*Article*

# The Potential of Digestate and the Liquid Fraction of Digestate as Chemical Fertiliser Substitutes under the RENURE Criteria

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**Abstract:** This study assessed how digestate and the liquid fraction (LF) of digestate would perform as candidate RENURE fertilisers (recovered nitrogen from manure) in nitrate vulnerable zones under the proposed criteria of the Joint Research Centre, namely, (i) a mineral nitrogen to total nitrogen ratio $\geq 90\%$ ($N_{min}$:TN $\geq 90\%$) or a total organic carbon to TN ratio $\leq 3$ (TOC:TN $\leq 3$); (ii) limits of $\leq 300$ copper (Cu) mg kg$^{-1}$ and $\leq 800$ Zinc (Zn) mg kg$^{-1}$. These criteria were applied to unpublished data (n = 2622) on digestate compositional properties, further amended with data from the literature (n = 180); digestate analysis from seven full-scale biogas facilities (n = 14); and biogas industry stakeholders (n = 23). The results showed that Cu and Zn mostly met the criteria, with compliance rates of 94.7% (of 1035 entries) and 95.0% (of 1038 entries), respectively. Just above 5% (of 1856 entries) met the $N_{min}$/TN $\geq 90\%$ criterion, while 36% (of 1583 entries) met the TOC/TN $\leq 3$ criterion, while total compliance was 32% (of 1893 entries). When targeting the LF, total compliance increased noticeably, between 43 and 58% depending on DM range, indicating that LFs are better suited RENURE candidate fertilisers than unseparated digestate.

**Keywords:** RENURE; digestate; manure; Nitrates Directive; SAFEMANURE; NUE; NFRV; liquid fraction; circular economy

## 1. Introduction

When poorly managed, livestock manure and slurries can lead to considerable amounts of methane ($CH_4$) and ammonia ($NH_3$), and to a lesser extent nitrous oxide ($N_2O$), being emitted into the environment [1]; as well nutrient leaching, mainly of nitrogen (N) and phosphorous (P) [2]. In the European Union (EU-28), it is estimated that the agricultural sector contributed almost 10% to total greenhouse gas (GHG) emissions in 2015, with manure management being responsible for 15% of the emissions within the agricultural sector [3]. As livestock husbandry in Europe follows a general trend towards intensification, as a corollary, considerable volumes of manure need to be processed safely to protect the environment (N leaching) and human health. Moreover, increased area specialisation tends to further compound geographical imbalances of nutrients, with N surpluses overlapping areas traditionally associated with intensive livestock production [4,5]. Under the Nitrates Directive (ND) (91/676/EEC), the use of animal manure is limited to 170 kg N ha$^{-1}$ y$^{-1}$ in areas where the Nitrates Action Programme applies—the so-called nitrates vulnerable zones (NVZ), which cover about 61% of European agricultural land [6]—to protect waters against pollution caused by nitrates ($NO_3^-$) from agricultural sources.

The 2030 Climate and Energy Framework includes EU-wide binding targets to cut GHG emissions by at least 40% below 1990 levels and increase the share of renewable energy by at least 32% in the final consumption by 2030. In the frame of the European Green Deal, the longer-term ambition is to reach net-zero GHG emissions by 2050. Anaerobic digestion (AD)—which produces renewable power, heat, and fuel from organic waste—can

be expected to play an important role in achieving the goals the EU has set for itself. The potential of biomethane in combination with carbon (C) capture and storage to decarbonise the gas grid [7,8] and the transport sector [9] has been documented. However, AD could also play a crucial part in the sustainable management of organic waste streams such as manure by simultaneously providing renewable energy, closing nutrient loops, and reducing GHG emissions [10,11].

The adoption of the new Fertilising Products Regulation (FPR) ((EU) 2019/1009) opens the door to organic and waste-derived fertilisers under a unified European market. Nevertheless, the current limit of 170 kg N ha$^{-1}$ y$^{-1}$ in NVZ applies to any fertilising product that is partially or entirely derived from livestock manure. Consequently, this legally binding threshold is possibly hampering the use of certain bio-based fertilising materials, which, in some cases, bear little to no resemblance to the manure from which their nutrients were extracted, as they can contain high amounts of mineral N ($N_{min}$), effectively bringing them closer to chemical fertilisers and further away from raw manure in terms of plant nutrient uptake (Figure 1). As things stand currently, farmers tend to top up with energy-demanding chemical fertilisers as a result of the 170 kg N ha$^{-1}$ y$^{-1}$ limit not meeting most crop requirements.
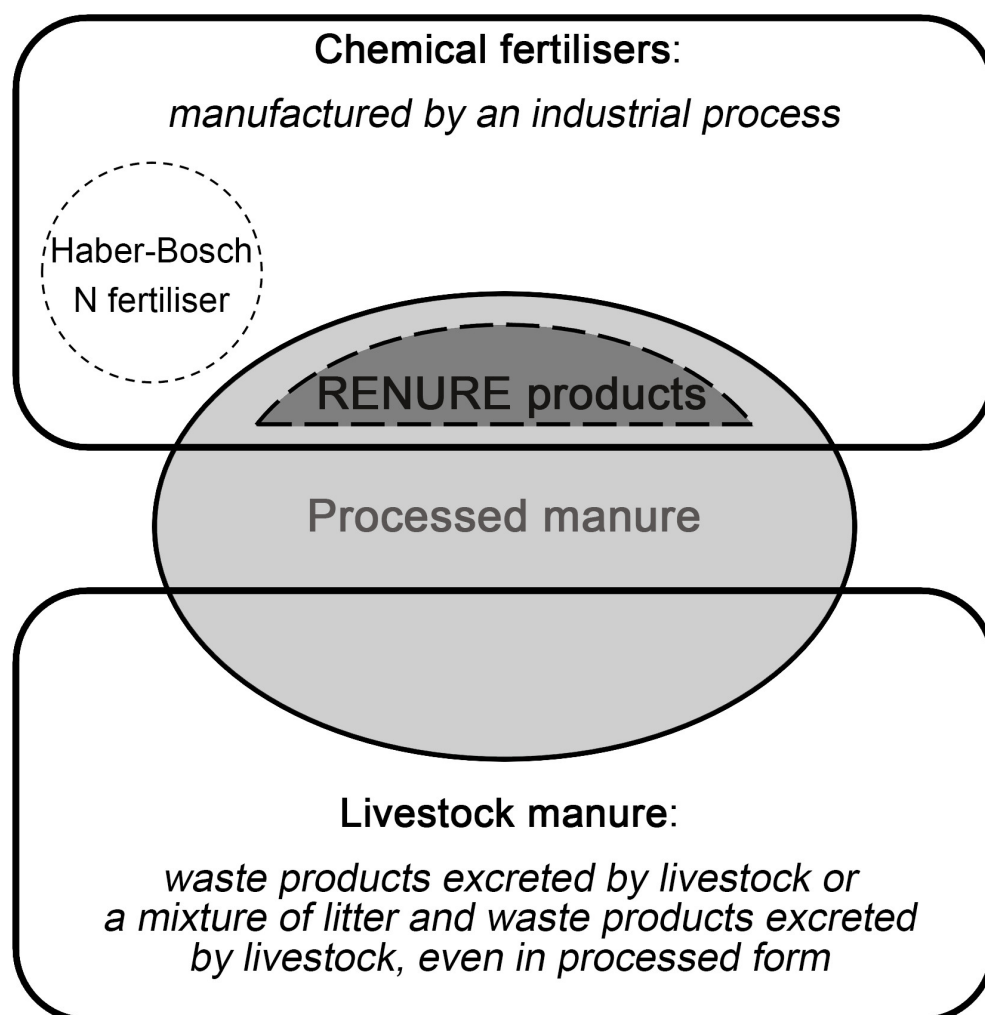


**Figure 1.** The diagram aims to clearly differentiate (i) unprocessed livestock manure; (ii) livestock manure that underwent some form of processing, but does not meet the RENURE requirements; (iii) chemical fertilisers; and (iv) RENURE-compliant "hybrids" derived from animal manure. Adapted from the RENURE study [12].

To lift barriers for such manure-derived products, the European Commission (EC) set up the SAFEMANURE study group to develop criteria for the safe use of processed manure materials in NVZ above the limits established in the ND (91/676/EEC). Such materials are known under the acronym 'RENURE' for "recovered nitrogen from manure", and are defined as "*any nitrogen containing substance fully or partially derived from livestock manure through processing that can be used in areas with water pollution by nitrogen following otherwise identical provisions applied to nitrogen containing chemical fertilisers as defined in the Nitrates Directive (91/676/EEC), while ensuring the achievement of the Nitrates Directive's objective and providing adequate agronomic benefits to enhance plant growth*" [12] (Figure 1).

