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Case Study of Separated Liquid Digestate Treatment for Water Recovery and Production of a Nutrient-Rich Solid Fraction

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Abstract

Separated liquid digestate management is a major challenge for anaerobic digestion facilities because of the digestate's high water content, nutrient load and potential environmental impacts when land application is not adequately controlled. This preliminary single-batch case study evaluated the application of Adiabatic Sonic Evaporation and Crystallisation (ASEC) treatment to an animal-derived separated liquid digestate, focusing on water recovery and the production of a dried nutrient-rich solid fraction. The raw liquid digestate, the ASEC-recovered water and the recovered solid fraction were characterised using physicochemical and microbiological indicators. The recovered water stream showed a strong reduction in electrical conductivity from 11,289 to 42 µS/cm, with turbidity <1 UNF, TSS < 5 mg/L and ammoniacal N < 0.15 mg/L; microbiological indicators were also favourable (no detection of *Salmonella* spp. or *Legionella* spp., absence of intestinal nematode eggs and *Escherichia coli*). The recovered solid fraction contained 95% dry matter (dm), 53.5% organic matter, 3.87% total N, 36,090 mg P_2O_5/kg dm and 32,800 mg K_2O/kg, supporting its preliminary classification as candidate organic-mineral NPK fertilising material. Metal(loid)s were below the contaminant thresholds used as references in the EU for solid organic fertilisers. For a 100 m^3/d treatment scenario, the estimated NPK nutrient replacement value was 63,543 USD/year; however, this value should be interpreted as a scenario-based estimate rather than a measured process-scale economic return.

Keywords: crystallised solids; Adiabatic Sonic Evaporation and Crystallisation; animal-derived digestate; nutrient recovery; circular economy; zero liquid discharge

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1. Introduction

Anaerobic digestion (AD) is a key technology in the transition towards renewable energy, circular bioeconomy and sustainable waste management. By converting biodegradable organic matter under oxygen-free conditions, AD enables the production of biogas or biomethane from livestock manure, agricultural residues, agro-industrial by-products, sewage sludge, food waste and separately collected bio-waste. In Europe, this process is particularly relevant because renewable gas production is being promoted as part of the decarbonisation of the energy system and the reduction of dependence on fossil natural gas (waste-to-energy, WTE) [1]. The European renewable energy framework recognises biogas and biomethane within this transition when they comply with sustainability and greenhouse-gas-saving criteria [2,3].

Besides biogas generation, AD generates residual material remaining after the anaerobic conversion of organic substrates: a pumpable separated liquid digestate (SLD) with a high nutrient-rich fraction and elevated water content, and a wet solid fraction enriched in organic matter and particulate nutrients [1]. These products have heterogeneous composition depending on the feedstock (food waste, sewage sludge, crop residues, dairy residues or mixed agro-industrial streams), digestion temperature, hydraulic retention time, organic loading rate, post-treatment operations and storage conditions [4,5]. Digestates commonly contain water, organic matter, ammoniacal nitrogen, organic nitrogen, phosphorus, K, Ca, Mg, trace metals and variable microbiological loads [6,7]. In addition to nutrients and trace elements, digestates may contain residual organic contaminants, antimicrobial residues, hormones or other emerging contaminants depending on the origin of the feedstock and previous treatment history [8,9]. This reinforces the need for case-specific chemical screening before recovered fractions are reused in agricultural or environmental applications. This composition makes digestate a resource with agronomic value, but also a material requiring controlled management. In the present case study, the sample corresponded to an animal-derived SLD associated with a biogas production context.

