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# Current Technology on Nutrients Removal, Recovery and Reuse from Liquid Fraction of Digestate

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**Abstract**

By-products of anaerobic digestion, digestate is commonly managed via several ways for its optimal transportion and application. The common practice for digestate management is through solid-liquid separation. The common use for solid fraction of digestates are either through land spreading; directly applied or after composting, as organic fertilizer. Several routes proposed for valorization of solid digestate include production of biochar, bio-fuel for domestic furnaces, bioethanol production after centrifugal milling as well as post treatments (enzymatic, thermal and alkaline) for the recovery of methane. Liquid fraction of digestates contain high concentration of nutrients; from 1.5 to 6.5 g/L total nitrogen and from 0.94 to 2.51 g/L total phosphorus (P2O5) as well as high ions concentrations from 0.5 to 3.1 g/L ammonium (NH4+), from 1.05 to 5.48 g/L potassium (K+) and from 0-2.13 g/L phosphate (PO43-). Besides it also contains other ions such as sodium, chloride, magnesium, calcium and sulfate. High nutrients concentration limits its application to land with maximum application of 60 kg/ha/y of phosphate and 100kg/ha/y of potassium. Therefore, the removal of these nutrients is important before land application or disposal. In addition, these nutrients could be marketed to regions with high demand of nutrient or to the non-agricultural sector. The opportunities for nutrients marketing from digestate are largely unexploited and the strategies for marketing is still immature. This paper reviews the current technology on the removal, recovery as well as reuse of nutrients from liquid fraction of digestate. The discussion on the removal of nutrients include ammonia stripping, anaerobic ammonium oxidation (ANNAMOX), direct contact membrane distillation, constructed wetland system and vapor pressure membrane contactor. Nutrients recovery technology discussed in this paper include vacuum evaporation, struvite recovery, vacuum thermal stripping with acid absorption, combined evaporation and reverse osmosis. Meanwhile, the current technology on nutrients reuse include cultivation with microalgal for biomass production, nutrients recycling back to digester, soil application and subsurface injection into soils.

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## 1. Introduction

The by-products of anaerobic digestion, digestate is commonly managed via several ways for its optimal transportion and application. The best practices for further use of digestate include solid-liquid separation and covered storage [1]. However, the retained undigested organic matter contained in digestate could cause emission of its residual biogas during its storage or application to land which later can contribute to emission of greenhouse gases (GHG) as well as pollution to the atmosphere [2]. For an example, a study shows that the storage of digestate emits up to 12% methane from the total methane produced annually from a digester co-digesting dairy manure with food industry [1]. Besides, the land application of digestate can cause nitrous oxide ($N_2O$) and ammonia ($NH_3$) emissions to air as well as nitrate ($NO_3^-$) leaching losses to water [3]. In addition, digestates contain high concentration of COD, ammonium, phosphate, salinity and colour which causes high risk to the environment as well as all organisms if it is improperly managed or exposed to agriculture land [4].

Furthermore, the volume of digestate produced daily increased drastically in recent years which causes digestate overproduction for local and regional use [5]. The transportation of more than 5-10 km of the excess digestate will exceed its fertilizer value cost [5] and consumes huge amount of fuel oil [6]. In addition, due to 80-90% of digestate mass are mainly liquid [7], on-site digestate solid-liquid separation is commonly performed to minimize the costs of digestate transportation [8]. Screw press, centrifuge and screening drum press (vibrating screen) are the types of solid-liquid separation that are the most commonly applied at full-scale biogas plants [8], [9].

Common practice for solid fraction of digestates disposal is by land spreading [6], either through direct application or after composting, as organic fertilizer [10], [11]. Besides, solid fraction of digestate can also be dried either by belt dryer, drum dryer, fluidized bed dryer, feed-and turn dryer, solar drying system or palletized to be marketed as bio-fertilizers [7]. Previously, solid fractions of digestates received high interest in research and application due to its high content of organic fraction as well as high concentration of nutrients [7]. Other routes that have also been suggested for valorization of solid digestate [12] include as biochar production [13], [14], bio-fuel in domestic furnaces [15], bioethanol production after centrifugal milling [16] as well as post treatments (enzymatic, alkaline and thermal) for methane recovery [17].

