher, Gistrup, pp 263–272 (**ISBN 9788770220583**)
28. Bahry H, Abdallah R, Chezeau B, Pons A, Taha S (2019) Biohydrogen production from carob waste of the Lebanese industry by dark fermentation fermentation. *Biofuels*. <https://doi.org/10.1080/17597269.2019.1669862>
29. Cieślak M, Dach J, Lewicki A, Smurzyńska A, Janczak D, Pawlicka-Kaczorowska J, Boniecki P, Cyplik P, Czekala W, Józwiakowski K (2016) Methane fermentation of the maize straw silage under meso- and thermophilic conditions. *Energy* 115:1495–1502
30. Carrillo-Reyes J, Tapia-Rodríguez A, Buitrón G, Moreno-Andrade I, Palomo-Briones R, Razo-Flores E, Aguilar Juárez O, Arreola-Vargas J, Bernet N, Maluf Braga AF et al (2019) A standardized biohydrogen potential protocol: an international round robin test approach. *Int J Hydrogen Energy* 44:26237–26247
31. Sekoai PT, Ayeni AO, Daramola MO (2019) Parametric optimization of biohydrogen production from potato waste and scale-up study using immobilized anaerobic mixed sludge. *Waste and Biomass Valoriz* 10:1177–1189
32. Sołowski G, Hrycak B, Czyłkowski D, Pastuszek K, Konkol I, Cenian A (2018) Hydrogen and methane production under conditions of dark fermentation process with low oxygen concentration. In: Sabu T (ed) *Proceedings of the International Conference on Reuse and Recycling (ICRM 2018)*, Kottayam, Kerala, India
33. Nasirian N, Almassi M, Minaei S, Widmann R (2011) Development of a method for biohydrogen production from wheat straw by dark fermentation. *Int J Hydrogen Energy* 36:411–420
34. Ghimire A, Valentino S, Frunzo L, Trably E, Escudé R, Pirozzi F, Lens PNL, Esposito G (2015) Biohydrogen production from food waste by coupling semi-continuous dark-photofermentation and residue post-treatment to anaerobic digestion: a synergy for energy recovery. *Int J Hydrogen Energy* 40:16045–16055
35. Sołowski G, Shalaby MS, Abdallah H, Shaban AM, Cenian A (2018) Production of hydrogen from biomass and its separation using membrane technology. *Renew Sustain Energy Rev* 82:3152–3167
36. Logan BE, Oh SE, Kim IS, Van Ginkel S (2002) Biological hydrogen production measured in batch anaerobic respirometers. *Environ Sci Technol* 36:2530–2535
37. Moriarty K (2013) Feasibility study of anaerobic digestion of food waste in St. Bernard, Louisiana: a study prepared in partnership with the environmental protection agency for the re-powering America's land initiative: siting renewable energy on potentially contaminate. National Renewable Energy, Golden, pp 1–51
38. Sluiter A, Hames B, Ruiz R, Scarlata C, Sluiter J, Templeton D (2008) Determination of sugars, byproducts, and degradation products in liquid fraction process samples laboratory analytical procedure (LAP) issue date: 12/08/2006. National Renewable Energy Laboratory, Golden
39. Sołowski G, Konkol I, Hrycak B, Czyłkowski D (2019) Hydrogen and methane production under conditions of anaerobic digestion of key-lime and cabbage wastes. *Agritech* 39:243–250
40. Pecorini I, Baldi F, Iannelli R (2019) Biochemical hydrogen potential tests using different inocula. *Sustain* 11:1–17
41. Atasoy M, Eyice O, Schnürer A, Cetecioglu Z (2019) Volatile fatty acids production via mixed culture fermentation: revealing the link between pH, inoculum type and bacterial composition. *Bioresour Technol* 292:121889

42. Sołowski G, Konkol I, Cenian A (2020) Methane and hydrogen production from cotton wastes in dark fermentation process under anaerobic and microaerobic conditions. *Biomass Bioenergy* 2020.
