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A CASE STUDY OF IMPLEMENTATION OF CIRCULAR ECONOMY PRINCIPLES TO WASTE MANAGEMENT: INTEGRATED TREATMENT OF CHEESE WHEY AND HI-TECH WASTE

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ABSTRACT: In a global context characterized by severe environmental problems and increasing resource scarcity, waste represents both a challenge and an opportunity. This study aims to demonstrate with a real case the potential for optimizing the waste valorization action attainable through the synergic application of different treatments to residues of equally different nature and origin. In particular, bio-chemical (dark fermentation), chemical-physical (selective leaching) and thermo-chemical (hydrothermal carbonization) treatments were applied for the integrated valorization of whey from sheep cheese production and Hi-Tech waste (discarded electrical and electronic equipment). The treatments were applied at a laboratory scale on real samples of these residues. The organic acids used for selective leaching of valuable metals from Hi-Tech waste were obtained by dark fermentation of the cheese whey, while hydrothermal carbonization was used to convert the waste from previous stages into hydrochar feasible as solid fuel or soil improver. The dark fermentation tests have highlighted the possibility of recovering ≈ 100 g of organic acids from 1 L of whey; furthermore, it is also possible to recover bio-hydrogen depending on the operating conditions applied and the type of targeted organic acids. The leaching tests have demonstrated how the organic acids from whey fermentation have selective and quantitative mobilization capacities comparable to those of the same acids available on the market. The carbonization tests produced carbon-enriched hydrochar with promising fuel properties, as well as process waters suitable for anaerobic digestion with methane production. The results of the project led to the filing of an international patent.

Keywords: processes integration, fermentation, green leaching, hydrothermal carbonization, waste, biorefinery

1. INTRODUCTION

The term sustainability demands meeting and conciliating various needs (productive, economic, political, social, environmental) that are strongly interconnected. In fact, humans have generated imbalances of such magnitude within each of the aforementioned sectors that the effects go beyond their own boundaries. Consequently, we live in an era characterized by complex problems to which it is not possible to give simple answers ("For every complex problem there is an answer that is clear, simple, and wrong." — H. L. Mencken). The possibility of overcoming current difficulties and guaranteeing a future for life on Earth lies in thinking in a complex way.

Waste management is no exception to this. In a global context characterized by severe environmental issues and resources which are progressively scarcer in relation to the increasing demand, waste represents an environmental burden to be properly managed, but also a potential resource.

It has come a long way for waste management. Initially, and for a long time, the approach was extremely basic: discard, collect, sink it into a faraway hole or burn it. We then moved on to the integrated management system in which valorisation assumed an important role, even if meant, almost, more as landfill avoidance than a real recovery of marketable resources. In particular, the valorization of organic waste was, and still currently is, pursued mostly in the form of a few products, e.g., biogas and compost, characterized by a relatively low value (Alibardi et al., 2020; Clarke, 2018).

Today, with the application of the principles of the circular economy, waste management must acquire characteristics of complexity to fully enter the economic-productive system where it contributes to the reduction of the consumption of raw materials/resources by delaying the becoming waste of objects (reuse/maintenance) or recovering resources (recycling) (Alibardi et al., 2020; Sarc et al., 2019; Vrancken et al., 2017; Walmsley et al., 2019). Although the implementation of the circular economy favors reuse, recycling still plays a main role for degradable goods and materials, for which the possibilities of reuse are understandably very limited. In this respect, organic residues may represent a widely available and renewable source for the recovery of products and energy to be used in other activities (Alibardi et al., 2020; Ma et al., 2018; Papież et al., 2018).

To be consistent with the principles of the circular economy, recycling must aim to minimize waste (approximation of the zero-waste objective) and to recover materials and compounds of interest for the production system.

Pursuing these objectives requires overcoming the logic based on the application of a single process for the recovery of a single product, in favor of the ability to combine processes synergistically in such a way that the outflow of one is the input of another and the different recovered products meet different market needs, in other words: the ability to think in a complex way applied to waste management (Asunis et al., 2020; Flórez-Fernández et al., 2021; Longati et al., 2020).