In alignment with the ND's main goal to protect waterbodies against $NO_3^-$ pollution, the primary objective of the RENURE study was to assess the agronomic performance of candidate fertilisers, defined by their leaching potential and nutrient assimilation capacity by plants or nitrogen use efficiency (NUE), which can be defined as the fraction of total N (TN) applied (imported N) absorbed by the plant (exported N). To this end, the nitrogen fertiliser replacement value (NFRV)—defined as the amount of $N_{min}$ fertiliser saved by using organic N materials while reaching the same crop yield [13]—of candidate RENURE products was determined by comparing their NUE against that of a chemical N Haber–Bosch fertiliser. Digestate is featured on the list of potential RENURE materials, albeit as a medium priority product, and has been gaining attention in recent years as a possible substitute to chemical fertilisers [14]. Pooled data compilations in the literature highlight the broad compositional ranges that can be expected from digestate. Dry matter (DM) content from various feedstocks was found to range anywhere between 15 and 457 g kg$^{-1}$ FM; organic matter (OM) between 386 and 770 g kg$^{-1}$ DM; total carbon (TC) and total organic carbon (TOC) between 360 and 450 and 273 and 374 g kg$^{-1}$ DM, respectively; the C/N ratio from 2.0 to 24.8; TN as low as 1.2 up to 15 g kg$^{-1}$ FM; ammonium nitrogen ($NH_4$-N) from 1.5 to 6.8 g kg$^{-1}$ FM; and pH from 7.3 to 9.0 [15–18].

Further treatment of digestate can help improve its environmental and agronomic predictability, while also allowing for lowered transport costs. One such way is mechanical separation (e.g., sedimentation, belt press, screw press, centrifugation), which reshuffles the nutrients into an N- and potassium-rich (K) liquid fraction (LF) and a P-rich solid fraction (SF) [15,19]. The dewatered SF can then more easily be transported while the LF can be applied as fertiliser or undergo further nutrient refinery and solids' removal [20]. Owing to their higher NUE and the incumbent lower risk of N leaching, chemical fertilisers obtained via the Haber–Bosch process were used as a benchmark to assess the suitability of candidate RENURE products. Accordingly, those that perform similarly to chemical fertilisers are proposed to be exempted from the thresholds currently in vigour for fertilising materials partially or entirely derived from livestock manure in NVZ (Figure 1). To assess the agronomic performance of RENURE materials, two key indicators have been put forward: (i) a mineral N to total N ratio equal or superior to 90% ($N_{min}$/TN $\geq$ 90%) or (ii) a total organic C to total N ratio equal or inferior to 3 (TOC/TN $\leq$ 3). Where a higher TOC/TN ratio indicates a more organic-like material, there is a higher likelihood of the N it contains being embedded in the organic matrix, which in turn is positively correlated with the risk of N leaching and negatively correlated with NUE, whereas the opposite holds true for $N_{min}$/TN.

This study aims to assess how digestate and the LF of digestate would perform when set against these RENURE limits. In this comparison, it is anticipated that the LF of digestate better matches these criteria than unseparated digestate. Therefore, a mechanical separation could constitute the lowest prerequisite to benefit from the RENURE status, thus phasing out the use of unseparated digestate in NVZ. Additionally, we assessed the performance of digestate against the heavy metals (HMs) limits that all RENURE fertilisers would have to comply with: (i) 300 mg kg$^{-1}$ DM for copper (Cu) and (ii) 800 mg kg$^{-1}$ DM for Zinc (Zn). To this end, unpublished data (n = 2622) on compositional characteristics of digestate, provided by the European Biogas Association (EBA), were collected from national biogas associations in five countries (Austria, Germany, Finland, France, and United Kingdom)

(Section 2.1.1). A separate digestate database was also created (Section 2.1.2). These two databases were screened and subsequently merged into a single database (Section 2.1) that was set against the RENURE criteria specified above.

## 2. Materials and Methods

### 2.1. Digestate Databases

The databases that were used for this study are detailed in the ensuing sections. For overall clarity and to better differentiate them throughout the remainder of this study, we shall refer to (i) "database A" (Table 1) as described in Section 2.1.1 and (ii) "database B" (Table 1) as described in Section 2.1.2. Databases A and B were subsequently merged together to form "database C", the solidified and final database used in this study (Section 3.3). In all subsequent tables, the mean and median values are based on the available number of datapoints for each considered parameter.

**Table 1.** Average compositional properties of digestate. Left: database A, datapoints obtained from the European Biogas Association (excluding outliers). Right: database B, datapoints compiled from (i) the literature review; (ii) the sampling campaign; and (iii) data collection from industrial stakeholders. Here, DM = dry matter; FM = fresh matter; OM = organic matter; TN = total nitrogen; $NH_4$-N = ammonium nitrogen ($N_{min}$); $K_2O$ = potassium oxide; $P_2O_5$ = phosphorus pentoxide; Zn = zinc; Cu = copper; Hg = mercury; n = digestate sample size.

| Parameter | Unit | (Left) Database A | | | (Right) Database B | | |
|---|---|---|---|---|---|---|---|
| | | Mean $\pm$ Stdev | Median | n | Mean $\pm$ Stdev | Median | n |
| pH | - | $7.9 \pm 0.3$ | 7.9 | 1808 | $8.2 \pm 0.4$ | 8.2 | 119 |
| DM | g kg$^{-1}$ FM | $57 \pm 26$ | 53 | 2100 | $56 \pm 28$ | 52 | 184 |
| OM | g kg$^{-1}$ DM | $699 \pm 106$ | 707 | 1831 | $652 \pm 94$ | 676 | 77 |
| TOC | g kg$^{-1}$ DM | $388 \pm 59$ | 393 | 1831 | $340 \pm 75$ | 352 | 86 |
| TN | g kg$^{-1}$ DM | $102 \pm 53$ | 89 | 1758 | $88 \pm 49$ | 79 | 178 |
| $N_{min}$ ($NH_4$-N) | g kg$^{-1}$ DM | $60 \pm 43$ | 46 | 1945 | $59 \pm 40$ | 46 | 166 |
| $K_2O$ | g kg$^{-1}$ DM | $55 \pm 29$ | 52 | 1447 | $60 \pm 35$ | 55 | 71 |
| $P_2O_5$ | g kg$^{-1}$ DM | $35 \pm 17$ | 33 | 1452 | $40 \pm 28$ | 31 | 126 |
| Zn | mg kg$^{-1}$ DM | $347 \pm 239$ | 299 | 1019 | $751 \pm 848$ | 425 | 37 |
| Cu | mg kg$^{-1}$ DM | $97 \pm 108$ | 70 | 1020 | $233 \pm 368$ | 103 | 39 |
| Hg | mg kg$^{-1}$ DM | $11 \times 10^{-2} \pm 13 \times 10^{-2}$ | $7 \times 10^{-2}$ | 994 | $15 \times 10^{-2} \pm 38 \times 10^{-2}$ | $5 \times 10^{-2}$ | 11 |
| $N_{min}$/TN | % | $58 \pm 18$ | 57 | 1729 | $61 \pm 14$ | 62 | 160 |
| TOC/TN | - | $4.8 \pm 2.8$ | 3.9 | 1518 | $6.5 \pm 9.1$ | 4 | 86 |

### 2.1.1. Database A

A digestate registry containing 2622 entries of compositional data on macro- and microelements, pH, OM, DM, HM, viable weed seeds, and common pathogens was used as starting point for this study. A preliminary screening of the dataset was performed to keep only the datapoints linked to feedstocks that used a combination of animal manure co-digested with biowaste and/or crops, which would make them suitable candidate RENURE materials. A univariate analysis was then carried out to discard unrealistic and potential outliers for each parameter using SPSS Statistics 27 (IBM Corp: Armonk, NY, USA). A multiplication factor of 1.5 of the interquartile range was used to earmark the lower and upper bounds for potential outliers. Following recommendations of our statistical department, the potential outliers were then compared to data from database B (as described in the following section)—obtained from the literature review, the digestate sampling and analysis campaign, and confidential data collected from the biogas industry— to determine whether they would be discarded or kept. As large as the database was, it did not contain any information on whether a separation technology (sedimentation, belt press, screw press, centrifugation) might have been used. Therefore, digestate entries had to be considered as containing unseparated and LFs of digestate based on their DM content. Two methods were employed to target the LF in database A (discussed in Section 4.7): (i) using DM ranges associated with LF referenced in literature; (ii) using the DM ranges

of the positively identified LF cases from database B (Section 4.6). The main results from database A are presented in Table 1.