43. García Depraect O, Muñoz R, van Lier JB, Rene ER, Diaz-Cruces VF, León Becerril E (2020) Three-stage process for tequila vinasse valorization through sequential lactate, biohydrogen and methane production. *Bioresour Technol* 307:123160
44. Keskin T, Arslan K, Nalakth Abubackar H, Vural C, Eroglu D, Karaalp D, Yanik J, Ozdemir G, Azbar N (2018) Determining the effect of trace elements on biohydrogen production from fruit and vegetable wastes. *Int J Hydrogen Energy* 43:10666–10677
45. Lakaniemi AM, Koskinen PEP, Nevatalo LM, Kaksonen AH, Puhakka JA (2011) Biogenic hydrogen and methane production from reed canary grass. *Biomass Bioenergy* 35:773–780
46. Spasiano D (2018) Dark fermentation process as pretreatment for a sustainable denaturation of asbestos containing wastes. *J Hazard Mater* 349:45–50
47. Pütün A, Özbay N, Önal E, Pütün E (2005) Fixed-bed pyrolysis of cotton stalk for liquid and solid products. *Fuel Process Technol* 86:1207–1219
48. Asfand S, Shah Y, Zeeshan M, Zohaib M, Ahmed N, Iqbal N (2019) Co-pyrolysis of cotton stalk and waste tire with a focus on liquid yield quantity and quality. *Renew Energy* 130:238–244
49. Sołowski G, Konkol I, Cenian A (2020) Methane and hydrogen production from cotton waste by dark fermentation under anaerobic and microaerobic conditions. *Biomass Bioenergy* 138:105576
50. Fagbohunbe MO, Onyeri C, Adewale C, Semple KT (2019) The effect of acidogenic and methanogenic conditions on the availability and stability of carbon, nitrogen and phosphorus in a digestate. *J Environ Chem Eng* 7:103138
51. Del YA, Acosta A, Alvarez LH, Bernardo R, Reyes G, Teresa M, González G, Carrillo J (2020) Biocatalysis and Agricultural Biotechnology Addition of electron shuttling compounds and different pH conditions for hydrogen production by a heat-treated sludge. *Biocatal Agric Biotechnol* 23:101507
52. Blasco L, Kahala M, Tampio E, Vainio M, Ervasti S, Rasi S (2020) Effect of Inoculum pretreatment on the composition of microbial communities in anaerobic digesters producing volatile fatty acids. *Microorganisms* 8:581
53. Mockaitis G, Bruant G, Guiot SR, Peixoto G, Foresti E, Zaiat M (2020) Acidic and thermal pre-treatments for anaerobic digestion inoculum to improve hydrogen and volatile fatty acid production using xylose as the substrate. *Renew Energy* 145:1388–1398
54. Su H, Cheng J, Zhou J, Song W, Cen K (2009) Improving hydrogen production from cassava starch by combination of dark and photo fermentation. *Int J Hydrogen Energy* 34:1780–1786
55. Monlau F, Aemig Q, Trably E, Hamelin J, Steyer JP, Carrere H (2013) Specific inhibition of biohydrogen-producing *Clostridium* sp. after dilute-acid pretreatment of sunflower stalks. *Int J Hydrogen Energy* 38:12273–12282
56. Fan YT, Zhang YH, Zhang SF, Hou HW, Ren BZ (2006) Efficient conversion of wheat straw wastes into biohydrogen gas by cow dung compost. *Bioresour Technol* 97:500–505
57. Sołowski G, Hrycak B, Czyłkowski D, Pastuszak K, Cenian A (2018) Oxygen sensitivity of hydrogenesis' and methanogenesis'. In: Pikoń K, Lucyna C (eds) *Contemporary problems of power engineering and environmental protection 2017*. Gliwice, Department of Technologies and Installations for Waste Management Copyright, pp 157–159 (ISBN 978-83-950087-1-9)
58. Cui M, Shen J (2012) Effects of acid and alkaline pretreatments on the biohydrogen production from grass by anaerobic dark fermentation. *Int J Hydrogen Energy* 37:1120–1124
59. Batista AP, Gouveia L, Marques PASS (2018) Fermentative hydrogen production from microalgal biomass by a single strain of bacterium *Enterobacter aerogenes*—effect of operational conditions and fermentation kinetics. *Renew Energy* 119:203–209
60. Phummala K, Imai T, Reungsang A, Chairattanananokorn P, Sekine M, Higuchi T, Yamamoto K, Kanno A (2014) Delignification of disposable wooden chopsticks waste for fermentative hydrogen production by an enriched culture from a hot spring. *J Environ Sci (China)* 26:1361–1368
61. Noblecourt A, Christophe G, Larroche C, Fontanille P (2018) Hydrogen production by dark fermentation from pre-fermented depackaging food wastes. *Bioresour Technol* 247:864–870

Publisher's Note Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.