From an agronomic perspective, SLD can contribute to nutrient recycling because a substantial part of the N, P, K and micronutrients contained in the original substrates remains in the final residue after AD [5,6]. Compared with raw manure, AD can increase the proportion of mineral nitrogen, particularly ammonium, which may improve short-term nitrogen availability for crops [6]. In addition, the residual organic matter can contribute to soil organic carbon inputs and improve nutrient cycling when application rates are properly adjusted to crop requirements and soil properties [5]. For this reason, SLD is frequently discussed as a potential organic fertiliser, biofertiliser or soil amendment within circular agriculture strategies [4,10]. However, direct land application of untreated SLD is associated with environmental risks, such as ammonia volatilisation, nitrate leaching, nitrous oxide emissions, phosphorus runoff, eutrophication and soil salinisation [5,11]. The risk is particularly high in livestock-dense regions where nutrient production exceeds the assimilative capacity of nearby agricultural soils. Elements such as Cu and Zn, associated with animal feeding supplements, may accumulate in soils [5,12]. On the other hand, SLD often has a high water content, which increases storage, handling and transport costs and limits its economic use when production sites are located far from available agricultural land.

This balance between nutrient recovery and pollution prevention is reflected in European legislation. The Nitrates Directive (Council Directive 91/676/EEC) limits the application of livestock manure in nitrate-vulnerable zones to an amount containing 170 kg N per ha and year, unless derogations are specifically authorised under safeguards [13]. The Waste Framework Directive (Directive 2008/98/EC) establishes the waste hierarchy prioritising prevention, reuse, recycling and recovery over disposal [14]. Its revised framework strengthens the role of separate bio-waste collection and circular management of biodegradable residues [2,14], increasing the availability of organic feedstocks for AD and the need for effective digestate management. The EU Fertilising Products Regulation (EU) 2019/1009 provides a framework for fertilising products derived from digestates from authorised input materials and compliant with processing, stability, contaminant hygiene and safety requirements [15]. Therefore, digestate treatment technologies should facilitate resource recovery, reduce waste volume and support the production of safe, traceable and standardised fertilising products.

Several technologies have been proposed to process digestate and recover valuable fractions, including mechanical solid–liquid separation, centrifugation, screw pressing, drying, composting, ammonia stripping, pressure-driven membrane filtration, reverse osmosis, evaporation, nutrient precipitation and struvite recovery [16–20]. Recent independent reviews show that these technologies can contribute to digestate volume reduction, water recovery and nutrient recovery, but their performance remains strongly dependent on the digestate composition, operating scale and target product quality. Mechanical separation mainly redistributes solids and particulate nutrients between fractions; membrane-based systems may produce high-quality permeates but are limited by fouling and concentrate management, while ammonia stripping and struvite precipitation are more selective for N and P recovery but usually require chemical dosing, pH control or additional post-treatment [16–20]. Recent techno-economic assessments also indicate that evaporation, electrodialysis and intensive treatment of the digestate liquid fraction may be constrained by energy demand, operating costs and scale-up limitations [18,19]. Therefore, technologies capable of simultaneously reducing liquid volume, recovering water and concentrating nutrients into a stable solid fraction remain of interest for biogas plants, although direct quantitative comparison requires replicated process data and full mass and energy balances.

In this context, Adiabatic Sonic Evaporation and Crystallisation (ASEC) technology offers a potential route for integrated digestate treatment and valorisation. ASEC was designed to separate contaminated aqueous matrices into a recovered water stream and a dry solid or crystallised fraction. Previous studies have evaluated ASEC in other complex environmental matrices, including brines, textile wastewater, mining waters and contaminated leachates, showing its potential within zero liquid discharge and circular economy strategies [21–23]. However, its application to digestates and SLD remains unexplored. The present study evaluates the effectiveness of ASEC for treating an SLD generated from animal-derived digestates associated with a biogas production context based on analyses of samples and by-products.

