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Article

Batch Reactor Design and Conception at Laboratory Scale for Solid-State Anaerobic Digestion: Practical Comparison Between 3D-Printed Digesters and Conventional Methods

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Abstract: Solid-state anaerobic digestion (SS-AD) is a promising technology for treating organic waste and producing renewable energy. This study explores the feasibility of using 3D printing to rapidly design cost-effective laboratory-scale digesters for optimization experiments. Batch reactors were designed using fused deposition modeling (FDM) with polylactic acid (PLA) and stereolithography (SLA) with High Temp V2 resin. PLA had a negligible impact on methane yields, while raw SLA resin positively influenced methanogenic potential, likely due to residual isopropanol used in post-processing, causing a 19% increase in CH₄ yield. The performance of the 3D-printed reactors was compared to that of a conventionally machined PMMA reactor using cattle manure as a substrate, showing comparable methane yields and process stability. Three-dimensional printing technologies have demonstrated remarkable efficiency in designing laboratory-scale digesters, with a 70% cost reduction for SLA technology and an 80% reduction in design time compared to conventional reactors designed by plastics processing, while maintaining comparable biogas production. FDM technologies with PLA have shown that they are not suitable for these uses. This study demonstrates the potential of additive manufacturing to accelerate SS-AD research and development. However, care must be taken in material selection and post-processing to avoid introducing experimental bias.

Keywords: 3D printing; conception; anaerobic digestion; solid state



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1. Introduction

In the contemporary landscape of sustainable energy solutions, anaerobic digestion (AD) has emerged as a promising avenue for addressing pressing environmental and energy challenges. AD offers a dual benefit of waste management and renewable energy generation by harnessing the natural processes of microbial degradation in the absence of oxygen. This process involves the conversion of organic substrates into biogas, primarily composed of methane and carbon dioxide, along with nutrient-rich digestate, which can serve as a valuable soil amendment. AD has garnered considerable attention from researchers, policymakers, and industry stakeholders worldwide with the increasing recognition of

its potential to mitigate greenhouse gas emissions, reduce dependence on fossil fuels, and divert organic waste from landfills. By the end of 2018, Europe had registered over 18,200 units. Cogeneration from biogas accounted for the generation of 63.5 TWh of electricity, while biomethane injected into the natural gas network amounted to 22.78 TWh [1]. Moreover, the environmental benefits of AD are numerous and include a reduction in greenhouse gas emissions and the diversion of organic waste from landfill disposal [2]. Two categories of anaerobic digestion (AD) processes are distinguished based on the total solid content (TS): liquid-state AD and solid-state anaerobic digestion (SSAD). SSAD involves substrates with a solid content exceeding 15%, making it particularly suitable for the degradation of materials with heterogeneous compositions and high solid contents [3,4]. While SSAD has gained traction in recent years, it remains less prevalent than liquid-state AD, primarily due to limited understanding and the operational complexities required to maintain stable production [2].

Despite the potential of SSAD technologies, their widespread adoption has been hindered by technical, economic, and scientific challenges, including incomplete understanding of the process dynamics, hydrodynamics, and susceptibility to inhibitory factors [2,3]. These scientific hurdles have yet to be overcome, despite many research studies in this field. Depending on the studies carried out, optimization experiments and the design of experiments may be necessary to increase our knowledge of the anaerobic digestion process [5], but the lab scale is restrictive in terms of the number of experiments that can be carried out. Lab-scale digester configurations are usually continuously stirred tank reactors (CSTRs) and batch digesters constructed using conventional manufacturing methods, such as machining or plastics processing [6]. While these methods have served as the foundation of AD research and practice, laboratory equipment often has limitations in terms of design flexibility, scalability, and cost-effectiveness. In addition, depending on demand, the build time for machining laboratory-scale reactors can be very long, ranging from several months to a year.

In the context of these challenges, additive manufacturing has emerged as a disruptive technology with the potential to revolutionize the design and manufacture of laboratory-scale reactors. Unlike traditional manufacturing techniques, which rely on subtractive processes to shape materials into the desired form, 3D printing—the most popular type of additive manufacturing—enables the layer-by-layer deposition of materials based on digital design specifications. This additive approach offers unprecedented flexibility in the creation of complex geometries, intricate internal structures, and customized designs, overcoming many of the limitations associated with conventional manufacturing methods. Various 3D printing technologies are currently being investigated for application to laboratory-scale SSAD reactor manufacturing, including fused deposition modeling (FDM) and stereolithography (SLA). Each of these techniques has its own advantages and limitations, ranging from print resolution and material compatibility to structural integrity and cost-effectiveness. FDM, which involves the extrusion of thermoplastic filaments layer by layer, is known for its affordability and ease of use, but it can have limitations in terms of achieving high-resolution prints, the characteristics of the materials used, and utilizing hermetic parts. On the other hand, SLA involves the use of photopolymerization processes to polymerize liquid resin into solid objects with great precision, making it suitable for producing complex, detailed components, but it is more expensive, its post-processing process is more time-consuming, and the process involves solvents that can adversely affect anaerobic digestion. These technologies are widely used in research, particularly for the design of photo-reactors [7,8], micro-reactors [9,10], and flexible sensors [11]. Regarding anaerobic digestion, there are very few studies on the use of 3D-printed reactors, but some papers have addressed the use of 3D printing for determining residence times or for determining concentrations of ammonia and sulfide in samples [12,13].

Despite the potential of 3D printing for accelerating the development of SSAD technologies, there are still significant gaps in the literature regarding its application in this field. The majority of studies have focused on the use of 3D printing for specific components or sampling devices, rather than the design and fabrication of complete reactors. Furthermore, the impact of different 3D printing materials and processes on the anaerobic digestion process itself has not been thoroughly investigated. There is a need for a comprehensive evaluation of the feasibility of using 3D printing technologies for the rapid and cost-effective design of laboratory-scale SSAD reactors, as well as an assessment of their performance compared to conventionally manufactured reactors.

To address these gaps, the present study aims to explore the potential of 3D printing technologies, specifically FDM and SLA, for the design and the manufacturing of laboratory-scale SSAD reactors. The novelty of this research lies in its systematic approach to evaluating the impact of 3D printing materials and processes on the anaerobic digestion process, as well as the direct comparison of the performance of 3D-printed reactors with that of a conventionally machined reactor with the same shape and volume for conducting cost-effective optimization experiments. By providing insights into the feasibility and effectiveness of 3D printing for SSAD reactor design, this study seeks to contribute to the advancement of sustainable energy solutions and the optimization of anaerobic digestion processes.