### 2.1.2. Database B

Database A was further amended with digestate compositional characteristics collected from three additional sources (described in the following subsections): (i) a review of existing literature, (ii) experimental characterisation of fresh digestate samples, and (iii) data collection from industrial biogas stakeholders. The compiled data from these three sources, referred to as database B, can be found in Table 1 (average values from the three individual data sources can be found under Tables A1–A3 of Appendix A). Database B was screened for extreme outliers, defined as three times the value of the interquartile range below the first quartile or above the third quartile. Database B was then used as a reference against which database A was compared. In this way, the lowest and highest values for each parameter found in database B were used to adjust the ranges from database A in a pairwise comparison, thus correcting, when necessary, the values that had erroneously been identified as outliers via the initial statistical screening. Once database A was processed this way, the two datasets (A and B) were combined to form the final database C (Table 2), against which the RENURE criteria were assessed. The main results of the three combined sets of data used for database B are shown in Table 1.

**Table 2.** Left: database C, the final digestate compositional ranges resulting from the screening and subsequent merging of databases A and B. Right: compositional range of the digestate entries from database C that complied with $N_{min}/TN \geq 90\%$ or $TOC/TN \leq 3$. Here, DM = dry matter; FM = fresh matter; OM = organic matter; TN = total nitrogen; $NH_4$-N = ammonium nitrogen ($N_{min}$); $K_2O$ = potassium oxide; $P_2O_5$ = phosphorus pentoxide; Zn = zinc; Cu = copper; Hg = mercury; n = digestate sample size.

| Parameter | Unit | (Left) Database C | | | (Right) Database C: Compliance with $TOC/TN \leq 3$ or $N_{min}/TN \geq 90\%$ | | |
| --- | --- | --- | --- | --- | --- | --- | --- |
| | | Mean $\pm$ Stdev | Median | n | Mean $\pm$ Stdev | Median | n |
| pH | - | $8.0 \pm 0.3$ | 8 | 1905 | $8.0 \pm 0.3$ | 8.1 | 519 |
| DM | g kg$^{-1}$ FM | $57 \pm 26$ | 53 | 2265 | $38 \pm 14$ | 35 | 606 |
| OM | g kg$^{-1}$ DM | $697 \pm 106$ | 705 | 1887 | $612 \pm 86$ | 621 | 575 |
| TOC | g kg$^{-1}$ DM | $386 \pm 61$ | 391 | 1896 | $338 \pm 51$ | 342 | 587 |
| TN | g kg$^{-1}$ DM | $100 \pm 53$ | 87 | 1913 | $158 \pm 45$ | 151 | 606 |
| $N_{min}$ ($NH_4$-N) | g kg$^{-1}$ DM | $60 \pm 43$ | 45 | 2087 | $108 \pm 37$ | 108 | 590 |
| $K_2O$ | g kg$^{-1}$ DM | $56 \pm 29$ | 52 | 1495 | $54 \pm 27$ | 50 | 578 |
| $P_2O_5$ | g kg$^{-1}$ DM | $35 \pm 18$ | 33 | 1555 | $41 \pm 16$ | 40 | 588 |
| Zn | mg kg$^{-1}$ DM | $361 \pm 294$ | 300 | 1035 | $353 \pm 224$ | 296 | 487 |
| Cu | mg kg$^{-1}$ DM | $102 \pm 131$ | 71 | 1038 | $87 \pm 81$ | 60 | 487 |
| Hg | mg kg$^{-1}$ DM | $10.2 \times 10^{-2} \pm 13.2 \times 10^{-2}$ | $7 \times 10^{-2}$ | 984 | $10.5 \times 10^{-2} \pm 15.6 \times 10^{-2}$ | $7 \times 10^{-2}$ | 481 |
| $N_{min}/TN$ | % | $58 \pm 17$ | 57 | 1856 | $70 \pm 17$ | 68 | 590 |
| $TOC/TN$ | - | $4.9 \pm 3.5$ | 3.9 | 1583 | $2.3 \pm 0.9$ | 2.3 | 587 |

#### Literature Review

A dataset containing 180 datapoints of digestate (unseparated and LF) compositional properties was collected from 41 peer-reviewed articles found mainly via the ScienceDirect and Web of Science platforms. The following parameters were compiled: DM; OM; TOC; TN, $NH_4$-N; $P_2O_5$; $K_2O$; Cu; and Zn [21–61]. The average compositional values can be found in Table A1 (Appendix A).

#### Sampling and Analysis Campaign

Fourteen digestate samples were collected from seven full-scale AD facilities throughout Europe (Belgium, Germany, Italy, The Netherlands, UK). These digestates were chosen for their relevance within the scope of the RENURE framework as suitable candidate materials, hence the samples were of either separated LF or unseparated digestate. All

digestate samples were stored at 4 °C in polyethylene containers. DM was determined by placing triplicate samples at 105 °C for 24 h. The oven-dried samples were then placed in a muffle furnace at 550 °C for 4.5 h to determine OM content. pH-KCl was measured with an Orion Star A211 pH meter in a 1/5 (*w/v*) ratio of fresh sample to 1 M KCl. TN and TC were measured on a PRIMACS100 Analyzer series (Skalar B.V., Breda, The Netherlands). $NH_4$-N and $NO_3$-N were determined from digestate filtrates on a continuous flow auto-analyser (Chemlab System 4, Skalar). Previously, the samples were processed in a 1/5 (*w/v*) ratio of fresh digestate to KCl (1 M) and placed in an orbital shaker for 30′. The filtrates were obtained by pouring the KCl with digestate solutions over paper filters (Whatman No. 43). Macro- and micro elements were measured on an inductively coupled plasma emission spectrometer (ICP-OES) (Varian Vista-MPX, Palo Alto, CA, USA). The average results are listed in Table A2 (Appendix A).

Data Collection from Stakeholders

A call for data addressed to stakeholders from the biogas industry resulted in the collection of 23 confidential datapoints from Belgium, Czech Republic, France, Italy, Latvia, and Sweden. Some of these datapoints were averages from several digestate samples, several biogas plants, and in some cases averages over several years; the added value of these entries is thus higher than what the 23 datapoints let on. The average results are listed in Table A3 (Appendix A).

*2.2. Data Uniformization*

2.2.1. Unit Conversions

To ensure uniformity across all datasets, all values were expressed on a DM basis. Consequently, any source that expressed values on a FM basis was converted to DM. Conversely, whenever data were expressed as elemental P and K, these were systematically converted to their oxide forms—$P_2O_5$ and $K_2O$—using the respective multiplication factors of 2.29 and 1.20. Total solids and volatile solids were considered as DM and OM, respectively. Total Kjeldahl nitrogen was considered as TN, as $NO_3^-$ and nitrites ($NO_2^-$) concentrations in digestate and LF of digestate can be considered negligible.

2.2.2. TOC/OM Ratio

Database A (Table 1) did not contain data on TOC, yet it contained 1831 usable entries on OM. As digestate is a heterogenous material that can contain varying amounts of TC and TOC stemming from different types of feedstocks, all the listwise entries pairing OM and TOC (n = 56) were gathered from database B to calculate a median TOC/OM ratio. This method resulted in a 0.55 TOC/OM conversion factor, which is almost on par with the value of 0.56 recommended for use in the FPR ((EU) 2019/1009). This conversion factor was then applied to database A to derive TOC values (Section 3.2).

2.2.3. Mineral Nitrogen Content

While database A (Table 1) did not contain information on $N_{min}$, it did include data on $NH_4$-N. Therefore, the assumption was made that $NH_4$-N could be interchanged with $N_{min}$, as it is the predominant mineral form of N in digestate [15]. The same assumption was made in the RENURE study, where a 1:1 ratio for $NH_4^+/N_{min}$ was also considered.

2.2.4. Decimal Places

For RENURE compliance results, all values were rounded to one decimal place.

**3. Results**

*3.1. Database B*

The average results of digestate properties from (i) the literature review, (ii) the sampling campaign, and (iii) the data collection are presented in Table 1 (right). For DM, an unusually high value of 293 g kg$^{-1}$ FM was discarded. It was identified as digestate

from organic household waste from a mesophilic digestion process [24], which in any case would not be eligible under the RENURE criteria. The interquartile range was 38 to 65 g kg$^{-1}$ FM. Mean and median were 56 and 52 $\pm$ 28 g kg$^{-1}$ FM, respectively. Minimum and maximum values were 8 and 174 g kg$^{-1}$ FM, respectively. Mean pH was 8. An uncharacteristically low pH value of 5.6 for digested cattle slurry [28] was excluded. Mean OM was 652 $\pm$ 94 g kg$^{-1}$ DM with a total range of 541 (minimum and maximum ranges were 354 and 895, respectively).