In the case of biodegradable waste management, the synergistic combination of processes is often referred to as waste biorefinery, a concept that is considered pivotal for the implementation of the evolution of the circular economy known as circular bioeconomy. Indeed, the waste biorefinery is an evolution of the biorefinery concept to include waste as an alternative to dedicated biomass and to enhance the recovery of organic waste (Alibardi et al., 2020). Since, as it was already said, in the circular economy the economic aspect (marketing) must be as important as the environmental one (waste treatment), the ideal waste biorefinery must be able to recover from waste biomass products characterized by value on the market and, not insignificantly, usable also in production processes that have nothing in common with those that originated the waste biomass, in order to provide flexibility and resilience on the market.

In this framework, the project "Integrated treatment of cheese whey and Hi-Tech waste: a demonstration of synergistic waste management in a circular economy perspective" represents a case study of the application of the principles of the circular economy/bioeconomy to two completely different production sectors such as the dairy sector and the electrical and electronic goods one.

The paper presents the preliminary results of the project that, although they require further investigation, highlight the potential of the concept of integrated valorisation.

The project was conceived taking into consideration some principles consistent with what was stated above:

- the valorization of residues characterized by different origin and nature;
- the integration of different processes (bio-chemical, physico-chemical, thermo-chemical);
- the recovery of products characterized by high added value and flexibility of use on the market;
- the integration between the solution of potential environmental problems and the opportunity to strengthen and expand the economic assets of specific production sectors;
- waste valorisation as element of connection between different productive sectors.

In particular, bio-chemical (dark fermentation-DF), chemical-physical (selective leaching) and thermo-chemical (hydrothermal carbonization-HTC) treatments were applied for the integrated valorization of whey from sheep cheese (CW) production and Hi-Tech waste (generic term used to describe all types of old, end-of-life or discarded electrical and electronic equipment). In particular, the treatments were applied at a laboratory scale on real samples of the residues under concern. The organic acids used for selective leaching of valuable metals from printed circuits boards-PCB were obtained by DF of the CW, while HTC was used to convert the waste from previous stages into hydrochar feasible as solid fuel or soil improver.

The processes selected to be jointly applied were chosen according to the characteristics of the residue under concern:

- compatibility with high organic load substrates (DF, HTC);
- capability to manage streams with high water content (DF, leaching, HTC);
- capability to simplify complex organic molecules by preparing them for subsequent phases of recovery (DF);
- capability to recover compounds characterized by high added value (DF, leaching of valuable metals from Hi-Tech waste).

Cheese whey is the most important residue of the dairy industry (0.8-0.9 L produced per L of milk processed) and is considered a potential environmental risk due to its quantity, high organic load, presence of salts and tendency to acidify. Improper management leads to anoxia, toxicity, contamination of groundwater and soil. Discharge of CW into sewer systems can affect the efficiency of water treatment plants and the high content of lactose and minerals causes problems if used as animal feed (Akhlaghi et al., 2017). So far, chemical-physical and biological treatments have been applied mainly aimed at removing the organic load. Some characteristics of CW have also proven to be problematic for the application of valorization options such as methanogenic anaerobic digestion (Asunis et al., 2020).

Dark fermentation represents a mainstay towards full-scale implementation of the waste biorefinery concept. In waste management, DF has always been seen as an intermediate stage of anaerobic digestion to be overcome as quickly as possible in order not to affect the recovery of biomethane. In the new context of the circular bioeconomy, the ability of DF to mildly decompose the organic matter is instead a strong point. In fact, DF is a versatile process that can be addressed towards the desired products through the proper setting of the main operating parameters (operating pH and temperature, organic loading rate, hydraulic retention time, etc.) to recover gaseous and soluble substances/compounds that can be used for energy purposes or as building blocks or substrates for further processes. Moreover, DF not only allows the recovery of marketable products, but it is also ideal as preparation of the original organic substrate for other processes aimed at recovering further energy and materials. Finally, DF can be preceded by treatments aimed at the extraction of intact compounds with high added value (*e.g.*, for the medical, pharmaceutical, nutraceutical sector). In this respect, DF may represent a promising option for CW valorization (Asunis et al., 2020; Carvalho et al., 2013). The issue has been already addressed by several studies with emphasis on H_2 production, but much less attention has been paid to the possibility of recovering also other valuable products. The authors have been performing studies on the application of DF to CW for years, though with the aim of recovering bio-hydrogen, a type of recovery that is also compatible with the recovery of most of the organic acids targeted in the present study, with the exception of lactic acid (Akhlaghi et al., 2017; Asunis et al., 2020).