2. Materials and Methods

2.1. Substrate and Inoculum Characteristics

Cattle manure (CM) is a widely used substrate for solid-state anaerobic digestion. It has a high solid total content, good biomethane potential, and low risk of inhibition. CM was chosen as a substrate for experiments and sampled from the farm of the Uni-LaSalle Polytechnic Institute (Beauvais, France). The CM did not undergo any preliminary treatment. Liquid manure (LM) was used as an *inoculum* and was sampled from the post-digester at the Ferti-Oise site (Coudun, France). This digester processes energy crops, crop residues, and agricultural wastes at a rate of 30,000 tons per year, which allowed us to obtain an *inoculum* concentrated in microorganisms that was perfectly suited for the digestion of CM. Finally, the polylactic acid (PLA) used in the construction of the test reactors was biodegradable at 60 °C. This is why the experiments detailed in the next section were conducted under mesophilic conditions, even though PLA has also been characterized as a substrate for thermophilic conditions. All analyses were carried out in triplicate. The total solid content (TS) and volatile solid content (VS) of the *inoculum*, CM, and LM were assessed through drying at 105 °C for 24 h followed by combustion at 550 °C for 2 h, as per APHA standards [14]. The pH of the inoculum was measured using a pH meter from Mettler Toledo, Switzerland. Additionally, the total volatile fatty acid content (FOS) and buffer capacity (TAC) were determined through titrations using sulfuric acid with an automatic titrator from Mettler Toledo, Switzerland. These analyses were conducted before the experiments, and the results are presented in Table 1.

Table 1. Chemical characteristics of substrate, *inoculum*, and PLA used.

Raw Material	TS	VS	pH	FOS	TAC	BMP
Unit	%	% _{TS}	-	mg·L ⁻¹	mg·L ⁻¹	NL·kg _{VS} ⁻¹
CM (38 °C)	22.00 ± 0.15	87.70 ± 0.02	7.51 ± 0.01	-	-	142.9 ± 12.4
LM (38 °C)	3.62 ± 0.02	66.10 ± 0.04	7.84 ± 0.01	5.5 ± 0.2	12.0 ± 0.1	73.0 ± 0.8
LM (60 °C)	3.62 ± 0.02	66.10 ± 0.04	7.84 ± 0.01	5.5 ± 0.2	12.0 ± 0.1	17.3 ± 0.8
PLA (60 °C)	99.70 ± 0.01	99.98 ± 0.01	-	-	-	513.6 ± 21.2

2.2. Characteristics of 3D-Printed Materials

The 3D-printed reactors were designed using two different materials based on the design method: the reactors printed via FDM were made of white PLA from Ultimaker (Utrecht, The Netherlands), and the reactors printed using SLA were made of High Temp V2 resin from Formlabs (Sommerville, MA, USA). The FDM 3D printer used was Ultimaker S5, and the SLA 3D printer was the form3 printer from Formlabs. PLA was chosen for FDM printing because it is an easy-to-print, biodegradable, and cost-effective material. It is a thermoplastic derived from renewable resources, such as corn starch or sugarcane, making it an environmentally friendly option [15]. PLA has a low glass transition temperature (T_g) of around 60 °C and a melting temperature (T_m) of approximately 150–160 °C [16]. These thermal properties make it suitable for printing at relatively low temperatures, reducing energy consumption and potential thermal degradation during the printing process. However, PLA has certain limitations: it cannot withstand temperatures above 50 °C for extended periods and it is permeable to gases and moisture [17]. The PLA used in this study was a mixture of various Ultimaker PLA residues ground after recovery. It has a glass transition temperature of around 60 °C, is highly permeable to water and oxygen, and has a density of 1.25 g/cm³. Therefore, it is theoretically suitable for use as a material in the design of laboratory-scale anaerobic digestion reactors under mesophilic conditions but must be sealed beforehand with a layer of inert varnish. Regarding the reactor design, the reactors were coated with a waterproof and inert varnish available commercially: XTD-3D. Preliminary tests revealed that this varnish is non-toxic to microorganisms and resistant to the operational conditions associated with methanization.

Concerning SLA printing, High Temp V2 resin from Formlabs was chosen because it is transparent, airtight, and reputedly thermoresistant, with a glass transition temperature of 238 °C. SLA printing involves using a laser to selectively cure and harden a photopolymer resin layer by layer, resulting in high-resolution prints with smooth surfaces [18]. High Temp V2 resin is a proprietary material developed by Formlabs that is specifically designed for applications requiring high heat resistance and dimensional stability. It has a tensile strength of 61.5 MPa, a tensile modulus of 3.3 GPa, and an elongation at break of 2.2% [19]. Furthermore, SLA printing ensures the sealing of the printed parts, making it suitable for anaerobic digestion applications where gas leakage must be prevented. Reactors were printed and cleaned with isopropanol before being treated under a UV lamp at 60 °C for 1 h using Form Cure from Formlabs (post-treatment). This post-treatment process helps optimal mechanical properties to be achieved and removes any residual uncured resin. The reactors designed with this material can thus be reused for subsequent experiments within the laboratory.

2.3. Characterization of the Interaction Between the 3D-Printed Material and the AD Phenomenon

Experiments were conducted on a small scale and under various comparative conditions to study the interactions between the materials comprising the 3D-printed reactors and the anaerobic digestion process. An AMPTS (Automatic Methane Potential Test System) from Bioprocess Control (Lünd, Sweden) was used to conduct these experiments with working volumes of 400 mL and gas heads of 100 mL. The AMPTS is a widely used system for the determination of biomethane potential (BMP) and anaerobic biodegradability of organic substrates [20]. It consists of three main components: a water bath with 15 reactor bottles, a CO₂ absorption unit, and a gas volume measuring device. The reactor bottles are made of glass and have a total volume of 500 mL, with a working volume of 400 mL. The bottles are sealed with a rubber septum and an aluminum crimp cap, ensuring an airtight environment for anaerobic digestion. In this study, three bottles were filled with LM alone (control), three with LM + cellulose (reference), three with LM + cellulose + PLA, and three

with LM + cellulose + High Temp V2 resin before and after post-treatment. Cellulose (Avicel PH-101, Sigma-Aldrich, St. Louis, MO, USA) was used as a reference substrate to assess the performance of the anaerobic digestion process. The bottles containing PLA and High Temp V2 resin were prepared to evaluate the potential impact of these materials on the anaerobic digestion process. A total of 20 g of PLA was added to the bottles after grinding into flakes with an average thickness of 0.5 mm, while 20 g of resin was added in the form of plates printed with dimensions of 20 mm × 10 mm to maximize the surface contact area. The bottles were then incubated in the AMPTS water bath at 38 °C (mesophilic conditions) and 60 °C (thermophilic conditions) for 35 days. During this period, the methane production was continuously monitored using the AMPTS system, which automatically recorded the cumulative methane volume produced in each bottle. After 35 days of anaerobic digestion, the experiment was stopped, and the results were exported for further analysis.