2. Materials and Methods

2.1. ASEC Technology

The ASEC system is an innovative treatment technology designed to achieve zero liquid discharge (ZLD) in contaminated aqueous matrices. It operates through the atomisation of liquid streams into micron-sized droplets using high-frequency sonic energy, which promotes rapid adiabatic evaporation under moderate temperature conditions. The evaporated fraction is subsequently condensed to obtain recovered water, while dissolved salts and nutrients are concentrated into a dry solid residue with energy consumption of approximately 20 kWh per cubic meter [20,23]. In this study, the ASEC treatment was performed using a pilot-scale unit in a single bath experiment (100 L of SLD, 1.20% dry matter) achieving two recovered fractions: recovered water and a nutrient-rich solid fraction (Figure 1). It was possible to recover 96% of the water and 1.26 kg of dried solid (<5% moisture) with energy consumption of 19.7 kWh/m^3.

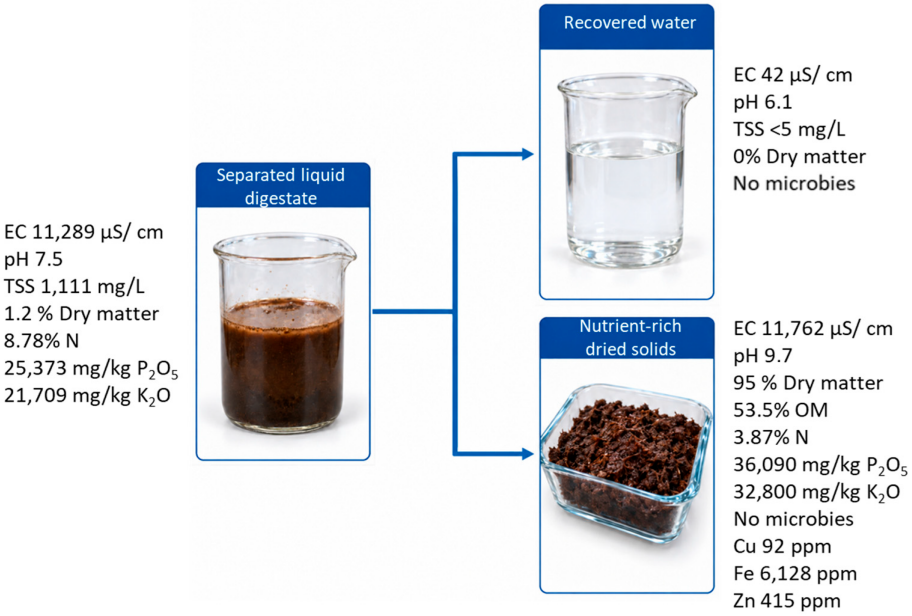


Figure 1. Samples before and after ASEC treatment: the separated liquid digestate was fractionated into recovered water and a nutrient-rich dry solid fraction.

2.2. Sampling and Analytical Procedures

Liquid samples (animal-derived SLD and recovered water) were collected in sterile bottles and stored at 4 °C until analysis. The recovered solid fraction was collected in a sterile plastic container. Samples were analysed by certified laboratories.

For liquid samples, EC and pH were determined at 25 °C by conductimetry and potentiometry, respectively. In the solid fraction, both parameters were measured in a 1:5 aqueous extract. Turbidity was determined by UV-Vis spectrophotometry; TSS and dry matter were determined by gravimetry, total organic matter by calcination, and oxidisable organic matter by UV-Vis spectrophotometry. Total nitrogen was determined by Dumas combustion, ammoniacal N by volumetry, nitric N by ion chromatography, and organic and ureic nitrogen by calculation. Phosphorus was analysed as total P_2O_5, Olsen P_2O_5 and water-soluble P_2O_5, using ICP-OES, UV-Vis spectrophotometry and ion chromatography, respectively. Elements (As, Cd, Cu, Cr, Fe, Hg, K, Ni, Pb and Zn) were determined by ICP-OES or ICP-MS, while Cr(VI) was determined by UV-Vis spectrophotometry. Microbiological analyses included *Salmonella* spp. (UNE-EN ISO 6579-1:2017), *Escherichia coli* (Colilert-18), *Legionella* spp. (UNE-EN ISO 11731:2017) and intestinal nematode eggs (by counting). In a complementary condensate sample, pesticide residues were analysed by gas chromatography coupled to tandem mass spectrometry (GC-MS/MS). Because the study was based on a single ASEC treatment batch, results are reported as analytical values for the tested samples rather than as replicated means with statistical variability.