Printed circuit boards are of great interest due to their high content of valuable and critical elements, on the basis of which they can be considered secondary sources of raw materials. However, current industrial recovery exploits energy-intensive thermal treatments and/or chemical processes based on the use of aggressive leaching agents such as HNO_3 solutions through a non-selective approach (Burkovic, 2007; Carvalho et al., 2013; Iannicelli-Zubiani et al., 2017). Several alternatives have been proposed, highlighting the potential of weak organic acids as a more sustainable and selective solution (Jadhav et al., 2016).

Hydrothermal carbonization is a wet thermochemical process where heat and pressure are applied to treat organic matter in the presence of water and under subcritical conditions (Libra et al., 2011). Heat and pressure are applied to process organic matter in presence of water and under subcritical conditions (Kambo & Dutta, 2015). The resulting material is a carbonaceous solid, characterized by high chemical stability and energetic value and a moderately high surface area, suitable as a fuel, adsorbent or soil improver, as well as to ensure stable carbon storage. HTC is currently considered a promising approach, in particular for the valorization of organic wastes characterized by a high water content such as agro-industrial residues and fermented/digested substrates (Farru et al., 2022a; Kim et al., 2014; Li et al., 2020). The obtained material is a coal-like hydrochar characterized by high chemical stability and energetic value, and a moderately high surface area, which is suited as fuel or adsorbent or soil amender, as well as to ensure stable carbon storage. HTC is currently considered a promising approach, in particular for the valorization of organic waste characterized by high-water content, such as agro-industrial residues and fermented/digested substrates.

The novelty of the study is not only in the application of the individual treatments on the individual waste under concern, but above all in the integrated application of processes for the joint valorization of CW and PCBs. To the best of our knowledge, this approach has never been studied, and the results obtained have led to the filing of an international patent. Given the limited number of studies reported in the literature, or full-scale applications, which refer to synergistic and combined management of residues of different nature and origin, this project is characterized by a level of innovation and interest that involves not only the single processes and the residue under concern, but more in general, the approach to the management of any waste from production activities. In this respect, the proposed study is believed to contribute to further research aimed at exploring innovative waste management and valorization strategies.

2. OBJECTIVE

The general target of the project is the study of an innovative and integrated system for the valorisation of sheep cheese whey and Hi-Tech waste. The system combines, through an appropriate sequence of processes, the control of the negative impact deriving from inappropriate management with the recovery of resources, according to the principles of sustainability and circular economy.

The general target was divided into specific goals:

- Production of organic acids through CW dark fermentation performed under operating conditions suitable to guarantee process stability and maximize production yields.
- Recovery of metals from Hi-Tech waste through selective leaching performed by using the CW dark fermentation products as waste-derived and gentle green chemicals, as alternative to conventional approaches where aggressive unselective leaching agents are used and subsequent separation and purification phases are required.
- Complete the valorisation process through HTC of the residues deriving from the clarification of the fermented CW and leaching of metals from Hi-Tech waste.

The general description of the experimental activities as well as the preliminary results obtained, are reported below.

3. MATERIALS AND METHODS

3.1 Cheese whey and printed circuit boards

Samples of ovine CW were collected at a diary plant in Sardinia (Italy). Since it is a biodegradable material, the samples were stored in a refrigerator, taking the quantities necessary to carry out the activities from time to time. The CW was then subjected to chemical-physical characterization analyses functional to the application of the valorization treatment by means of DF.