2.4. Batch Reactor Specifications

The design of the reactors, as shown in Figure 1, in this project was carried out through a series of steps. The first step was the sizing of the reactors to ensure proper immersion of the solid substrate under study, good percolation of the liquid phase through the solid mass, easy temperature control while ensuring the representativeness of the phenomenon, and satisfaction of minimal space requirements for the laboratory [4,21,22]. The reactors were thus sized in a cylindrical shape with an internal diameter of 150 mm and a height of 150 mm. Recirculation of the reactors was ensured with an external removable peristaltic pump, and the temperature was controlled using a thermostatted oven for the 3D-printed reactors and with a thermostatted bath controlled via an immersion heater. All reactors were removable to allow easy cleaning and simpler intervention in case of percolation issues. An outlet with a ¼-inch thread was utilized to discharge the biogas produced by anaerobic digestion. An inlet at the bottom of the reactor and an outlet at the top of the reactor with a ½-inch thread were used to allow recirculation of the liquid phase using an external pump. A Tygon pipe was employed for liquid recirculation as well as for biogas evacuation to a manual gas meter described in Coutu et al. [6]. The lids were also 3D-printed, and a groove was used for 2 seals.

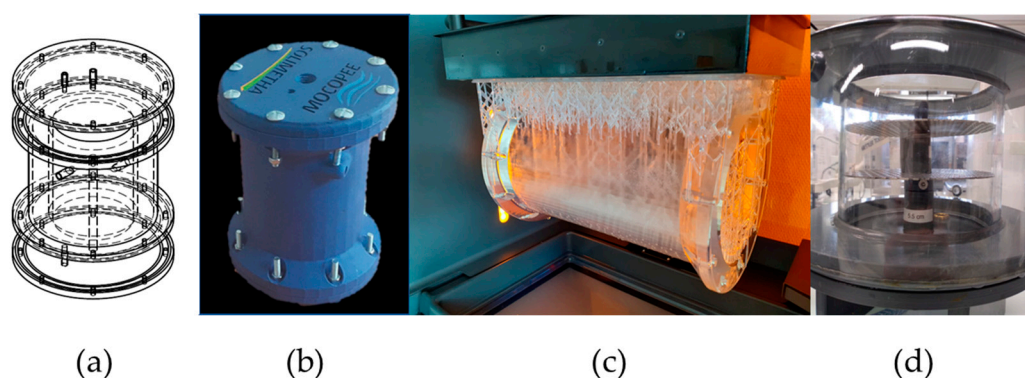


Figure 1. Designed reactor. (a) Computer-aided design of a reactor. (b) PLA 3D-printed reactor produced via FDM method. (c) High Temp V2 resin reactor 3D-printed via SLA method. (d) Machined reactor.

2.5. Processing and Finishing Methods for Machined Reactors

Once the technical solutions were defined, the reactors were designed through 3D modeling using CATIA V5 software, a powerful computer-aided design (CAD) tool widely used in various industries, including aerospace, automotive, and manufacturing industries [23]. The 3D model of the reactor was created using the part design and assembly design modules of CATIA V5, considering the specified dimensions, features, and constraints. The

model was then exported as a unique STL file, which is a standard file format for 3D printing and computer-aided manufacturing [24]. The machined reactor was designed to be made of polymethyl methacrylate (PMMA), also known as acrylic or plexiglass. PMMA is a transparent thermoplastic material with good thermal resistance, mechanical strength, and chemical stability [25]. Its transparency allows for the observation of the methanization phenomenon during digestion, which is a valuable feature for studying anaerobic digestion processes. The machined reactor included a double-envelope system to easily maintain its temperature, which is essential for maintaining stable anaerobic digestion conditions. The PMMA was cut to the required dimensions using a laser cutting machine, ensuring high precision and smooth edges. The reactor body, lid, and other components were then machined using a computer numerical control (CNC) milling machine, following the specifications of the 3D model. The machining process involved the removal of material from the PMMA sheets to create the desired shapes and features, such as the inlet and outlet ports, the double-envelope system, and the grooves for the seals. After machining, the reactor components were polished to achieve a smooth and transparent surface, which facilitated the observation of the anaerobic digestion process. The machined reactor components were then assembled using a special acrylic adhesive, ensuring strong and leak-proof bonds between the parts. The double-envelope system was sealed, and the inlet and outlet ports were fitted with the appropriate connectors and valves. The assembled reactor was then tested for leaks and functionality before being used in the experimental validation study. The entire design process of the machined reactors is presented in detail in the article by Coutu et al. [6], which serves as a reference for the design and fabrication of laboratory-scale anaerobic digestion reactors using conventional machining methods.

2.6. Experimental Validation Method

A unique experiment was carried out in triplicate to compare the use of the different reactor design technologies and verify the repeatability of the results obtained during the anaerobic digestion process. The experimental setup is shown in Figure 2, which illustrates the arrangement of the reactors, the substrate and inoculum loading, and the monitoring equipment. The substrate used in this study was CM, which is detailed in Table 1. For each reactor, 200 g of CM and 1 L of LM were loaded inside the digester. After loading the substrate and inoculum, the digesters were sealed, and the AD process was initiated. The reactors were operated in a batch mode, meaning that no additional substrate or inoculum was added during the experimental period. The temperature of the reactors was maintained in mesophilic conditions using a thermostatted oven for the 3D-printed reactors and a thermostatted bath with an immersion heater for the machined reactor. The immersion of the solid mass was set to 100%, ensuring that all the substrate was in contact with the inoculum and the liquid phase. The recirculation of the liquid phase was set to $0.64 \text{ L}\cdot\text{min}^{-1}$ for one minute per day, using an external peristaltic pump. This recirculation helped to homogenize the digester content, prevent the formation of dead zones, and improve the mass transfer between the solid and liquid phases [22]. The sealing of the reactors was also tested before the start of the experiment to ensure that no biogas leaks occurred during the anaerobic digestion process. This was achieved by pressurizing the reactors with nitrogen gas and checking for any pressure drops using a pressure gauge. During the experimental period, the biogas production was monitored using a manual gas meter, as described in Coutu et al. [6]. The gas meter consisted of a graduated bottle filled with demineralized water with a gas collection chamber called a Mariotte bottle, inspired by the Mariotte vase principle. The biogas produced in the reactors was directed to the Mariotte bottle, replacing the water with biogas. The volume of biogas produced was then

measured by reading the level of water inside the Mariotte bottle. The biogas composition was then analyzed using a gas chromatograph (GC).