Mean and median TOC values were 340 and 352 $\pm$ 75 g kg$^{-1}$ DM, respectively. For N, the data compilation showed a mean TN value of 88 $\pm$ 49 g kg$^{-1}$ DM, ranging from 6 to 367. The latter value was reported for the LF of digested pig slurry processed via reverse osmosis [42], which may explain the exceptionally high value relative to other entries in the dataset. The second highest value (229 g kg$^{-1}$ DM) was obtained via the same process and feedstock. These values, although high in comparison with the 78 g TN kg$^{-1}$ DM median value, were kept so as to avoid excluding any digestate upgrading technology, which could prove useful to meet RENURE criteria. One seemingly extreme outlier of 315 g NH$_4$-N kg$^{-1}$ DM was discarded, which stemmed from confidential data shared by a stakeholder. Conversely, two low values (1 and 2 g kg$^{-1}$ DM) were also excluded [24]. This resulted in a mean NH$_4$-N of 59 $\pm$ 40 (46 median) with minimum and maximum values of 13 and 228 g NH$_4$-N kg$^{-1}$ DM, respectively. Mean P$_2$O$_5$ and K$_2$O were 40 $\pm$ 28 and 60 $\pm$ 35 g kg$^{-1}$ DM, respectively.

Mean values for Cu and Zn were 233 $\pm$ 368 mg Cu kg$^{-1}$ DM (median 103) and 751 $\pm$ 848 mg Zn kg$^{-1}$ DM (median 425), respectively. In the case of Cu, minimum and maximum values were 8.6 and 1682 mg Cu kg$^{-1}$ DM, respectively, with the stem and leaf plot considering anything above 741 as an extreme outlier. For Zn, the total range was 0.2 to 3685 mg Zn kg$^{-1}$ DM, and anything above 2112 mg Zn kg$^{-1}$ DM was flagged as extreme. For both Cu and Zn, the flagged values stemmed from the same study [50] where anaerobically digested pig slurry was used. Albeit extreme, these values were kept nonetheless as references owing to known instances of pig manure containing high concentrations of these elements [62,63].

### 3.2. Database A

As mentioned in the 'Materials and Methods' section, the data collected in the section hereabove was compared to, and subsequently merged with, entries from database A, in order to form the final database C used in this study (Table 2). Potential outliers were defined using a 1.5 multiplication factor of the interquartile range as a first approximation. For DM, potential outliers were reported starting from 149 g kg$^{-1}$ FM. However, database B (Table 1, right) displayed values ranging from 7.9 to 174 g kg$^{-1}$ FM. Therefore, 175 g kg$^{-1}$ FM was chosen as the cut-off point, and anything above (values ranging from 176 up until 977 g kg$^{-1}$ FM) was discarded as a probable SF, which was unlikely to meet the RENURE requirements. All the other compositional data associated with values above 175 g kg$^{-1}$ FM were also excluded from the database at this point. The resulting mean and median DM values for database A (Table 1, left) were 57 and 53 $\pm$ 26 g kg$^{-1}$ FM, respectively. For pH, potential outliers were flagged below 7 and above 8.9. As the pH range in database B (Table 1, right) was between 6.9 and 9.0, all pH values within this range in database A (Table 1, left) were kept. This resulted in a final mean pH of 7.9 $\pm$ 0.3 for database A.

For OM, potential outliers were identified below the 360 g kg$^{-1}$ DM threshold, whereas minimum and maximum ranges in database B (Table 1, right) were 354 and 895, respectively. To keep the data as representative as possible, the cut-off point was accordingly lowered to 354, whereas the highest value of 690 g OM kg$^{-1}$ DM in database A was kept. As a result, mean and median OM were 699 and 707 $\pm$ 106 g OM kg$^{-1}$ DM, respectively. Database B contained 56 listwise entries pairing both OM and TOC with a mean TOC/OM of 54% $\pm$ 9.3 (55% median). Applying the median value to the 1831 entries on OM in database A (Table 1, left) resulted in an estimated mean value of 388 $\pm$ 59 g TOC kg$^{-1}$ DM (393 median).

The initial statistical screening identified values above 242 g TN kg$^{-1}$ DM as possible outliers in database A (Table 1, left). In database B, however (Table 1, right), the compiled data reported TN values ranging from 6.5 to 367, the latter value corresponding to processed digestate via reverse osmosis. Consequently, the maximal limit was readjusted to 367 g TN kg$^{-1}$ DM in database A so as to include possible upgrading technologies that would typically result in higher N concentrations. The updated thresholds led to a mean and median of 102 and 89 $\pm$ 53 g TN kg$^{-1}$ DM. The exercise was repeated for NH$_4$-N, for which anything above 179 g kg$^{-1}$ DM was marked as a potential outlier. Turning to the compiled dataset from database B (Table 1, right), NH$_4$-N values fell between 13 and 228 g NH$_4$-N kg$^{-1}$ DM. To avoid excluding valid entries, database A (Table 1, left) was thus expanded to include NH$_4$-N entries up until 228 g NH$_4$-N kg$^{-1}$ DM. This led to a mean and median of 60 and 46 $\pm$ 43 g NH$_4$-N kg$^{-1}$ DM, respectively.

Outliers were discarded in the same way for K$_2$O and P$_2$O$_5$, for which the limits were determined at 117 g K$_2$O kg$^{-1}$ DM and 79 g P$_2$O$_5$ kg$^{-1}$ DM, respectively. A maximum limit of 174 g K$_2$O kg$^{-1}$ DM in database B (Table 1, right) was traced back to the LF of digested pig manure from the sampling campaign. Still, in database B (Table 1, right), the highest P$_2$O$_5$ value, which was 159 g P$_2$O$_5$ kg$^{-1}$ DM, came from the literature review [50]. Therefore, the thresholds in database A (Table 1, left) were adjusted accordingly to reflect these upper limits, resulting in mean values of 55 $\pm$ 29 g K$_2$O kg$^{-1}$ DM and 35 $\pm$ 17 g P$_2$O$_5$ kg$^{-1}$ DM. The highest value for Zn in database A (Table 1, left) was 1831 mg Zn kg$^{-1}$ DM, whereas database B (Table 1, right) showed possible values of up to 3685 mg Zn kg$^{-1}$ DM. Consequently, all Zn entries from database A were kept, resulting in mean and median values of 347 and 299 $\pm$ 239 mg Zn kg$^{-1}$ DM, respectively. For Cu, an upper limit of 1682 mg Cu kg$^{-1}$ DM was found in database B (Table 1, right), whereas one extreme outlier in database A (Table 1, left) was flagged at 1770 mg Cu kg$^{-1}$ DM. The latter value was thus kept in the dataset as a possible, albeit extreme, value (the second highest value was 798 mg Cu kg$^{-1}$ DM). The resulting mean and median values in database A were 97 and 70 $\pm$ 108 mg Cu kg$^{-1}$ DM, respectively.

### 3.3. Database C

The screened results from databases A (n = 2114) and B (n = 194) were subsequently combined to form database C (n = 2308), considered as the final database representative of digestate compositional ranges used for this study. Mean and median results of database C, upon which all subsequent analyses were based, are shown in Table 2 (left). The frequency distributions of DM, OM, TOC, TN, N$_{min}$ (NH$_4$-N), Zn, Cu, and Hg are presented in Figure 2.

The mean N$_{min}$/TN value from the final dataset was 58 $\pm$ 17 (n = 1856), with a 57 median. For TOC/TN, mean and median values were 4.9 and 3.9 $\pm$ 3.5 (n = 1583). Regarding HMs, the final values were 361 $\pm$ 294 mg Zn kg$^{-1}$ DM (n = 1035); 102 $\pm$ 131 mg Cu kg$^{-1}$ DM (n = 1038); and 10 $\times$ 10$^{-2}$ $\pm$ 13 $\times$ 10$^{-2}$ mg Hg kg$^{-1}$ DM (n = 984). The average compositional properties of the digestate cases that complied with either N$_{min}$/TN $\geq$ 90% or TOC/TN $\leq$ 3 are also presented (Table 2, right). Under these criteria, it can be observed that the DM content dropped from 5.3% to 3.5% FM (median values), while median TOC/TN and N$_{min}$/TN went from 3.9 to 2.3 and 57 to 68%, respectively.