RAM type PCBs were sampled at a recycling plant for waste from electrical and electronic equipment, pre-treated through mechanical comminution to increase the specific surface area and characterised by ICP-AES/MS, XRD, SEM-EDS.

3.2 Treatment tests

3.2.1 CW dark fermentation tests

Mesophilic batch DF tests were carried out, without adding any external inoculum, using automatically controlled bioreactors, adopting three different pH setpoints (6, 6.5 and 7). Liquid and gaseous products were characterized; in particular, lactose and HLa concentration were monitored over time in DF step 2 and VFAs concentration in DF step 3. Finally, a predictive model of the evolution of the process based on the pH value, initial lactose concentration, HLa/VFAs ratio was derived.

The CW organic load was exploited through a 3 steps DF process, without pre-treatments or external inoculum in order to reduce the complexity and cost of the process: 1) lactose hydrolysis to glucose and galactose, 2) monomeric sugars conversion to lactic acid (HLa), 3) HLa conversion to soluble (volatile fatty acids-VFAs) and gaseous (H_2 and CO_2) products (Asunis et al., 2020). In view of the subsequent steps of the integrated process, both the HLa produced during DF step 2 and the mixture of VFAs produced during DF step 3 are of interest. In this respect, the DF process was properly managed and monitored. More specifically, when HLa was the product of interest, the optimal operating pH and duration of DF step 2 were optimized in order to maximize the production of HLa and avoid its conversion into VFAs. Conversely, when the product of interest was a mixture of VFAs, the operating pH and the overall duration of DF (steps 1, 2 and 3) were optimized to obtain the maximum conversion of HLa to VFAs. Furthermore, DF step 3 can occur according to different metabolic pathways with consequent effects in terms of presence of acetic, propionic and butyric acid in the fermentation broth with different reciprocal mass ratios. Therefore, the effect of the pH on the composition of the DF broth was evaluated.

3.2.2 Tests aimed at recovering high-value metals from Hi-Tech waste

Appropriate conditions were, hence, applied to dissolve and recover selectively specific classes of metals from Hi-Tech wastes. Specifically, the fermented CW, rich in HLa or mixtures of VFAs, was used after a simple clarification step (addition of ethanol 5:3 in vol. (Morr & Lin, 1970)), as an agent for leaching base metals (metals characterized by low reduction potential, E° < 0, like lead, tin, iron, cobalt, etc.) under gentle experimental conditions (low concentration, temperatures spanning from room to 100 °C, open environment at atmospheric pressure) from comminute waste Printed Circuit Boards (PCBs), as a potentially greener and safer alternative to the conventional use of strong oxidising mineral acids. The use of fermented CW was integrated in a previously established innovative process where base metals were leached by using commercial citric acid solutions as a preliminary step to the low environmental impact and selective recovery of noble metals (metals characterized by highly positive reduction potential, E° > 0, like copper, silver and gold) (Rigoldi et al., 2019). Powdered samples of RAM PCBs were reacted with fermented aqueous CW mixtures on varying the temperature (25-100 °C), the primary leaching agent (HLa or VFAs) and the lixiviant concentration (0.5-3M) in order to set up the best conditions for a selective and efficient base metals leaching. Experiments were also performed with pure commercial lactic, acetic, propionic and butyric acids, as a reference. The leaching results were investigated through an accurate chemical-physical characterization of the materials, mainly by ICP-AES/MS, XRD, SEM-EDS, before and after chemical treatment. Activities are, hence, progressing in two different directions: i) base metals are selectively recovered from the leachate by conventional fractional precipitation procedures, specifically separating toxic lead in form of low solubility halogenide, and providing HLa-rich waste solutions useful as feedstocks for HTC valorization; ii) the residual solid fraction is treated for noble metals recovery as for the recovery scheme already established.