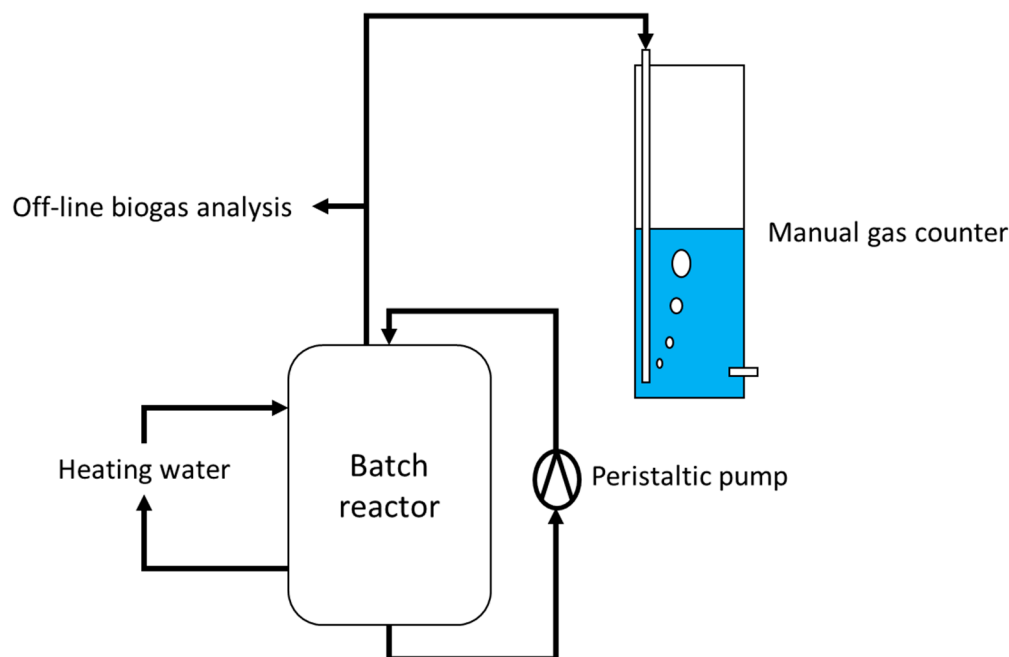


Figure 2. Experimental scheme of the experimental setup.

The experiment was conducted for a period of 35 days for AMPTS experiments, which is a typical duration for batch anaerobic digestion tests [26], and 17 days for batch experiments to obtain enough data to compare the reactors. During this period, the biogas production and composition were monitored daily, and the pH of the digestate was measured weekly using a pH meter. At the end of the experimental period, the reactors were opened, and the digestate was sampled for further analysis, including TS, VS, and other relevant parameters. The results obtained from this experimental validation study were then analyzed to compare the performance of the different reactor design technologies and to assess the repeatability of the anaerobic digestion process. The biogas production and composition data were used to calculate the specific methane yield of the CM, expressed in $L CH_4/kg Vs$. The methane yield is a key parameter for evaluating the efficiency of anaerobic digestion processes and for comparing the performance of different reactor designs [27]. The repeatability of the results was assessed by calculating the relative standard deviation (RSD) of the SMY values obtained from the triplicate reactors of each design. An RSD value below 10% is generally considered acceptable for anaerobic digestion experiments [26]. In addition to the biogas production and composition, parameters such as the pH, TS, and Vs of the digestate were analyzed to evaluate the stability and efficiency of the anaerobic digestion process. The results obtained from this experimental validation study provide valuable insights into the performance and repeatability of the different reactor design technologies as well as the suitability of 3D printing for the fabrication of laboratory-scale anaerobic digestion reactors.

3. Results

3.1. Characterization of the Interaction Between the 3D-Printed Material and the AD Phenomenon

The results of the study of the interaction between 3D-printed materials and the anaerobic digestion process are presented in Figure 3. The cumulative methane production curves obtained from the different experimental conditions are shown, along with the final methane yields achieved after 35 days of anaerobic digestion. The results demonstrate

that the inoculum used in this study (LM) was well-acclimated to mesophilic conditions (38 °C), as evidenced by the significant methane production observed in the control bottles containing only LM. The addition of cellulose to the bottles (LM + cellulose) resulted in higher methane production than the control, confirming the suitability of cellulose as a reference substrate for anaerobic digestion studies [26]. The presence of PLA in the bottles (LM + cellulose + PLA) had a negligible impact on the methane production, with cumulative methane curves and final methane yields like those obtained with LM + cellulose. This indicates that PLA, at the concentration and particle size used in this study, does not inhibit the anaerobic digestion process under mesophilic conditions. This finding agrees with previous studies that have reported the biodegradability of PLA under anaerobic conditions [28,29]. On the other hand, the presence of raw SLA resin in the bottles (LM + cellulose + raw resin) resulted in significantly higher methane production than the other conditions. The cumulative methane curve of the bottles containing raw resin showed a steeper slope and reached a higher final methane yield than the other conditions. This suggests that the raw SLA resin, which contains residual monomers and other compounds, may have a stimulatory effect on the anaerobic digestion process. This effect could be due to the release of easily biodegradable compounds from the resin, which can serve as additional substrates for the anaerobic microorganisms [30]. However, when the SLA resin was subjected to a post-treatment process (LM + cellulose + post-treated resin), the stimulatory effect on methane production was reduced but still higher than the control and the bottles containing PLA. This indicates that the post-treatment process, which involves washing the resin with isopropanol and curing it under UV light, removes some of the compounds responsible for the stimulatory effect. The residual compounds present in the post-treated resin may still have a positive impact on the anaerobic digestion process, albeit to a lesser extent than the raw resin. These results suggest that the choice of 3D printing material and post-processing methods can have a significant impact on the anaerobic digestion process when used in the fabrication of laboratory-scale reactors. PLA appears to be a suitable material for this application, as it does not inhibit the anaerobic digestion process and is biodegradable under anaerobic conditions. On the other hand, SLA resins may introduce a bias in the experimental results, especially if not properly post-processed to remove residual compounds that can stimulate methane production. The SLA resin tests are illustrated in Figure 3.

The analysis of standard deviations across the three cellulose-based systems reveals distinct behavioral patterns and the impact of different treatments. Cellulose demonstrates remarkable stability, reaching a maximum standard deviation of 6.35 early in the process before stabilizing at 3.57, indicating a well-controlled system. The addition of raw resin dramatically alters this stability, leading to consistently increasing variability that peaks at 28.95, suggesting that the resin introduces significant heterogeneity into the anaerobic digestion process, probably explained by the presence of excess liquid resin. Interestingly, when the cellulose–resin mixture is treated with isopropanol, an intermediate level of variability is observed, with a final standard deviation of 23.44. This indicates that the isopropanol cleaning process moderates the destabilizing effect of the resin, possibly by removing excess liquid resin material, though it does not fully restore the stability seen in the raw cellulose system because of the presence of isopropanol residue. These findings highlight how each processing step and additive significantly influences anaerobic digestion stability. Additional experiments were conducted under thermophilic conditions (60 °C) to further investigate the impact of 3D-printed materials on the anaerobic digestion process. The results of these experiments are presented in Figure 4, which shows the cumulative methane production curves and final methane yields obtained after 35 days of anaerobic digestion. Under thermophilic conditions, the methane production was generally higher

than under mesophilic conditions, which is consistent with the higher metabolic activity of thermophilic anaerobic microorganisms [31]. However, the impact of 3D-printed materials on the anaerobic digestion process was less pronounced than under mesophilic conditions.

