

Transforming Dairy Processing Sludge and their Digestate via Ultrasound-Assisted Humic Substances Extraction: A Sustainable Resource Recovery Study

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The rapid expansion of biomethane infrastructure across Europe necessitates a strategic transition from conventional sludge management toward the digestate valorisation, thereby enhancing resources recovery within the anaerobic digestion (AD) value chain. The deployment of AD across the sectors is driven by decarbonisation targets and circular bioeconomy strategies. The global dairy processing industry generates substantial effluent streams, primarily dairy wastewater (DWW) and dairy processing sludge (DPS). Within the EU-27, annual DWW generation is estimated at approximately 192.5×10^6 m³ (Stasinakis et al., 2022). For every 100 L of milk processed, approximately 2 kg of sludge and 1,000 L of wastewater are produced (Mohapatra et al., 2025), typically reduced by 80–90% through downstream biological treatment (Ashkuzzaman et al., 2019; Bella & Rao, 2021). While AD effectively stabilises readily degradable organics, it transfers the management challenge to digestate, which retains high organic matter (~70%) and significant nutrient concentrations, including ammoniacal nitrogen (70-80 % NH₄-N), phosphorus (55-65 % PO₄-P), potassium, and micronutrients (Logan et al., 2022). This composition positions DPS and dairy processing sludge digestates (DPSD) as promising feedstock for value-added resource recovery.

This study investigates the recovery of humic substances (HS), humic acid (HA) and fulvic acid (FA), from DPS and DPSD using conventional alkaline extraction (CE) and an intensified ultrasound-assisted extraction (UAE) approach. While CE remains the most widely applied method, it is constrained by slow mass transfer, longer duration, high reagent demand, and elevated energy consumption (Allahkarami et al., 2025). UAE was evaluated as a process intensification strategy to enhance matrix disruption, improve solvent penetration, and reduce extraction time and energy requirements.

Process optimisation using response surface methodology yielded HA recoveries of 303.63 mg g⁻¹ d.b. for DPS and 429.64 mg g⁻¹ d.b. for DPSD under optimised UAE conditions (100% amplitude, 16.9 min). Compared to CE, UAE increased HA recovery by 365.76% (DPS) and 383.12% (DPSD), while FA recovery improved by 58.68% and 46.27%, respectively. The UAE reduced the process time by 98.24% (960 to 16.9 min), and energy consumption by 87.54% (0.281 to 0.035 kWh), demonstrating substantial process intensification (Table 1). Experimental results closely matched model predictions within a 3.5% error margin, confirming model robustness

and operational reliability.

Table 1. Humic substance yields obtained using conventional extraction (CE) and ultrasound-assisted extraction (UAE), expressed as mean \pm standard deviation on a dry basis (d.b).

Label:	DPS	DPS	%	DPSD	DPSD	%
Extraction method:	CE	UAE	Change	CE	UAE	Change
Extraction time (min)	960	16.9	-98.24	960	16.9	-98.24
Power \pm S.D. (kWh)	0.281 \pm 0.00	0.035 \pm 0.00	-87.54	0.281 \pm 0.00	0.035 \pm 0.00	-87.54
HA \pm S.D. (mg.g _{db} ⁻¹)	65.19 \pm 0.33	303.63 \pm 0.05	365.76	88.93 \pm 0.32	429.64 \pm 0.07	383.12
FA \pm S.D. (mg.g _{db} ⁻¹)	403.27 \pm 0.51	639.92 \pm 0.29	58.68	380.30 \pm 0.69	556.29 \pm 0.22	46.27

Spectroscopic analysis revealed increased abundance of chromophoric and functional groups, including nitro ($-\text{NO}_2$), amine ($-\text{NH}_2$), and alkene ($\text{C}=\text{C}$) functionalities, as well as aromatic ($-\text{C}=\text{C}-$) and ketonic ($\text{C}=\text{O}$) structures in digestate-derived HA compared to coal-based standard HA. Elemental profiling indicated elevated nitrogen incorporation associated with digestate feedstocks. Despite minor differences between extraction routes, HS recovered via UAE retained key phenolic and carboxylic functionalities and exhibited enhanced functional diversity. Overall, ultrasound-assisted extraction enables highly efficient valorisation of dairy AD by-products by dramatically reducing energy demand while simultaneously increasing humic substance yield and shortening processing time. The simultaneous achievement of $>85\%$ energy reduction and multi-fold yield enhancement demonstrates UAE as a viable process-intensification pathway for sustainable resource recovery. This approach supports circular bioeconomy implementation by transforming digestate from a residual management burden into a high-value product stream, enabling cleaner production and improved resource efficiency in the agri-food sector.

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