3. Results and Discussion

3.1. ASEC-Recovered Water

Table 1 summarises the characterisation of the SLD before and after ASEC treatment. The raw digestate was a nutrient-rich matrix with low dry matter content (1.2%) and high EC (11,289 µS/cm), with nutrient concentrations including 8.78% total N, 5.58% ammoniacal N, 25,373 mg/kg total P_2O_5, 2690 mg/kg Olsen P_2O_5, 2464 mg/kg water-soluble P_2O_5 and 21,709 mg/kg K_2O. The BOD_5/COD ratio was approximately 0.20, indicating a relatively low biodegradable organic fraction, consistent with an already digested effluent.

Table 1. Characterisation of raw animal-derived SLD and recovered water after ASEC treatment compared with Regulation (EU) 2020/741 for agricultural water reuse.

Parameter	Unit	SLD	ASEC-Recovered Water	EU Regulation
EC (25 °C)	µS/cm	11,289	42	n.r.
pH (25 °C)		7.5	6.1	
Turbidity	UNF	n.a.	<1	<5
TSS	mg/L	1111	<5	≤10 for Class A; ≤35 for Classes B–D
Dry matter	%	1.20	<0.01	
Oxidisable organic matter	% dm	16.8	<0.1	
Organic matter	% dm	35.8	<0.1	
Ammonium (NH_4)	% dm	5.58	<0.000015	
Organic N	%	3.09	<1	
Nitrate N	%	0.1061	<1	
Total N	%	8.78	<1	
Ureic N	%	<0.50	<0.50	
Nitric N	mg/kg	1061	<1	
Olsen P_2O_5	mg/kg	2690	<0.01	
Water-soluble P_2O_5	mg/kg	2464	<0.01	
Total P_2O_5	mg/kg	25,373	<0.01	
K_2O	mg/kg	21,709	<1	
Salmonella spp.		n.d.	n.d.	
BOD_5	mgO_2/L	4894	<10	
COD	mgO_2/L	24,850	<15	
Escherichia coli	MPN/100 mL	0	0	≤10/100 mL for Class A; ≤100 for Class B; ≤1000 for Class C; ≤10,000 for Class D
Legionella spp.	CFU/1000 mL	n.a.	n.d	<1000 CFU/L
Intestinal nematode	eggs/L	n.a.	0	≤1 egg/L

n.d.: not determined; n.a.: not analysed.

After ASEC treatment, the recovered water showed a strong reduction in mineral load. EC decreased to 42 µS/cm, turbidity was below 1 UNF, and total suspended solids (TSS) were below 5 mg/L. *Salmonella* spp. and *Legionella* spp. were not detected, *Escherichia coli* was at 0 MPN/100 mL, and intestinal nematode eggs were absent. These results indicate that ASEC generated a clarified, low-conductivity recovered water stream from the original digestate. Microbiological indicators were not available for the inlet SLD in the same analytical batch; therefore, these results were interpreted as quality indicators of the recovered fractions rather than as removal efficiency calculations.

This result is technologically relevant because SLD management is often limited by high water content, salinity and ammonium load, which increase storage and transport costs and may contribute to ammonia volatilisation, nitrate leaching and soil salinisation when mismanaged [5,7]. Nevertheless, the recovered water should not be classified as irrigation-ready or discharge-compliant based only on the available dataset. Regulation (EU) 2020/741 requires risk-based assessment and specific quality standards for agricultural water reuse [24]. This was used only as a reference framework for preliminary screening, not as a formal compliance assessment.