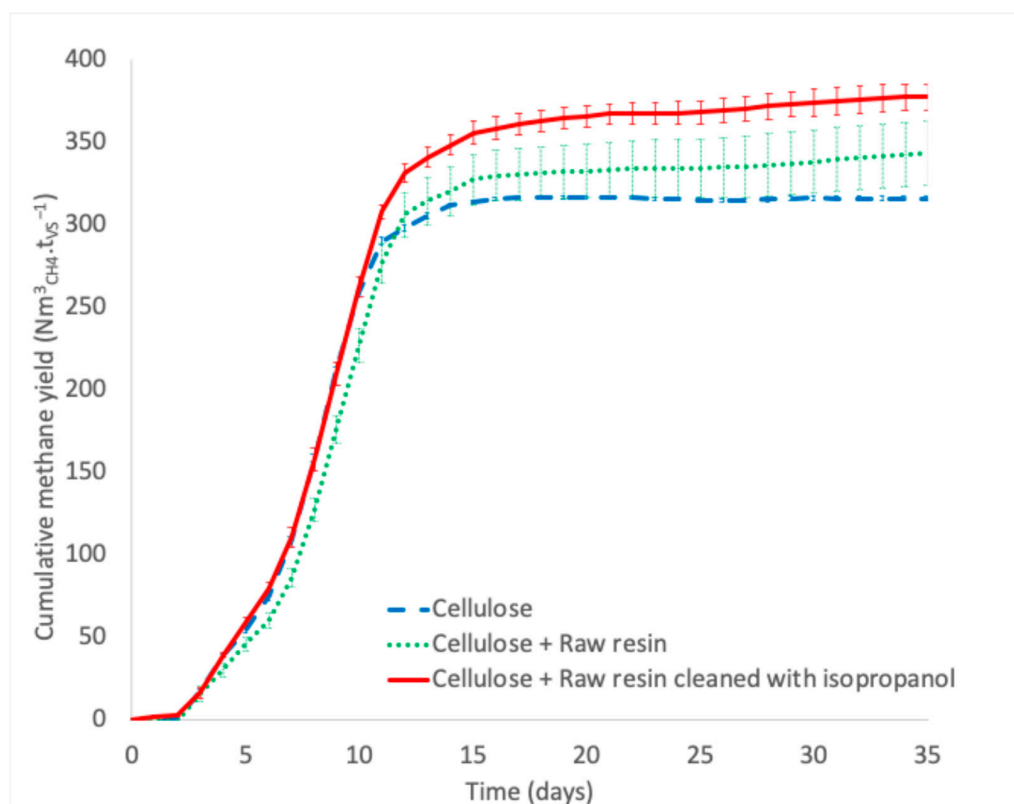


Figure 3. AMPTS experimental results of the interaction between High Temp V2 resin and the AD phenomenon to determine the impact of SLA resin on the anaerobic digestion process.

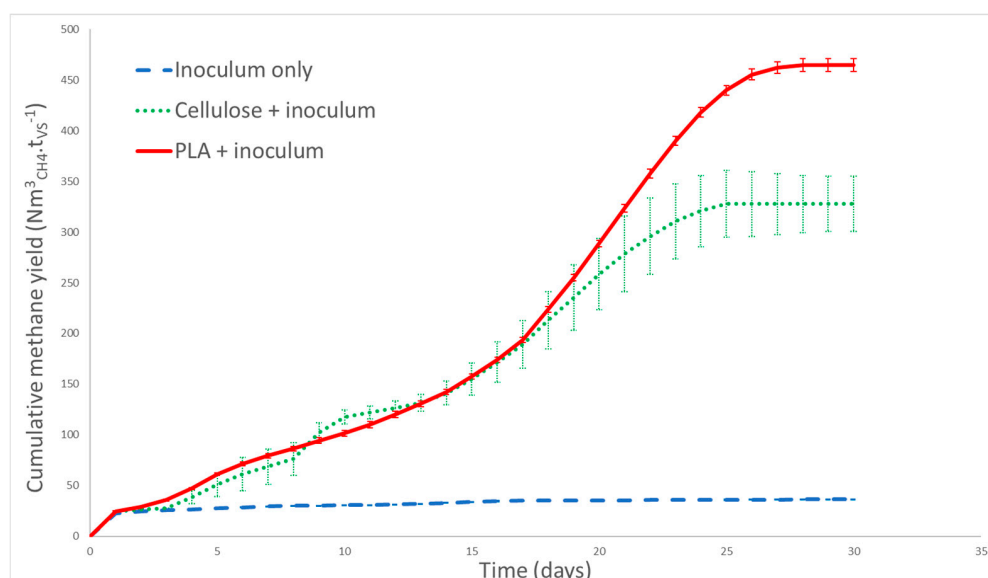


Figure 4. PLA degradation in thermophilic conditions to determine the degradation potential of PLA.

Looking at the standard deviation data from anaerobic degradation experiments reveals interesting patterns across the three conditions. The PLA with inoculum shows a steady increase in variability, starting near zero and progressively rising to 6.43, suggesting a gradual and consistent degradation process. In stark contrast, the inoculum alone

maintains remarkably low variability throughout the experiment, with standard deviations barely exceeding 0.45, indicating stable baseline conditions. The most dramatic behavior is observed in the inoculum with cellulose condition, which demonstrates rapid initial fluctuations followed by a substantial increase in variability, peaking at approximately 37.73 before slightly decreasing to 27.20. This dramatic difference compared to the other conditions suggests that cellulose triggers a more vigorous and heterogeneous degradation process, possibly due to the readily biodegradable nature of cellulose compared to PLA. The much higher standard deviations in the cellulose condition also indicate that the microbial population in the inoculum responds more actively and perhaps less uniformly to cellulose than to PLA, reflecting the different mechanisms and rates of biodegradation between these materials. The pH values remained within the optimal range for anaerobic digestion (6.8–7.4) in all the experimental conditions, indicating that the presence of 3D-printed materials did not cause a significant acidification or alkalization of the digestate. Overall, the results of the interaction study demonstrate that PLA is a suitable material for the fabrication of laboratory-scale AD digesters in mesophilic conditions, as it does not inhibit the AD process or negatively affect the stability of the digestate. On the other hand, SLA resins may introduce a bias in experimental results under mesophilic conditions due to the release of compounds that can stimulate methane production. This bias can be reduced by subjecting the SLA resin to a post-treatment process. The choice of 3D printing material and post-processing methods should therefore be carefully considered when designing laboratory-scale anaerobic digestion reactors, considering the specific operating conditions and the potential impact on the experimental results.