3.2.3 HTC tests

The residues deriving from the clarification of the fermented CW and the metal leaching process were valorized through hydrothermal carbonization. For the sake of comparison, HTC was applied also on the raw CW. The influence of the main operating parameters (temperature and residence time) was tested and optimized in order to improve the product properties. Three different temperature setpoints and three different residence times were investigated. The process temperature was set between 180 and 220 °C, over a duration of 0.5, 1 and 2 hours, allowing hydrolysis, condensation, decarboxylation and dehydration reactions whose effect is to reduce the hydrogen and oxygen content of the residue, while carbon and energy content are densified in the solid material (Farru et al., 2022b). Batch HTC runs were performed on the CW and the two process residues in a 1.5 L pressurized and stirred reactor equipped with an electric jacket for temperature control. The produced hydrochar was characterized in terms of proximate and elemental analyses and calorific value measurement. Properties, such as pH, electric conductivity, nutrient availability, thermal stability, type of surface functional groups and germination ability, useful to evaluate several hydrochar applications, were assessed. The HTC process water was also characterized in order to explore possible applications on soil and for energy production (through germination test and BMP-Biomethane potential test). A schematic representation of the studied process is provided in Fig. 1.

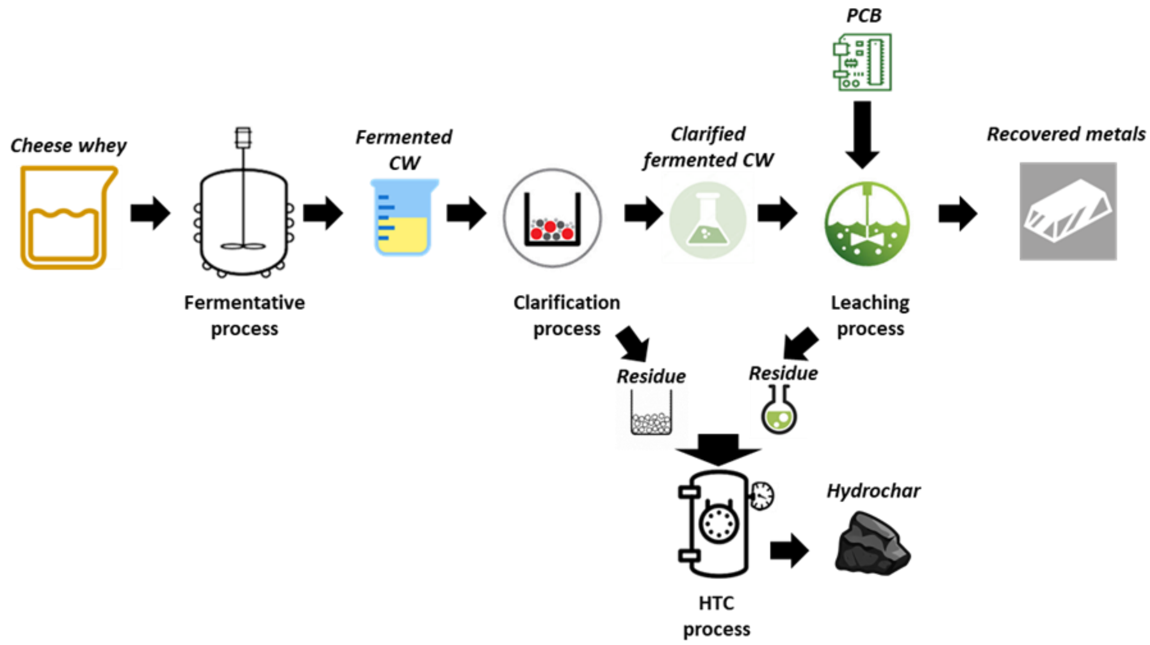

Figure 1. Schematic representation of the proposed integrated valorization of sheep cheese whey and Hi-Tech waste

4. RESULTS

The obtained results can be summarized as follows.