A more detailed look at RENURE compliance showed that 5% of the datapoints (n = 1856) met the N$_{min}$/TN $\geq$ 90% criterion, 36% met the TOC/TN $\leq$ 3 criterion (n = 1583), while 32% (n = 1893) met either one or the other criterion (further discussed in Section 4.4). Regarding HMs, 95% met either $\leq$300 mg Cu kg$^{-1}$ DM (n = 1038) or $\leq$800 mg Zn kg$^{-1}$ DM (n = 1035) (further discussed in Section 4.3).
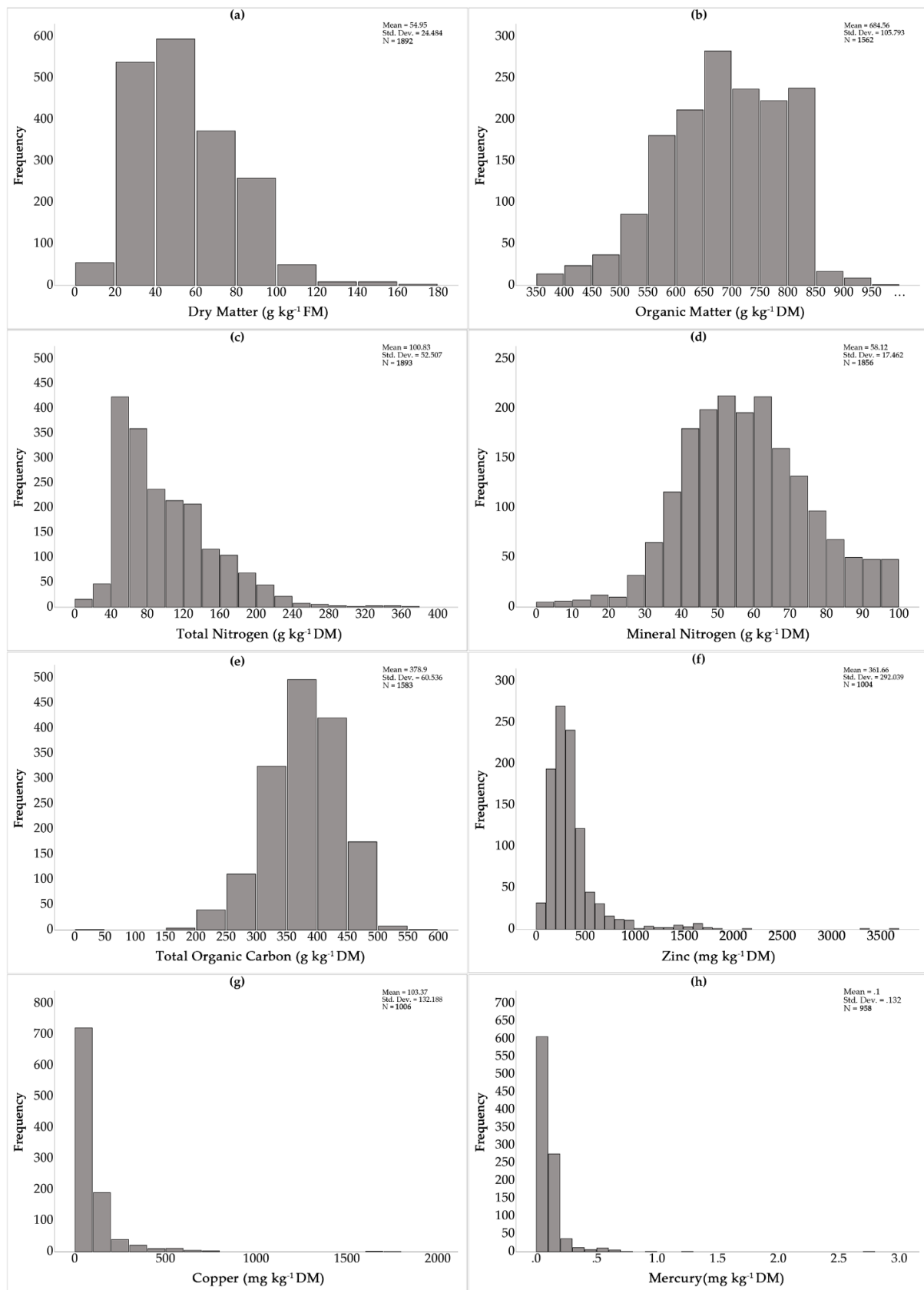
**Figure 2.** Database C's frequency distribution for (**a**) dry matter; (**b**) organic matter; (**c**) total nitrogen; (**d**) mineral nitrogen; (**e**) total organic carbon; (**f**) zinc; (**g**) copper; and (**h**) mercury. Here, n = digestate sample size.

## 4. Discussion

### 4.1. Broad Range of Digestate and Feedstock Characteristics

As a result of digestate chemical properties covering such broad ranges, it was expected that compositional data were in most cases not normally distributed (Shapiro–Wilk test). Conversely, information on whether a separation technology was used (sedimentation, belt press, screw press, centrifugation) was not available from database A (Table 1, left), which further complicated any type of categorisation. Therefore, to err on the side of caution, digestate entries were treated as a whole, as possibly containing a mix of raw and LF of digestate based on the DM content (Section 2.1.1).

### 4.2. Gross Estimation of Nutrients and Carbon Potential from Digestate

The yearly amount of animal manure in Europe is estimated at 1400 Mt [64], which carries with it an energy potential from biogas of 0.64 to 0.92 PJ [65], while the energy of combined agricultural feedstocks (manure, grass, and straw) has been shown to lie between 1.2 and $2.3 \times 10^3$ PJ in the EU in 2030 [66]. With an estimated 180 Mt of digestate produced in the EU-28 each year [67], it is believed that less than 8% of the livestock manure in the EU finds its way to processing facilities [64]. This indicates that, on one hand, there is still significant leeway to increase GHG emission abatement from manure by way of biogas production [11,68] and, on the other hand, that there is still room for growth for nutrient recovery and recycling.

As an order of magnitude, the amount of 180 Mt fresh digestate produced yearly in the EU-28 [67] was used as a starting point to provide a ballpark estimate of the nutrients and C potential it might contain. Based on the assumptions made in this study, the 10th and 90th percentiles from database C characteristics (Table 2) were used as lower and upper ranges, respectively. It was thus calculated that 180 Mt digestate could contain anywhere between 5 Mt and 16.5 Mt DM. In turn, this amount of DM was shown to potentially contain between 2.8 and 13.6 Mt OM; 1.5 and 7.5 Mt TOC; 0.25 to 2.9 Mt TN; 0.09 to 2.1 Mt $N_{min}$; 0.1 to 1.5 Mt $K_2O$; and 0.08 to 0.9 Mt $P_2O_5$ (Table 3). With a 9% increase since 2010, chemical N fertiliser consumption was estimated at 11.6 Mt in the EU-28 in 2017 [69]. The median and upper values for $N_{min}$ (Table 3) would suggest a gross potential from digestate as substitute to Haber–Bosch-derived chemical N-based fertilisers of approximately 4 to 18% (excluding ammonia volatilisation and NFRV), thereby also decreasing the high energy consumption and carbon dioxide emissions associated with the process. Precision injection techniques have been shown to reduce ammonia ($NH_3$) losses; therefore, subtracting a mean approximate value of 10% of the total ammoniacal nitrogen, if precision injection were to be used [51,70], would lead to a corrected estimate ranging from 3 to 16% of total $N_{min}$ consumption. Factoring in a replacement value of 82%, which was the average NFRV associated with the 90% to 100% $N_{min}$/TN pool in the RENURE metanalysis [12], would theoretically result in a gross N substitute value of 3 to 13% (assuming all digestates meet the RENURE criteria). Nevertheless, if the 32% compliance rate (Table 4) is any indication, then a more realistic $N_{min}$ replacement value in the current state of affairs, at least in NVZs, would be 1 to 4% when considering the estimated 180 Mt fresh digestate. With P having been placed on the list of 20 critical raw materials by the EC and considering the possibilities for sustainable use and recycling thereof via AD, P from digestate would represent roughly (when converted to its elemental form) 0.04 to 0.4 Mt P, thus representing, based on the 1.2 Mt P consumed in the EU-28 in 2017 [69], between 3 and 33% in P savings. Furthermore, the AD of feedstocks will have converted between 20 and 95% of the ingoing OM into biogas [15]; therefore, the estimated 1.5 to 7.5 Mt TOC (Table 3) is likely to be in a more stable form. This residual C can thus be considered to have a higher potential for sequestration in the form of soil organic carbon [71] and could thus yield considerable environmental benefits.