3.2. Complementary Condensate Characterisation

In addition to that for the main ASEC-recovered water sample, a complementary condensate analysis is available in Table 2. This sample was analysed under the Spanish drinking-water quality framework established by RD 3/2023 and should therefore be interpreted as complementary evidence of condensate quality. The condensate showed low mineralisation, with EC below 50 µS/cm, a chloride concentration of 9.63 mg/L, Na at 13.86 mg/L and a sulphate concentration of 0.89 mg/L. The organic load was also low, with TOC under 1 mg/L and permanganate oxidisability below 0.5 mg O_2/L. Nitrogen species were present at low concentrations, with nitrate at 8.49 mg/L and nitrite below 0.0250 mg/L, while ammonium was below 0.05 mg/L. The pH was 6.69, and the turbidity was below 0.20 NTU. Regarding trace elements, As, Cd, Pb, Mn and U were below quantification limits, while Cu, Cr, Fe, Hg, Ni and Se were detected at low concentrations (between 0.05 and 56.7 µg/L). Microbiological indicators were favourable, with *Escherichia coli*, total coliforms, intestinal enterococci, *Clostridium perfringens* and somatic coliphages reported as 0 units/100 mL, and colony counts at 22 °C under 1.0 CFU/mL.

Table 2. Complementary characterisation of the ASEC condensate sample under Royal Decree 3/2023.

Parameter	Unit	Condensate	Reference RD 3/2023 Framework
EC (20 °C)	µS/cm	<50.0	2500
TOC	mg/L	<1.0	5.00
pH		6.69	6.5–9.5
Turbidity	NTU	<0.20	4.00
Ammonium	mg/L	<0.05	0.50
Nitrate	mg/L	8.49	50.0
Nitrite	mg/L	<0.025	0.10
Permanganate oxidisability	mg O_2/L	<0.50	5.00
Chlorides	mg/L	9.63	250
Sulphates	mg/L	0.89	250
Total pesticides	µg/L	<0.03	0.50
Sum of 20 PFAS	µg/L	<0.01	0.10
Total trihalomethanes	µg/L	28.4	100
As	µg/L	<1.00	10.0
B	mg/L	0.08	1.00
Cd	µg/L	<0.02	5.00
Cu	mg/L	0.005	2.00
Cr	µg/L	17.6	50.0
Fe	µg/L	56.7	200
Mn	µg/L	<2.50	50.0
Hg	µg/L	0.23	1.00
Na	mg/L	13.86	200

Table 2. *Cont.*

Parameter	Unit	Condensate	Reference RD 3/2023 Framework
Ni	µg/L	15.2	20.0
Pb	µg/L	<0.50	10.0
Se	µg/L	0.27	20.0
U	µg/L	<1.00	30.0
Colony count (22 °C)	CFU/mL	<1.00	100
Escherichia coli	CFU/100 mL	0	0
Total coliforms	CFU/100 mL	0	0
Intestinal enterococci	CFU/100 mL	0	0
Clostridium perfringens	CFU/100 mL	0	0
Somatic coliphages	PFU/100 mL	0	0

The complementary condensate characterisation included a broad pesticide screening comprising 79 individual pesticide substances (Supplementary Material Table S1), grouped as general pesticides, organophosphorus pesticides, organochlorine pesticides, organonitrogen pesticides, carbamates, pyrethroids, phenylurea herbicides and other pesticides. All individual pesticide concentrations were below their corresponding quantification limits, and the total pesticide concentration was below 0.030 µg/L, below the parametric value of 0.50 µg/L established under RD 3/2023.

3.3. Characterisation of the Recovered Solid Fraction

The recovered solid fraction retained after ASEC treatment (Table 3) had less than 5% moisture, confirming effective concentration of the digestate solids. This is a key operational advantage, because the high water content of digestate is one of the main factors limiting its storage, transport and agricultural use [5,6]. The solid fraction had a pH of 9.7 and EC of 11,762 µS/cm (in aqueous extract), with a relevant organic fraction (53.5% total OM and 41.1% oxidisable OM). These values suggest potential use as an organic-mineral amendment, although its agronomic performance will depend on its stability, mineralisation dynamics and soil conditions [7].