3.2. Batch Reactor Performance and Repeatability Analysis

The performance of the 3D-printed batch reactors (the FDM-printed PLA reactor and the SLA-printed High Temp V2 resin reactor) was compared to that of the conventionally machined PMMA reactor in a solid-state anaerobic digestion experiment using cattle manure as a substrate. The cumulative methane production curves obtained from the different reactor types over a 35-day digestion period are presented in Figure 5. To assess the repeatability of the experimental results obtained using the different reactor types, the solid-state anaerobic digestion experiment was conducted in triplicate for each reactor configuration. The cumulative methane production curves of the triplicate reactors of each type presented in Figure 4 were very similar, with overlapping curves and small standard deviations, confirming the high repeatability of the methane production kinetics. These results demonstrate that the 3D-printed reactors can provide consistent and reliable data on the anaerobic digestion process, not only in terms of the final methane yield but also in terms of the kinetics and the stability of the process. The low CV values (<10%) indicate good repeatability of the experimental results for all reactor types. However, the FDM-printed PLA reactor exhibited a slower initial methane production rate and a more irregular curve shape than the other two reactor types. Upon further investigation, it was observed that the PLA reactor had partially melted in several places, creating gas leaks. This cannot be attributed to the limited plasticity of PLA, which has a glass transition temperature of around 60 °C [15]. These gas leaks resulted in a lower and more irregular methane production curve for the FDM-printed PLA reactor. In contrast, the SLA-printed High Temp V2 resin reactor and the machined PMMA reactor maintained their structural integrity throughout the experiment, showing no signs of deformation or gas leaks. High Temp V2 resin has a higher glass transition temperature (238 °C) than PLA, making it more suitable for applications that require elevated temperatures. Similarly, PMMA has a glass transition temperature of around 105 °C [25], which is well above the operating temperature of the anaerobic digestion process. The analysis of standard deviations can

be refined with these important details. Among the three manufacturing experiments, the machined reactors demonstrate the most predictable behavior, with a progressive increase in standard deviation values from 0 to 6.32, suggesting a stable and controlled biological process. The SLA 3D-printed reactors, while showing higher initial standard deviations around 5.72, maintain relatively consistent standard deviations between 7 and 8 throughout the process before reaching 7.71, indicating a stable process despite greater baseline heterogeneity. However, the constant standard deviation observed in the FDM PLA 3D-printed reactors (starting with high variations of 8.31 and 10.15 in early stages before remaining at 7.84 for an extended period) is not indicative of process stability, as previously suggested, but rather represents a failure in the experimental process. This failure likely prevented any further biological activity or evolution of the system, resulting in artificially static measurements. The final methane yields achieved after 17 days of digestion were 125.14 ± 4.65 NL/kg Vs for the machined PMMA reactor, 83.68 ± 5.91 NL/kg VS for the FDM-printed PLA reactor, and 122.94 ± 5.22 NL/kg Vs for the SLA-printed High Temp V2 resin reactor. These values are within the typical range of methane yields reported for cattle manure in the literature [32,33], indicating that the SLA 3D-printed reactors performed as well as the conventionally machined reactor in terms of methane production. The similarity in methane production between the different reactor types suggests that the 3D printing materials and manufacturing methods used in this study did not have a significant impact on the anaerobic digestion process. The lower methane yield obtained with the FDM-printed PLA reactor can be attributed to the gas leaks caused by the partial melting of the material, which resulted in a loss of biogas and reduced overall methane production. These results suggest that FDM-printed PLA reactors are not suitable for anaerobic digestion experiments under mesophilic conditions due to the limited plasticity of PLA and its tendency to deform and create gas leaks at temperatures close to its glass transition temperature. This limitation is expected to be even more pronounced under thermophilic conditions (50–60 °C), where the softening and deformation of PLA would be more severe.

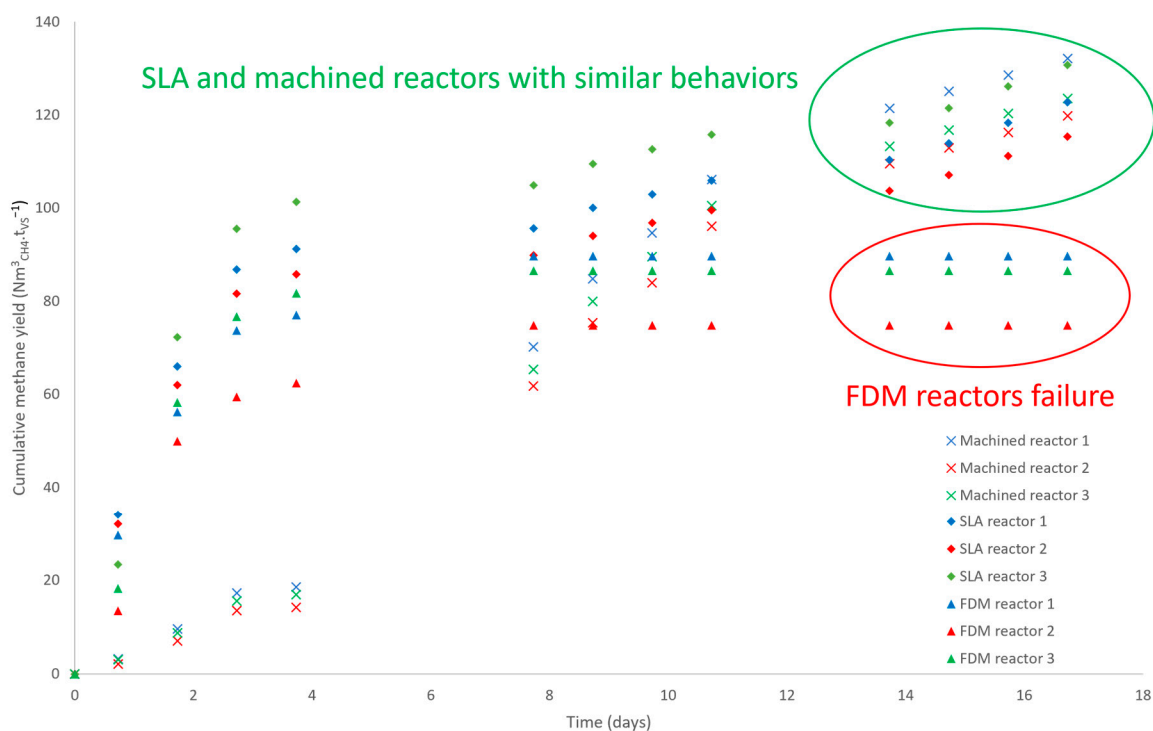


Figure 5. Repeatability analysis and batch reactor performance comparison.