**Table 3.** Estimation of gross potential of nutrients and C, on a dry matter basis, contained in a yearly estimate of 180 Mt of digestate. Here, Mt = megatonnes; kt = kilotonnes; FM = fresh matter; DM = dry matter; OM = organic matter; TOC = total organic carbon; TN = total nitrogen; $N_{min}$ = mineral nitrogen; $K_2O$ = potassium oxide; $P_2O_5$ = phosphorus pentoxide.

| | 180 Mt FM y$^{-1}$ Digestate | | |
|---|---|---|---|
| **Parameter** | **kt y$^{-1}$ Equivalent** | | |
| | **10th Percentile** | **Median** | **90th Percentile** |
| DM | 5040 | 9540 | 16,506 |
| OM | 2787 | 6726 | 13,551 |
| TOC | 1547 | 3730 | 7527 |
| TN | 247 | 830 | 2856 |
| $N_{min}$ (NH$_4$-N) | 86 | 429 | 2096 |
| $K_2O$ | 111 | 496 | 1519 |
| $P_2O_5$ | 81 | 315 | 924 |

**Table 4.** Compliance of digestate under the current proposed RENURE criteria. Here, Cu = copper; Zn = zinc; TOC = total organic carbon; TN = total nitrogen; $N_{min}$ = mineral nitrogen; DM = dry matter.

| | $\leq$**300 mg Cu kg$^{-1}$ DM** | $\leq$**800 mg Zn kg$^{-1}$ DM** | **$N_{min}$/TN $\geq$ 90%** | **TOC/TN $\leq$ 3** | **$N_{min}$/TN $\geq$ 90% or TOC/TN $\leq$ 3** |
|---|---|---|---|---|---|
| Compliance (cases) | 986/1038 | 980/1035 | 96/1856 | 574/1583 | 606/1893 |
| Compliance (%) | 95 | 95 | 5 | 36 | 32 |

*4.3. Compliance with $\leq$300 mg Cu kg$^{-1}$ DM and $\leq$800 mg Zn kg$^{-1}$ DM Criteria*

Many investigations showed that Cu and Zn concentrations in pig manure could be significantly higher than for other types of animal manures as a result of these microelements being added to feed to stimulate growth [62,72]. Understandably, these microelements were kept as RENURE criteria as their presence in excess can lead to adverse effects on the environment (soil contamination) and human health (food contamination). However, in adequate amounts, Cu and Zn are essential cofactors in various physiological processes in plants [73,74]. The average Zn and Cu concentrations reported in the Joint Research Centre's (JRC) sampling campaign for LF of digestate were 357 and 127 mg kg$^{-1}$ DM [12], respectively, against 361 and 102 mg kg$^{-1}$ DM in this study (Table 2, left).

The three highest values in the final database C (Table 2, left)—ranging from 2112 to 3685 mg Zn kg$^{-1}$ DM—were from the literature review and were positively identified as digestate from pig slurries. The fourth highest value, 1831 mg Zn kg$^{-1}$ DM, was traced back to database A (Table 1), although information on the type of animal manure was not available. For Cu, the highest value—1770 mg Cu kg$^{-1}$ DM—came from database A (for which the type of animal manure is not specified). The second and third highest concentrations (1611 and 1682 mg Cu kg$^{-1}$ DM) came from the literature review and were linked to digested pig manure. In other terms, when the information was available in the database, the highest values for Cu and Zn were usually associated with pig manure, which tends to justify the need for additional scrutiny for these elements. The majority of entries in database C (Table 2, left) came from database A (Table 1), for which information on the type of manure (pig, bovine, poultry) is not specified. So, while a further categorisation of the types of manure cannot be ascertained, the results from this study tend to show that Cu and Zn from co-digested manure mostly met the RENURE criteria, and thus would not pose any environmental risk: 5.3% of the 1035 considered cases were above the 800 mg Zn kg$^{-1}$ DM limit; 5.0% of the 1038 available cases for Cu were above the 300 mg Cu kg$^{-1}$ DM limit (Table 4).

### 4.4. Compliance with $N_{min}$/TN ≥ 90% or TOC/TN ≤ 3 RENURE Criteria

When considering the $N_{min}$/TN ≥ 90% criterion, 5.2% of the cases met the requirement, i.e., 96 of the 1856 cases presented values of 90% or higher (Table 4). When applying the TOC/TN ≤ 3 criterion, 36.3% of the total 1583 considered cases met the proposed requirement. Total compliance, taking into account both criteria, was 32%.

In the JRC study, candidate RENURE product characterisation pointed towards the TOC/TN criterion displaying slightly more flexibility than the $N_{min}$/TN criterion, though in all likelihood, not to the scale observed in Table 4 (7:1 ratio). For the cases that complied with either of the RENURE criteria, the median TOC/TN value was 2.3 (Table 2, right), indicating that most entries already found themselves in the upper tier of the allowed range.

The TOC/TN ≤ 3 criterion was applied to a listwise comparison of all entries containing data on both RENURE criteria (n = 1546). The resulting 558 compliant cases (36% overall compliance) had mean and median TN values of 157 and 152 ± 37 g kg$^{-1}$ DM, respectively, and $N_{min}$/TN values of 68 and 67 ± 16%, respectively, thereby already indicating that most digestates that met the TOC/TN criterion would not qualify under the 90% $N_{min}$/TN threshold. In fact, of the 558 cases that complied with TOC/TN ≤ 3, less than 10% (53 cases) also complied with the $N_{min}$/TN ≥ 90% criterion. Conversely, applying the $N_{min}$/TN ≥ 90% criterion to the 1546 listwise cases returned 77 compliant entries, of which 69% (53 cases) also complied with the TOC/TN ≤ 3 criterion. This seemed to confirm that, on one hand, the studied digestate cases were far more likely to qualify under the TOC/TN criterion than the $N_{min}$/TN one and, on the other hand, that the cases that complied with $N_{min}$/TN were also more likely to meet the TOC/TN criterion.

### 4.5. Dry Matter Content of RENURE Compliant Digestates

Looking at the DM content of the cases that were below or equal to TOC/TN ≤ 3, there was no apparent linear correlation between DM and TOC/TN ($R^2$ = 0.218). Nonetheless, it could be observed that the DM values dropped noticeably—with mean and median values of 38 and 35 ± 14 g kg$^{-1}$ FM, respectively—in comparison with the entries that were above TOC/TN ≤ 3, which displayed mean and median values of 65 and 62 ± 24, respectively. The same drop in DM content was observed for the $N_{min}$/TN criterion, for which mean and median values of digestate entries above or equal to 90% $N_{min}$/TN were 34 and 32 ± 13 g kg$^{-1}$ FM, respectively. The cases that met either $N_{min}$/TN ≥ 90% or the TOC/TN ≤ 3 returned mean and median DM values of 38 and 35 ± 14 g kg$^{-1}$ FM, respectively, against 57 and 53 ± 26 g kg$^{-1}$ FM, respectively, in database C (Table 2, left), i.e., a drop in DM content of over 30%. Thus, while it cannot be ruled out that some unseparated digestates successfully met the criteria, a median value of 3.5% DM would typically suggest that the digestate underwent some form of separation resulting in a LF and possibly an additional processing treatment (stripping, scrubbing). This trend is visible in the boxplots in Figure 3, where it can be observed that the datapoints with lower DM ranges are in general more successful in meeting either the TOC/TN or $N_{min}$/TN requirements.

### 4.6. Identified LF Cases and Associated Separation Technologies

There are 58 known cases (Table 5) in which the LF of digestate is mentioned explicitly, either from the experimental assays—for which background information on each process is known in detail—or the literature review—in which a technology was explicitly mentioned. Of these 58 entries, 26 contained data both on TOC/TN and $N_{min}$/TN, and 49 contained data on $N_{min}$/TN. The distribution of cases according to separation technology and RENURE criterion is presented in Table 5.
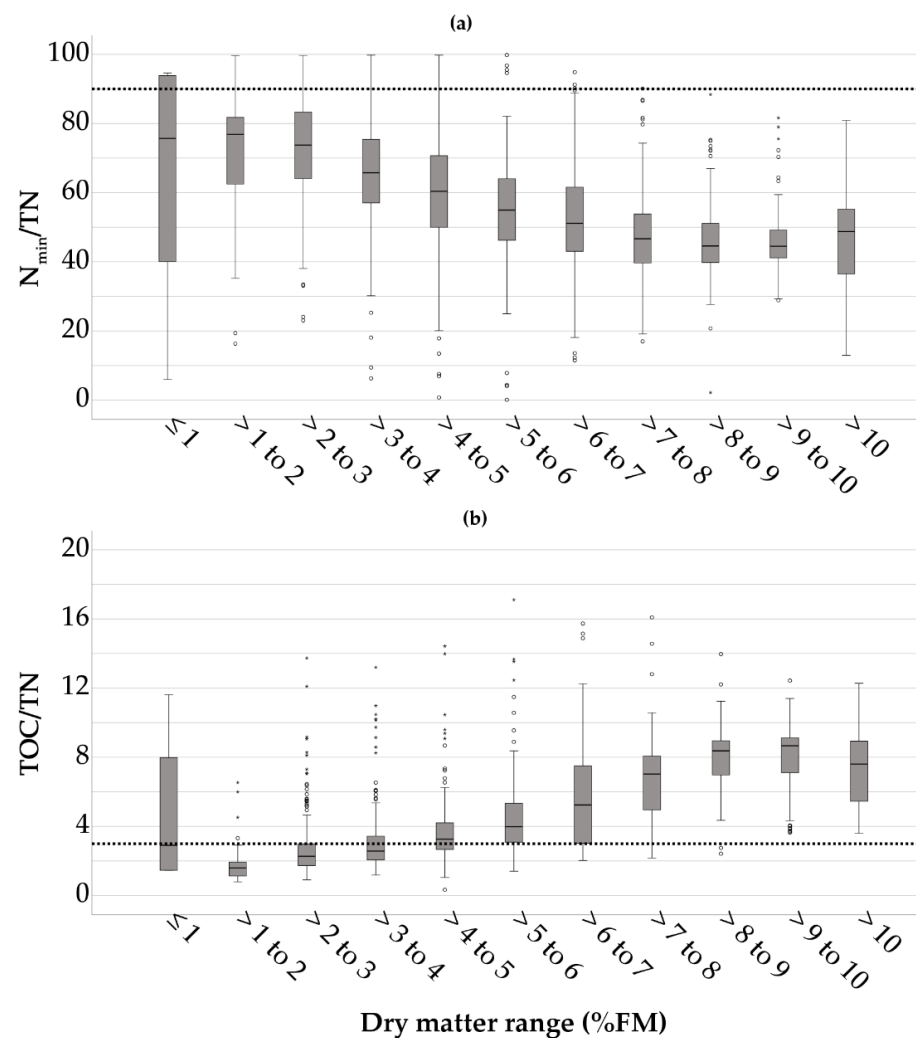
**Figure 3.** Box plot distribution of (**a**) $N_{min}/TN$ and (**b**) TOC/TN (excluding seven entries with TOC/TN > 20) by dry matter range. In both graphs, the dotted line marks the RENURE threshold.