Table 3. Characterisation of the recovered solid fraction after ASEC treatment (over dry matter) compared with a European Regulation for solid organic fertilisers (2019/1009).

Parameter	Unit	Recovered Solid Fraction	EU Regulation (2019/1009)
EC (25 °C)	µS/cm	11,762	
pH (25 °C)		9.7	
Dry matter	%	95.0	
Total organic matter	%	53.5	
Oxidisable organic matter	%	41.1	
Total nitrogen	%	3.87	≥2.5% N if N is the only declared primary nutrient in solid organic fertiliser

Table 3. *Cont.*

Parameter	Unit	Recovered Solid Fraction	EU Regulation (2019/1009)
Organic nitrogen	%	3.78	
Ammoniacal nitrogen	%	0.12	
Ureic nitrogen	%	<0.50	
Nitric nitrogen	mg/kg	33.3	
Total phosphorus	mg/kg	36,090	≥2% P_2O_5 if P is the only declared primary nutrient in solid organic fertiliser
Olsen phosphorus	mg/kg	1240	
Water-soluble P	mg/kg	318	
Total K_2O (K_2O)	mg/kg	32,800	
As	mg/kg	5.42	≤40 mg/kg dm inorganic As
Cd	mg/kg	0.24	≤1.5 mg/kg dm
Cu	mg/kg	92.6	≤300 mg/kg dm
Cr	mg/kg	21.1	
Fe	mg/kg	6128	
Hg	mg/kg	0.056	≤1 mg/kg dm
Ni	mg/kg	12.3	≤50 mg/kg dm
Pb	mg/kg	11.8	≤120 mg/kg dm
Zn	mg/kg	415	≤800 mg/kg dm
Cr(VI)	mg/kg	<0.50	≤2 mg/kg dm
Salmonella spp.	in 25 g	n.d.	Absence in 25 g
Escherichia coli	MPN/g	0	≤1000 CFU/g

The recovered solid contained 3.87% total N, of which 3.78% corresponded to organic N, while ammoniacal N was low (0.12%) and ureic N was below 0.50%. This profile suggests that the solid fraction may exhibit a slower nitrogen release pattern. Phosphorus was also concentrated in the solid, with 36,090 mg/kg total P_2O_5, although Olsen P_2O_5 and water-soluble P_2O_5 were lower, at 1240 and 318 mg/kg, respectively. Potassium was also present at a relevant concentration, with 32,800 mg K_2O/kg dm. Therefore, considering its total N, P_2O_5 and K_2O contents, the recovered solid can be cautiously described as a candidate organic-mineral NPK fertilising material. This terminology is compositional and preliminary, because nutrient availability, release kinetics, stability and agronomic efficiency were not assessed in this study. Therefore, the fertilising value of this material cannot be inferred from its total nutrient content alone; nutrient availability should be confirmed through incubation assays and plant-growth trials [25,26].

The recovered solid retained trace elements (Table 3) in the following order: Fe (6128 mg/kg dm) > Zn (415 mg/kg dm) > Cu (92.6 mg/kg dm) > Cr (21.1 mg/kg dm) > Ni (12.3 mg/kg dm) > Pb (11.8 mg/kg dm) > As (5.42 mg/kg dm) > Cd (0.24 mg/kg dm) > Hg (0.056 mg/kg dm). The concentrations of Cd, Cr(VI), Hg, Ni, Pb, Cu and Zn were below the contaminant thresholds established for organic fertilisers under Regulation (EU) 2019/1009 [15]. Total As was also low, although this comparison should be interpreted cautiously because the Regulation refers to inorganic As, whereas the present study measured total As. Cu and Zn require attention because these elements are commonly associated with animal-derived residues and may accumulate in agricultural soils after repeated

applications [12,24,27]. Therefore, future application rates should consider not only NPK supply but also cumulative trace-element loading.