The limited thermal stability and mechanical properties of PLA can be attributed to its molecular structure and the characteristics of the FDM printing process. PLA is a semi-crystalline thermoplastic polyester with a relatively low molecular weight and a high degree of crystallinity, which results in a brittle behavior and a limited ability to withstand sustained mechanical stress, especially at elevated temperatures [15]. The FDM printing process, which involves the extrusion of a molten PLA filament through a heated nozzle and the layer-by-layer deposition of the material onto a build platform, can further exacerbate these limitations. The rapid cooling and solidification of the extruded PLA filament can lead to the formation of internal stresses and weak interlayer bonding, resulting in a more brittle and less homogeneous structure compared to SLA-printed or conventionally machined parts [34]. Moreover, the porosity and surface roughness of FDM-printed parts, which are inherent to the layered structure and the resolution limitations of the process, can act as stress concentrators and initiation sites for cracks and fractures, further reducing the mechanical stability and gas tightness of the PLA reactors under the demanding conditions of the anaerobic digestion process.

In contrast, the SLA 3D-printed reactors demonstrated comparable repeatability to the conventionally machined reactor, further supporting their suitability for solid-state anaerobic digestion studies at a laboratory scale. The good repeatability of the SLA 3D-printed reactors can be attributed to several factors. The use of standardized 3D printing materials with consistent properties ensures that the reactors have similar chemical resistance, thermal stability, and permeability, minimizing the variability associated with the reactor material. The post-processing methods used in this study, such as the post-curing of the SLA-printed High Temp V2 resin reactor, contribute to the uniformity and stability of the reactor surface, reducing the potential for leaks and interactions with the substrate and inoculum. This finding agrees with the results of the interaction study, which showed that post-treated SLA resin did not inhibit or significantly stimulate methane production under mesophilic conditions. The faster methane production observed in the SLA-printed High Temp V2 resin reactor than in the machined PMMA reactor can be attributed to three key factors: enhanced heat exchange, surface roughness, and the presence of isopropanol residues. The SLA printing process allows for the creation of complex internal structures, such as fins or lattices, which can significantly increase the surface area for heat transfer [35]. This improved heat exchange ensures a more uniform temperature distribution within the reactor, promoting optimal conditions for the growth and activity of the anaerobic microbial community. Moreover, the layer-by-layer nature of the SLA printing process results in a slightly rougher surface than that of machined PMMA [18]. This increased surface roughness provides a larger surface area for microbial attachment and biofilm formation, which is crucial for the efficient conversion of organic substrates into methane. Additionally, the post-processing of SLA-printed parts involves cleaning with isopropanol to remove any uncured resin [19]. Residual isopropanol on the reactor surface may act as a substrate for certain anaerobic microorganisms, promoting their growth and activity. The combination of enhanced heat transfer, surface roughness, and the presence of isopropanol residues in the SLA-printed reactor likely contributed to the faster methane production rate observed in this study.

3.3. Economic and Practical Issues

The economic and practical aspects of using 3D-printed reactors for solid-state anaerobic digestion studies were evaluated and compared to those of conventionally machined reactors. The manufacturing costs, lead times, and key practical considerations for the different reactor types are presented in Table 2.

Table 2. Cost comparison between each design method.

Reactor Type	Manufacturing Cost (EUR)	Lead Time (Weeks)	Practical Considerations
Machined PMMA	1500–2000	6–8	Requires specialized machining equipment Limited design flexibility High cost for small quantities
FDM-printed PLA	50–60	1–2	Requires coating for sealing Limited temperature resistance Potential for biodegradation over long-term use
SLA-printed High Temp V2	400–450	1–2	Requires post-processing Potential experimental bias from solvents

The manufacturing costs for 3D-printed reactors were significantly lower than those for machined PMMA reactors, with FDM-printed PLA reactors being the most cost-effective option. The cost of producing a single FDM-printed PLA reactor was estimated to be EUR 60, including the filament and the post-processing materials (varnish). In comparison, the cost of producing a single machined PMMA reactor was estimated to be between EUR 1500 and 2000, including the cost of the raw materials, the machining time, and the labor. The cost difference between the two reactor types becomes even more significant when considering the production of multiple reactors, as the cost of 3D printing scales more favorably with the number of units produced than that of machining. The lead times for 3D-printed reactors were also considerably shorter than those for machined reactors. The production of an FDM-printed PLA reactor, from the design to the final post-processing, can be completed in 1 to 2 weeks, depending on the availability of the 3D printer and the post-processing materials. In contrast, the production of a machined PMMA reactor can take 6 to 8 weeks, depending on the availability of the machining equipment and the workload of the machining workshop. The shorter lead times for 3D-printed reactors allow for faster iteration and optimization of reactor designs, enabling more efficient and responsive research and development of anaerobic digestion technologies.

In terms of practical considerations, 3D-printed reactors offer several advantages over conventionally machined reactors. First, 3D printing allows for greater design flexibility, enabling the fabrication of reactors with complex geometries, such as internal baffles, mixing elements, and heat exchange surfaces, which are difficult or impossible to produce using machining methods. This design flexibility can be used to optimize the reactor performance, for example, by improving the mass and heat transfer or by reducing the dead zones and short-circuiting. Second, 3D printing enables the rapid prototyping and testing of different reactor designs, allowing for faster optimization and validation of the reactor geometry and operating conditions. This rapid prototyping capability is particularly useful in the early stages of process development, where multiple reactor configurations may need to be evaluated before selecting the optimal design for scale-up. However, 3D-printed reactors also have some limitations and challenges that need to be considered. FDM-printed PLA reactors have limited temperature resistance, with a maximum operating temperature of around 50–60 °C, which may not be suitable for certain anaerobic digestion applications that require higher temperatures, such as thermophilic digestion. Additionally, PLA is biodegradable, which may lead to the deterioration of the reactor over long-term use, especially if exposed to high temperatures and humid environments. This last consideration may impact the reactor performance in the case of prolonged use.

SLA-printed High Temp V2 resin reactors offer higher temperature resistance and better chemical stability than FDM-printed PLA reactors, making them suitable for a wider range of anaerobic digestion applications. However, SLA printing requires post-processing,

such as cleaning with isopropanol and UV curing, which can be time-consuming and may introduce experimental bias if not performed properly. Additionally, the cost of SLA printing is higher than that of FDM printing due to the higher cost of the resin and the more complex post-processing requirements.