**Table 5.** Overview of the number of identified LF entries in the database per separation technology and RENURE compliance. Here, TOC = total organic carbon; TN = total nitrogen; $N_{min}$ = mineral nitrogen.

| | | Separation Technology | | | | | |
|---|---|---|---|---|---|---|---|
| **Renure Criterion** | **Compliance** | **Reverse Osmosis** | **Screw Press** | **Decanter** | **Centrifuge** | **Belt Press** | **Unspecified** |
| $N_{min}/TN$ | Cases | 2/4 | 0/30 | 0/3 | 0/4 | 0/3 | 0/5 |
| | (%) | 50 | 0 | 0 | 0 | 0 | 0 |
| TOC/TN | Cases | - | 4/13 | 1/3 | 3/4 | 0/2 | 2/4 |
| | (%) | - | 31 | 33 | 75 | 0 | 50 |

For the TOC/TN criterion, 10 out of the 26 available cases (38%) displayed a value below or equal to 3. The associated separation technologies were screw press, centrifuge, decanter, and unspecified technology. The $N_{min}/TN \geq 90\%$ criterion appeared more discriminatory than the TOC/TN $\leq 3$ factor as only 2 of the 49 cases qualified (4%): both being mineral concentrates from the reverse osmosis of digested pig slurry. The technologies associated with the remaining 47 LF entries were screw press, centrifuge, belt press, decanter, and reverse osmosis. Of the 30 known cases that resorted to a screw press, none qualified under the $N_{min}/TN$ criterion. Moreover, of these 30 cases, 13 had corresponding data on TOC/TN, of which 4 met the criterion, seemingly showcasing the

greater tolerance of the TOC/TN criterion over $N_{min}$/TN, as was already pointed out in Table 4. Overall, out of 26 known LF cases that contained data on both RENURE criteria, 10 met one or the other requirement. These results are in the same order of magnitude as those reported in the RENURE document: 0% compliance for screw press against 13% in this study; 80% after centrifuge or enhanced solids removal against 75% in this study; 50% for LF against 38% in this study [12]. While it is outside of the scope of this study to pinpoint clear-cut favourite technologies, i.e., producing biofertilisers that would meet the proposed RENURE criteria, as highlighted in Table 5, not all mechanical separation technologies are made equal, as indeed, some have superior solids' removal efficiency than others [19]. These results (Table 5) agree with the RENURE study, which had already noted that further refining steps involving membrane separation (reverse osmosis, micro-, or ultrafiltration) and nutrient stripping would probably be a prerequisite to reach the necessary $N_{min}$ concentrations and/or remove sufficient OM.

### 4.7. RENURE Compliance of the Targeted LF in the Database

As mentioned in Section 2.1.1., two methods were employed to target the LF within the digestate database. In the first, DM ranges of LF reported in the literature were used [15,18,23]. These values, ranging from 14 to 66 g kg$^{-1}$ FM, were applied to the database as possibly representing LF of digestate. This resulted in 1324 entries containing data on TOC/TN or $N_{min}$/TN. Regarding TOC/TN, mean and median values dropped from 4.9 and 3.9, respectively, in database C (Table 2, left) to 3.8 and 3.0, respectively, showcasing a 50% compliance rate (Table 6, left). For $N_{min}$/TN, mean and median values increased from 58 and 57 (Table 2, left), respectively, to 63 and 62%, respectively, reaching a 7% compliance. Total compliance (both criteria together) was 43%. Incidentally, no notable differences in Cu and Zn concentrations or compliance were observed in the targeted LF population in comparison with the results reported in Table 2. For Cu, median was 70 ± 118 mg Cu kg$^{-1}$ DM (96% compliance) and, for Zn, median was 309 ± 261 mg Zn kg$^{-1}$ DM (96% compliance).

**Table 6.** RENURE compliance of targeted LF of digestate, based on DM range. Left: DM ranges associated with LF reported in literature; right: DM ranges from identified LF cases in the database (using median value as cut-off point). Here, TOC = total organic carbon; TN = total nitrogen; $N_{min}$ = mineral nitrogen; DM = dry matter.

| | (Left) DM Range: 14 to 66 g kg$^{-1}$ FM | | | (Right) DM Range: 1.5 to 44 g kg$^{-1}$ FM | | |
|---|---|---|---|---|---|---|
| | $N_{min}$/TN ≥ 90% | TOC/TN ≤ 3 | $N_{min}$/TN ≥ 90% or TOC/TN ≤ 3 | $N_{min}$/TN ≥ 90% | TOC/TN ≤ 3 | $N_{min}$/TN ≥ 90% or TOC/TN ≤ 3 |
| Compliance (cases) | 90/1291 | 548/1087 | 575/1324 | 76/745 | 424/658 | 444/770 |
| Compliance (%) | 7 | 52 | 43 | 10 | 64 | 58 |

In the second method, to try and further isolate the LF in the database, the median DM value of 44.9 g kg$^{-1}$ FM from the 58 known cases of LF in the database (reported in Table 5) was used as the cut-off point. This resulted in a targeted DM range between 1.4 (lowest value in database C) and 44.9 g kg$^{-1}$ FM (cut-off). Further lowering the DM range resulted in a higher RENURE compliance across all criteria, taken either separately or together. However, a similar gap between compliance with TOC/TN (64%) or $N_{min}$/TN (10%) was observed once more, while overall compliance (both criteria together) was the highest of all at 58%. Thus, it might be argued that narrowing the scope, based on lower DM ranges, was effective in targeting the LF in the database as RENURE compliance increased substantially with both methods explained above (Table 6).

### 4.8. Best Management Practices: Reaping Optimal Benefits and Minimising Environmental Risks

In the RENURE study, the proposed threshold values for $N_{min}$/TN and TOC/TN were based on the respective NFRV of the potential candidate RENURE fertilisers, where the highest observed NFRV values (82 and 83% mean, respectively) corresponded to the

90 to 100% $N_{min}$/TN pool and the 0 to 3 TOC/TN pool. The high correlation between the top tier NFRV values of candidate fertilisers and the proposed criteria of $N_{min}$/TN $\geq$ 90% and TOC/TN $\leq$ 3 meant that the door was left open for a candidate product to meet either one or the other, so as to allow more flexibility. In this study, the close scrutiny of digestate properties uncovered a trend in which the TOC/TN criterion appeared less restrictive than the $N_{min}$/TN criterion (Tables 4 and 6). As discussed above, a listwise comparison of both criteria returned a median value of $N_{min}$/TN of 67% for digestates that had met the TOC/TN $\leq$ 3 threshold. As a result, while available field-based evidence suggests that the use of digestate can lead to similar amounts of residual $NO_3^-$ in the soil and NFRV as that of chemical fertilisers [14], and in some cases even lead to reduced $NO_3^-$ leaching [75], the fact that considerably more digestates complied with TOC/TN than $N_{min}$/TN in this study (Tables 4 and 6) might warrant further research into the interaction between soil–plant systems in relation to both criteria for any given digestate product.