- Assessment of the proper operating conditions to obtain a stable and high conversion of the CW organic load into products to be used for subsequent process phases.
- Assessment of the maximum yields obtainable for the products of interest: lactic, acetic, propionic, and butyric acid.
- Obtaining DF broths suitable as a leaching agent for the recovery of metals from Hi-Tech waste.
- Identification of the more selective leaching solution able to enhance recovery of single metals or classes of metals contained in powdered PCBs.
- Addressing, in an interactive way, DF of CW towards the production of the best leaching solution.
- Finding the best conditions for recovery of metals from real Hi-Tech waste, when necessary also combining the use of fermented CW with other safe and recycled reagents for high reduction potential metals (e.g., I3- for gold) in order to enhance the metal values from PCBs as much as possible.
- Assessing the operating conditions suitable to optimize the final quality of the HTC-produced hydrochar and process water.
- Obtaining HTC hydrochar suitable as solid fuel or soil improver, as well as process water suitable for methane production.

In details, the DF tests highlighted that the process develops according to two stages characterized by different products, both of interest and that can be recovered separately thanks to the adoption of suitable operating parameters: lactose is converted to lactic acid (1st step), and thereafter lactic acid is converted to VFAs (2nd step). In order to optimize the conversion yields and kinetics of both stages and allow for their separate management, the operating parameters that play the fundamental role are the operating pH, which must be controlled continuously, and the hydraulic retention time. In light of the results obtained, it is possible to hypothesize the following production yields: ≈ 60-70 g HLa L^{-1}, ≈ 10-15 g HBu L^{-1}, ≈ 7-10 g HPr L^{-1}. Moreover, the production of VFAs in the second stage may lead also to the recovery of 5-7 L H_2 L^{-1}.

Preliminary results on comminuted RAM PCBs have shown how HLa performs better as leaching agent than other VFAs contained in fermented CW, probably due to its lower pK_a in water. For this reason, further systematic experiments were focused on HLa-based solutions application. Indeed, 1:1 HLa/La^- buffer solutions (pKa = pH = 3.8) demonstrated the highest efficacy and selectivity towards base metals even at very low concentration level (0.5M) and room temperature, obtaining an almost quantitative leaching yield for lead and tin within 24 h against desired zero or negligible rates of copper, silver and gold dissolution. Experiments confirmed the support of dissolved oxygen in the efficacy of the leaching, as already observed in previous studies on cobalt recovery (Oumarou Amadou et al., 2023). Furthermore, fermented CW mixtures demonstrated to achieve higher leaching rates towards a selection of metals, i.e. iron (73%) and nickel (72%), which are leached at a lower extent (10 and 13%, respectively) by the HLa commercial agent under the same experimental conditions (Serpe et al., 2023). In that sense, the oxidizing and complexing properties of the different soluble products of CW controlled DF showed to play a relevant role, still under investigation, in the efficiency and selectivity of metal leaching. Leaching under refluxing temperature (100 °C) shows that a significant improvement occurs just on the effectiveness of Fe and Ni dissolution. The presence of significant concentrations of salts, e.g., chlorides, in the mixtures also affects the occurrence of soluble/insoluble species in the post-leaching aqueous and solid fractions and should be carefully considered in designing the steps of the recovery process. Selective precipitation recovery of metals by adding sequentially halogenides, hydroxides and sulfides, allowed to isolate valued and, also, toxic metals and to provide a solution suitable for the following HTC treatment (suitable organic loading; metals concentration: Ni = 10.0 ppm; Cu =11.4 ppm; Sn = 2.94 ppm; Fe = 2.18 ppm; Al = 0.21 ppm; Cd = 0.12 ppm; Pb, Cr, Co << 0.1 ppm).

So far, a set of HTC tests were performed at 220 °C for 1 hour on CW, fermented CW, and a mixture of fermented CW and the residues after leaching. Under these conditions, the produced hydrochar exhibited a carbon densification (up to +53%) and a decrease in ash content (up to -63%). The resulting process water have shown significant potential for biogas production during BMP test. Based on these preliminary results, the HTC products, including hydrochars and process water, show a potential for energy recovery. The hydrochars, with their increased higher heating value (HHV, up to +82%), can be used for heat production or converted into electrical energy. The process water can be valorized within anaerobic digestion systems aimed at biogas production. Germination tests on cress seeds conducted using hydrochar, process water as well as CW indicated a high level of inhibition across all tested materials. The primary source of inhibition observed in the solid materials (CW and hydrochar) can be attributed to their elevated fat content, while the phytotoxicity observed in the process water can be attributed to the presence of dissolved organic compounds (total organic carbon up to 12 g/L).