The choice of reactor manufacturing method for solid-state anaerobic digestion studies depends on several factors, including the specific application requirements, the available budget and resources, and the desired reactor features and performance. Three-dimensional printing offers a cost-effective, flexible, and rapid alternative to conventional machining for the fabrication of laboratory-scale anaerobic digestion reactors, enabling faster optimization and development of anaerobic digestion technologies. However, the limitations and challenges of 3D printing, such as the limited temperature resistance of PLA and the potential for experimental bias from the post-processing of SLA resins, need to be carefully considered and addressed to ensure the reliability and reproducibility of the experimental results. The economic and practical aspects of 3D printing for the fabrication of anaerobic digestion reactors are expected to improve further with the ongoing development and commercialization of new 3D printing materials and technologies. For example, the availability of high-performance thermoplastics, such as PEEK (polyetheretherketone) and PEI (polyetherimide), with better temperature resistance and chemical stability than PLA could expand the range of anaerobic digestion applications that can be studied using FDM-printed reactors [36]. Additionally, PLA's performance could be enhanced through various modifications: incorporating heat-stabilizing additives [37], developing PLA blends with higher-temperature-resistant polymers like ABS or PC [38], or implementing fiber reinforcement using materials such as carbon or glass fibers to improve both thermal and mechanical properties [39]. From a scalability perspective, these materials present different challenges in industrial applications.

Similarly, the development of new SLA resins with improved mechanical and thermal properties, as well as reduced post-processing requirements, could make SLA printing a more attractive and cost-effective option for the fabrication of anaerobic digestion reactors. Considering SLA-printed reactors, the post-processing protocols for SLA 3D-printed reactors could be enhanced too through several key mitigation strategies. For SLA-printed reactors, implementing an optimized post-processing sequence would be crucial: extending the UV curing time beyond standard recommendations to ensure complete resin polymerization, followed by a standardized multi-step isopropanol washing protocol with controlled washing times and fresh solvent for each step. This washing protocol could be complemented by a thermal post-treatment step designed to remove any residual isopropanol, thus minimizing its potential impact on the anaerobic digestion process. Additional surface treatments could then be applied, such as a UV-resistant coating to prevent any long-term degradation or a specialized sealant to reduce surface interactions with the biological medium. For FDM-printed reactors, post-processing could include implementing a temperature-controlled environment during printing to ensure optimal layer adhesion, applying post-printing annealing treatments to increase crystallinity and temperature resistance, and potentially developing specialized coating protocols to enhance gas impermeability. These enhanced post-processing protocols, particularly when standardized across laboratories, could significantly improve the reliability and reproducibility of 3D-printed reactors for anaerobic digestion applications.

4. Conclusions

The feasibility and potential of using 3D printing for the fabrication of laboratory-scale reactors for solid-state anaerobic digestion research were investigated in this study. The results demonstrated that 3D-printed reactors made of High Temp V2 resin via SLA printing

can provide reliable and reproducible results in terms of methane yield, process stability, and biogas composition, comparable to those of conventionally machined PMMA reactors. This finding highlights the immense potential of SLA 3D printing as a viable alternative to traditional manufacturing methods to produce high-quality laboratory-scale reactors.

However, the study also revealed that not all 3D printing technologies are suitable for this application. Fused deposition modeling (FDM)-printed reactors made of polylactic acid (PLA) were found to be unsuitable for anaerobic digestion experiments due to the material's limited plasticity and tendency to deform and create gas leaks at elevated temperatures. This finding underscores the importance of careful material selection and thorough evaluation of 3D printing technologies for specific applications, as the choice of material and printing method can significantly impact the performance and reliability of the resulting reactors.

The study also delved into the complex interactions between the 3D printing materials and the anaerobic digestion process. It revealed that PLA has a negligible impact on methane production under mesophilic conditions, which is a promising result for the potential use of this biodegradable material in other aspects of anaerobic digestion research. However, the study also revealed that SLA resins may introduce bias due to the release of compounds that can stimulate methane production. While this bias can be reduced by post-processing the SLA resin using UV curing and cleaning with isopropanol, it highlights the need for careful characterization and control of the materials used in 3D-printed reactors to ensure the accuracy and reproducibility of experimental results.

The batch reactor performance study provided valuable insights into the strengths and limitations of different 3D printing technologies for anaerobic digestion research. The superior performance of the SLA-printed High Temp V2 resin reactor, which demonstrated a faster methane production rate than even the conventionally machined PMMA reactor, can be attributed to several factors, including enhanced heat transfer, surface roughness, and the presence of isopropanol residues that may stimulate microbial activity. These findings suggest that SLA 3D printing not only offers a cost-effective and flexible alternative to conventional machining but may also provide opportunities for the optimization and enhancement of anaerobic digestion processes through the manipulation of reactor design and material properties. This study also highlighted the limitations of FDM-printed PLA reactors for anaerobic digestion experiments under mesophilic conditions, as the material partially melted and created gas leaks, resulting in a slower and more irregular methane production curve than the SLA-printed and machined reactors.

This study demonstrates the potential of SLA 3D printing as a viable alternative to conventional machining for the fabrication of laboratory-scale anaerobic digestion reactors, particularly under mesophilic conditions. The results also highlight the limitations of FDM-printed PLA reactors in terms of their thermal stability and tendency to deform and create gas leaks at elevated temperatures. However, it is important to note that FDM printing with PLA or other materials may still have valuable applications in anaerobic digestion research, such as for the fabrication of reactor components, accessories, or for experiments conducted at lower temperatures. Future research should focus on several key areas to fully realize the potential of 3D printing in anaerobic digestion studies. These include further optimizing the SLA printing process and post-processing methods to minimize potential biases introduced by resin materials, exploring new 3D printing materials with improved thermal and chemical stability, investigating the long-term performance and durability of 3D-printed reactors under continuous operation, and establishing standard protocols and guidelines for the design, fabrication, and operation of 3D-printed anaerobic digestion reactors. Additionally, the potential applications of FDM printing with PLA and

other materials in anaerobic digestion research should be further explored, considering their specific limitations and strengths.

Recent systematic evaluations of emerging technologies within the circular economy framework have highlighted the crucial role of 3D printing and bioplastics in enabling waste minimization and resource recovery. Building on these insights, future research should focus on several key areas: **Advanced Bioplastics Development:** Investigation of polyhydroxyalkanoates (PHAs) and their compatibility with 3D printing processes, including comprehensive life-cycle analyses to assess their sustainability impact. **Material Performance Studies:** Long-term assessment of material durability under thermophilic conditions, with particular emphasis on thermal stability and mechanical properties to support scalability. **Microbial–Material Interactions:** Implementation of molecular techniques, including DNA sequencing, to understand and optimize microbial–material interactions and ensure biocompatibility. **Economic and Standardization Framework:** Development of comprehensive life-cycle cost analyses and standardized protocols for fabrication and post-processing to facilitate broader adoption [40].

By fostering collaboration, knowledge sharing, and the adoption of best practices, the anaerobic digestion research community can harness the transformative potential of 3D printing technology and accelerate the development of sustainable waste management and renewable energy solutions. The adoption and standardization of 3D printing methods, including both SLA and FDM techniques, could lead to more efficient, cost-effective, and sustainable development of anaerobic digestion technologies, enabling their wider application across diverse settings and scales.

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