Microbiological indicators were favourable, with *Salmonella* spp. not detected and *Escherichia coli* reported as 0 MPN. These results support the hygienic quality of the analysed solid fraction, but they do not constitute full sanitary validation. Compliance with Regulation (EU) 2019/1009 [15] would require confirmation of the applicable product function category, component material category, stability, hygiene, contaminant limits and quality-control requirements.

3.4. Valorisation Potential and Process Limitations

The results support ASEC as a promising technology for digestate volume reduction, water recovery and nutrient concentration. Unlike mechanical separation, which only redistributes solids and nutrients between liquid and solid fractions, ASEC generated a low-conductivity recovered water stream and a dry organic-mineral solid. Compared with membrane-based systems, ASEC avoids producing a liquid concentrate requiring further management, although its energy demand and scalability must still be evaluated for digestate-specific applications. Conventional digestate treatments such as solid–liquid separation, ammonia stripping, membrane filtration and nutrient precipitation each address specific fractions or compounds, but their performance is often constrained by fouling, chemical demand, concentrate management or incomplete nutrient recovery [16,17].

The main value of ASEC in this study is therefore not selective nutrient crystallisation but integrated phase separation. The process produced a clarified recovered water stream and a nutrient-rich solid fraction, which together address two central limitations of digestate management: excessive liquid volume and low nutrient density. However, claims regarding the selective production of ammonium nitrate, phosphates or other defined fertiliser salts should not be made without additional mineralogical or crystallographic evidence.

Several limitations should be considered before assigning final reuse routes to the recovered fractions. For the recovered water, the available dataset confirms low EC, low turbidity, low suspended solids and favourable microbiological indicators, but it does not provide a full reuse assessment as a function of the final destination of this water, including complete organic load characterisation, major ions, trace metals, organic micropollutants and repeated-batch variability. In previous works depurating other kind of fluids these compounds were reported as being non-present in ASEC-condensated water, so further work is expected to have the same results. In any case, the recovered water should be considered a low-conductivity recovered stream that needs to analysed for each reuse proposed to demonstrate it. For the solid fraction, the available data support its preliminary description as a candidate organic-mineral NPK fertilising material. Finally, this study was based on a single treatment sequence, whereas digestate composition can vary substantially depending on the feedstock, digestion conditions and storage [5,6]. Future work should include replicated batches, measured inlet and outlet flows, mass and energy balances, nutrient-release assays, plant-growth tests, ecotoxicological assessment and full regulatory classification of the recovered solid.

3.5. Preliminary Economic Evaluation of the Recovered Solid Fraction

The preliminary economic evaluation was based on the analytical composition of the recovered solid fraction and on a treatment scenario of 100 m^3/d of SLD (1.20%), assuming a digestate density of 1 t/m^3 and complete retention of inlet dry matter in the recovered solid (Table 4). Therefore, the material most closely resembles a dry organic-mineral NPK fertilising material rather than a conventional mineral fertiliser.

Table 4. Yearly estimated NPK nutrient equivalence value of the recovered solid (95%) for an ASEC treatment scenario of 100 m^3/d of separated liquid digestate and feed density 1 t/m^3.

Nutrient	Production in Recovered Solid (t)	Fertiliser Reference	Nutrient Content in Reference Fertiliser	Equivalent Fertiliser Production (t)	Price (USD) *	Estimated Value (USD)
Total N	16.95	Urea	46% N	36.85	850	31,322
Total P_2O_5	15.81	TSP	46% P_2O_5	34.36	658.10	22,615
Total K_2O	14.37	KCl/MOP	60% K_2O	23.94	401.30	9606
Total NPK						**63,543**
Value per recovered solid	**461.1 t solid/yr**					**137.8 USD/t solid**
Value per SLD treated	**36,500 m^3/yr**					**1.74 USD/m^3 SLD**

* World Bank/YCharts commodity prices framework, April 2026.