5. DISCUSSION

The integrated treatment of cheese whey and Hi-Tech waste through DF, green leaching, and HTC demonstrates significant potential for the valorization of the residues under concern. However, the economic feasibility of the processes is crucial for their adoption in a circular economy. While this study is a preliminary report and does not include a detailed economic analysis, several factors indicate the potential for profitability. The reduction in disposal costs by converting waste into valuable products is a significant advantage. Additionally, the production of organic acids through DF, the recovery of metals from PCBs, and the production of hydrochar offer multiple revenue options. The application of green leaching agents and HTC reduces energy consumption compared to more conventional methods, decreasing the operating costs. Furthermore, the recovered metals and hydrochar have medium-high market value, thus support the economic viability. The same organic acids obtained by DF of CW could be placed on the market with good value regardless of the use as green leaching reagents. Furthermore, it is worth mentioning that the organic acids could also be used for other processes capable of recovering high added value products such as the production of biopolymers, and that also bio-H_2 is attainable during CW fermentation depending on the process operating conditions and the type of organic acid that is targeted (Asunis et al., 2020, 2022).

At a time when waste valorization seems to want to go beyond simple process schemes, characterized by the recovery of a single product of medium-low market value, to embrace multi-product approaches and oriented towards the highest added value, it is legitimate to ask whether this vision is actually applicable. In light of this, the development of studies such as the one illustrated cannot be limited to additional, mere experimental tests to further define and optimize the operating conditions, the achievable yields, etc. The in-depth study must also include technical-economic feasibility studies, performed in cooperation with the possible stakeholders and market players, and taking into consideration the differences that exist between the treatment of materials classified as waste, and the traditional treatment of raw materials or virgin biomass. The availability of materials and the characteristics of the supply basins, logistics (storage, transport), legislation, the very certainty of current legislation, are very different, and are reflected in fundamental aspects such as the minimum economically sustainable size of the plants.

Performing comprehensive life cycle assessments (LCA) to assess the environmental foot print of the integrated processes as compared to conventional waste management methods is crucial to better understand the attainable benefits and potential trade-offs.

To fully evaluate the scalability of the processes, the feasibility and LCA studies require the availability of reliable data. In this respect, it is important to start pilot scale experiments, possibly extended to other types of waste characterized by wide availability such as municipal solid waste organic fractions or wastewater treatment sludge.

6. CONCLUSIONS

The study and the project have produced experimental evidences that support the feasibility of an integrated approach to the valorization of different wastes through the application of equally different processes. The proposed approach is consistent with the principles of sustainability, circular economy, circular bioeconomy.

In particular:

- DF converts the organic load of CW into mixtures of organic acids useful for several applications; pH confirms to be the key operating parameter for properly addressing the DF towards the desired products. The result is of particular importance since it is obtained by the application of DF to a real substrate; the control of the DF process performed on real residual substrates and using indigenous microorganisms is not straightforward, as witnessed by the relatively low number of pilot plants and, to the best of our knowledge, the absence of full-scale application cases;
- the mixtures of organic acids obtained from CW fermentation are suitable for the selective and gentle mobilization of the metals present in PCBs, thus making possible a process that is less aggressive than those usually applied;
- the final residues are converted via HTC into a product that can be used as a solid fuel or soil improver, which makes it possible to close the valorization cycle of the residues under concern; additionally, a process water is also produced that is suitable for methane production through anaerobic digestion.

More generally, the study demonstrates how integrated waste management can systematize different production sectors, limiting their environmental problems and converting them into opportunities of potential economic interest. This is of particular importance for production sectors such as the dairy one, which are affected by periodic market fluctuations of their traditional products. Paradoxically, an environmental problem can increase the flexibility on the market and the economic resilience of a production activity.

Future research should focus on scaling up the studied processes, performing detailed economic analyses, and exploring the feasibility also for other types of waste.

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