A higher presence of the plant-available $NH_4$-N form and a lower C/N ratio [52] have led to instances of superior short-term fertility of digestate over undigested feedstocks [76] and, in some cases, to similar biomass yields to those obtained with chemical fertilisers [34,77,78]. However, the optimal time of application remains a challenge. In particular, autumn application of digestate, when crop uptake has slowed to a crawl, has been shown to substantially increase N volatilisation and $NO_3^-$ leaching [79], both of which can be further exacerbated on sandy soils with low water retention capacity. Digestate fertilisation in NVZ takes place from late winter through to the end of summer, starting at the earliest in February when plant N-demand is still low and mineralisation is slow because of the low soil temperatures. Thus, a better grasp of the interaction between TOC/TN and $N_{min}$/TN for a specific soil–plant system might help in determining the optimal time of application (thereby increasing NUE and NFRV), where it might be argued that a combination of N fertiliser from both mineral (immediate availability) and organic forms (delayed availability) might be preferable for early spring crops; whereas for summer crops, the high mineralisation activity and ensuing higher NUE would mean that reliance on digestate with a higher organic load would cover the plant's demands [80] without jeopardising the environment. Another path worth exploring is the use of catch crops to effectively reduce nutrients' runoff and leaching. In this respect, evidence was provided that the ensiling and subsequent co-digestion of catch crops with manure was an economically viable way to increase biogas production [81], all the while improving nutrient recycling and bringing grist to the mill of the circular economy.

The compositional properties of digestate can vary substantially according to a.o. processing technology, retention time, organic loading rate, digestion temperature, composition (cellulose, hemicellulose, and lignin), and ratio of the (co)digested feedstocks [82]. The management system—from collection to storage—also affects the compositional value of slurries and manure, which contain varying amounts of urine, water, faeces, and spilt feed, and will also depend on the diet, the growth stage, and the species [19]. So, the resulting wide range of digestate properties combined with the further breakdown and release of its organically bound nutrients can result in unpredictable N dynamics in which the mineralised N exceeds crop demands, giving rise to N leaching [44,83]. Furthermore, the influence of soil texture (sandy, loamy, clayey) and SOM content was also shown to influence $NH_3$ volatilisation [84–86].

If we take all of the above into consideration, the variability of N kinetics from digestate makes a convincing case for the RENURE limits to be considered in tandem with best management practices adapted to the local context. These include, among others, improved synchronisation between N application and plant demands, catch and cover crops (where applicable), spatially optimised precision fertilisation technologies (such as infrared spectroscopy), appropriate product storage (closed tanks), low $NH_3$ emission application techniques (such as injection and immediate incorporation), and soil compaction prevention techniques (such as reduced tillage, low pressure tires, and drag hose systems).

## 5. Conclusions

The study showed that Cu and Zn from co-digested manure mostly met the RENURE criteria: 5.3% of the 1035 considered cases were above the 800 mg Zn kg$^{-1}$ DM limit; 5.0% of the 1038 available cases for Cu were above the 300 mg Cu kg$^{-1}$ DM limit. Moreover, as Cu and Zn are essential co-factors in the plant's metabolic pathways, these numbers would suggest that digestate application would thus constitute an added benefit for crop growth in terms of Cu and Zn availability, rather than an environmental liability. When applying the $N_{min}$/TN $\geq$ 90% criterion to the digestate database, just above 5% (96 of 1856 entries) met the condition; while 36% (574 out of 1583 entries) met the TOC/TN $\leq$ 3 criterion. Thus, the $N_{min}$/TN criterion appeared more stringent than the TOC/TN criterion. As expected, targeting the LF of digestate led to a higher compliance rate, between 43 and 58% depending on DM range, indicating that LF is a better suited RENURE product than unseparated digestate. Our findings were consistent with those of the JRC's RENURE study regarding some separation technologies being more efficient than others in terms of solids' removal and/or $N_{min}$ upcycling, thus increasing the likelihood of these technologies to provide RENURE-compliant fertilisers.

## Appendix A

**Table A1.** NH$_4$-N = ammonium nitrogen (N$_{min}$); DM = dry matter; OM = organic matter; TOC = total organic carbon; TN = total nitrogen; K$_2$O = potassium oxide; P$_2$O$_5$ = phosphorus pentoxide; Zn = zinc; Cu = copper; n = digestate sample size.

| Parameter | Unit | Mean $\pm$ Stdev | Median | n |
|---|---|---|---|---|
| DM | g kg$^{-1}$ FM | 55.96 $\pm$ 31.57 | 51 | 153 |
| OM | g kg$^{-1}$ DM | 661.13 $\pm$ 83.37 | 684 | 47 |
| TOC | g kg$^{-1}$ DM | 353.79 $\pm$ 66.21 | 369 | 68 |
| pH | - | 8.14 $\pm$ 0.49 | 8.2 | 87 |
| TN | g kg$^{-1}$ DM | 85.48 $\pm$ 47.59 | 75.81 | 153 |
| N$_{min}$ (NH$_4$-N) | g kg$^{-1}$ DM | 56.97 $\pm$ 39.03 | 459.6 | 137 |
| K$_2$O | g kg$^{-1}$ DM | 57.98 $\pm$ 33.17 | 48.65 | 56 |
| P$_2$O$_5$ | g kg$^{-1}$ DM | 37.72 $\pm$ 27.53 | 29.5 | 107 |
| Zn | mg kg$^{-1}$ DM | 863.83 $\pm$ 1039 | 440 | 22 |
| Cu | mg kg$^{-1}$ DM | 312.39 $\pm$ 469.08 | 137.55 | 22 |

**Table A2.** NH$_4$-N = ammonium nitrogen (N$_{min}$); DM = dry matter; OM = organic matter; TOC = total organic carbon; TN = total nitrogen; K$_2$O = potassium oxide; P$_2$O$_5$ = phosphorus pentoxide; Zn = zinc; Cu = copper; Hg = mercury; n = digestate sample size.

| Parameter | Unit | Mean ± Stdev | Median | n |
|---|---|---|---|---|
| DM | g kg$^{-1}$ FM | 76.38 ± 29.04 | 82.15 | 14 |
| OM | g kg$^{-1}$ DM | 628.66 ± 128.11 | 644.95 | 14 |
| TOC | g kg$^{-1}$ DM | 267.75 ± 46.90 | 273.6 | 12 |
| pH | - | 8.09 ± 0.80 | 8.35 | 14 |
| TN | g kg$^{-1}$ DM | 99.89 ± 40.31 | 85.6 | 14 |
| N$_{min}$ (NH$_4$-N) | g kg$^{-1}$ DM | 62.10 ± 31.75 | 50.4 | 14 |
| K$_2$O | g kg$^{-1}$ DM | 76.70 ± 54.53 | 69.1 | 6 |
| P$_2$O$_5$ | g kg$^{-1}$ DM | 42.93 ± 16.84 | 43.95 | 6 |
| Zn | mg kg$^{-1}$ DM | 648.25 ± 342.32 | 649.5 | 4 |
| Cu | mg kg$^{-1}$ DM | 170.17 ± 168.26 | 76.5 | 6 |
| Hg | mg kg$^{-1}$ DM | 0.67 ± 0.88 | 0.67 | 2 |

**Table A3.** NH$_4$-N = ammonium nitrogen (N$_{min}$); DM = dry matter; OM = organic matter; TOC = total organic carbon; TN = total nitrogen; K$_2$O = potassium oxide; P$_2$O$_5$ = phosphorus pentoxide; Zn = zinc; Cu = copper; Hg = mercury; n = digestate sample size. Some of the datapoints were averages from several digestate samples, several biogas plants, and in some cases averages over several years; the added value of these entries is thus higher than what the 'n' datapoints let on.

| Parameter | Unit | Mean ± Stdev | Median | n |
|---|---|---|---|---|
| DM | g kg$^{-1}$ FM | 55.44 ± 37.42 | 50.9 | 22 |
| OM | g kg$^{-1}$ DM | 645.32 ± 93.79 | 658.59 | 16 |
| TOC | g kg$^{-1}$ DM | 332.91 ± 127.57 | 307.02 | 6 |
| pH | - | 8.02 ± 0.38 | 8.01 | 19 |
| TN | g kg$^{-1}$ DM | 114.61 ± 69.42 | 98.5 | 11 |
| N$_{min}$ (NH$_4$-N) | g kg$^{-1}$ DM | 79.14 ± 45.83 | 45.83 | 18 |
| K$_2$O | g kg$^{-1}$ DM | 61.27 ± 29.35 | 55.42 | 9 |
| P$_2$O$_5$ | g kg$^{-1}$ DM | 45.81 ± 36.73 | 32.2 | 13 |
| Zn | mg kg$^{-1}$ DM | 562.31 ± 460.83 | 423.55 | 11 |
| Cu | mg kg$^{-1}$ DM | 107.39 ± 62.15 | 90 | 11 |
| Hg | mg kg$^{-1}$ DM | $4.00 \times 10^{-2} \pm 4.50 \times 10^{-2}$ | $3.00 \times 10^{-2}$ | 6 |

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