Based on the recovered solid composition (Table 3) and the previous scenario, the estimated annual production would be 461.1 t of recovered solid and 36,038.9 m^3 of recovered water. Therefore, the recovered solid would retain approximately 234.3 t/year of organic matter, 16.95 t/year of total N, 15.81 t/year of P_2O_5 and 14.37 t/year of K_2O. The nutrient equivalence value was estimated using urea as the reference fertiliser for N, triple superphosphate as the reference fertiliser for P_2O_5 and potassium chloride or muriate of potash as the reference fertiliser for K_2O (reference prices [28–30]; Table 4). Based on this scenario, the estimated NPK nutrient replacement value was 63,543 USD/year, equivalent to 137.8 USD/t of recovered solid or 1.74 USD/m^3 of SLD treated.

This value should be considered a partial nutrient equivalence estimate rather than a market price for the recovered solid. It does not imply that the recovered solid is equivalent to soluble mineral NPK fertilisers, because most nitrogen was present as organic N and the availability of P_2O_5 and K_2O was not assessed through nutrient-release or plant-growth assays. It also excludes the potential value of organic matter, recovered water reuse, avoided transport, avoided storage and environmental benefits, as well as costs associated with post-treatment, product conditioning, certification, packaging, transport and regulatory registration.

The recovered solid also showed a favourable preliminary metal profile. Cd, Hg, Ni, Pb, Cu and Zn were below the contaminant limits used as references under Regulation (EU) 2019/1009 [15], while Cr(VI) was below the analytical quantification limit (Table 3). Total As was low, although the regulatory comparison remains conservative because the regulation refers to inorganic As. In the recovered solid, Cu and Zn were below these thresholds, but they should still be considered in future agronomic assessments because both elements are relevant in animal-derived residues and may accumulate in soil after repeated applications.

4. Conclusions

This case study evaluated the application of ASEC to an animal-derived SLD, focusing on the separation of the original matrix into a recovered water fraction and a dry organic-mineral solid fraction. The recovered water showed very low electrical conductivity (<50 µS/cm), low turbidity (<29 NTU), low suspended solids (<5 mg/L), pH~ 6 and favourable microbiological indicators, suggesting its potential to reduce the liquid burden associated with digestate management. The recovered solid contained less than 5% moisture, high organic matter content (53.5%), relevant total N (3.87%), total P_2O_5 (36,090 mg/kg dm) and total K_2O (32,800 mg/kg dm), supporting its preliminary description as a candidate organic-mineral NPK fertilising material. The analysed metals and

metalloids were below the key contaminant thresholds used as references for organic fertilisers under Regulation (EU) 2019/1009, although Cu and Zn should be considered in future cumulative soil-loading assessments.

For a simulated 100 m^3/d SLD (1.20% dry matter) treatment scenario, the estimated mass balance indicated the annual generation of 36,039 m^3 of recovered water and 461.1 t of recovered solid. Based on the N, P_2O_5 and K_2O contents of the solid fraction, the theoretical NPK nutrient replacement value was estimated at 63,543 USD/year, equivalent to 137.8 USD/t of recovered solid or 1.74 USD/m^3 of SLD treated. These values should be interpreted as preliminary scenario-based estimates rather than measured process-scale economic returns. Overall, this is a first appraisal of the valorisation potential of ASEC-derived by-products; the recovered water requires further chemical validation depending on its reuse pathway (industrial, irrigation, etc.), and the solid fraction requires agronomic, ecotoxicological and regulatory assessments before direct agricultural application or commercial classification as a fertilising product.

Supplementary Materials: The following supporting information can be downloaded at https://www.mdpi.com/article/10.3390/app162110559/s1. Table S1, Pesticides analysed (in µg/L) in the complementary condensate sample under Real Decreto 3/2023.

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