Meanwhile, liquid fraction of digestates still contain high concentration of nutrients; from 1.5 to 6.5 g/L total nitrogen and from 0.94 to 2.51 g/L total phosphorus ($P_2O_5$) as well as high ions concentrations from 0.5 to 3.1 g/L ammonium ($NH_4^+$), from 1.05 to 5.48 g/L potassium ($K^+$), 0-2.13 g/L phosphate ($PO_4^{3-}$). Besides it also contains other ions such as sodium, chloride, magnesium, calcium and sulfate [18]–[21]. High ions concentration limits its application to land with maximum application of 60 kg/ha/y of phosphate and 100kg/ha/y of potassium [22]. Therefore, the removal of these nutrients is important before land application or disposal. In addition, these nutrients could be marketed to regions with high demand for nutrient or to the non-agricultural sector. The opportunities for nutrients marketing from digestate are largely unexploited and the strategies of marketing is still immature [23]. Therefore, this paper reviews the current technology proposed for the removal, recovery and reuse of nutrients in liquid fraction of digestate.

## 2. Removal of Nutrients from Liquid Fraction of Digestate

### Ammonia stripping

One of the most effective post-treatment for nutrients removal in liquid fraction of digestate is ammonia stripping [24]. It is a process where $NH_3$ in liquid sample is converted to gas when it comes in contact with the air or steam that contains few or no $NH_3$ [24]. The main factors affecting ammonia stripping process include temperature, pH, pressure and air/liquid ratio [24]–[26]. High level of pH influence the most in ammonia stripping process followed by air flow rate and temperature [25]. In a study, continuous ammonia stripping showed ammonia and total nitrogen removal up to 92.8% and 88.3%, respectively from liquid fraction of digestate originating from pig slurry [25]. In another study, coupling a food waste anaerobic reactor with side-stream ammonia stripping columns operated semi-continuously using biogas as stripping medium, had shown $NH_4^+$-N removal up to 48% at high temperature ≥ 70°C and high pH of 10 [27]. Bousek et al. studied side stream ammonia stripping of liquid fraction of digestate sieved at 1 mm originating from pig manure, pig fodder, sugar and maize silage [28]. A removal of 86% $NH_4$-N after 4 hours were observed after the effect of oxygen contact during air stripping. Another option, flue gas can also be selected to avoid anaerobic micro flora inhibition where $NH_4$-N removal of up to 45% was observed after 4 hours compared to $NH_4$-N removal of 16 % after 4 hours if biogas is used. This is due to the performance of stripping that is negatively correlated to the level of $CO_2$ in the strip gas [28]. Besides, the addition of $Ca(OH)_2$ could also enhance ammonia stripping. In a study, $Ca(OH)_2$ was added at optimal concentration of 12 g/L at pH > 7 has resulted in $NH_4^+$-N removal of up to 89.9% from liquid fraction of digestate which was originated from pig manure [29]. Besides, a removal of up to 97.2% of soluble phosphorus was also observed by addition of $Ca(OH)_2$ due to precipitation [29].

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**Anaerobic ammonium oxidation (ANAMMOX)**

Anaerobic ammonium oxidation (ANAMMOX) is a process under anoxic condition which uses Planctomycetes-like bacteria to oxidize ammonium ($NH_4^+$) to nitrogen gas ($N_2$) using inorganic carbon as an electron donor and nitrite ($NO_2^-$) as an electron acceptor [24], [30]. The factors which affects ANAMMOX process are organic carbon (organic molecules such as glucose, fructose, propionate, acetate and lactate), substrate composition (nitrite, ammonium and inorganic carbon), temperature, pH, phosphate ($PO_4^{3-}$), sulfide ($S^{2-}$), dissolved oxygen, and salinity [30]. Furthermore, these parameters have their concentration range limits as shown in Table 1 which has an effect to the efficiency of nitrogen removal.

Table 1: The concentration range limit of ANAMMOX process (adapted from Magri et al [30] and Sheets et al [24].

| Parameter | Concentration range |
|---|---|
| pH | 6.5 - 8.8 |
| Temperature (°C) | 35 - 40 |
| Dissolved oxygen (DO) | 1% |
| Salinity | 8800 – 30,000 |
| Ammonium ($NH_4^+$) (g/L) | 0.6 – 5.9 |
| Ammonia ($NH_3$) (g/L) | 0.002 – 0.15 |
| Nitrite ($NO_2^-$) (g/L) | 0.1 – 0.182 |
| Phosphate ($PO_4^{3-}$) (g/L) | 0.031 – 0.620 |
| Sulfide ($S^{2-}$) (g/L) | 0.001 – 0.064 |
| Inorganic carbon (g/L) | 0.15 – 0.295 |
| Organic carbon (g/L) | 0.142 – 0.242 |

Complete autotrophic nitrogen removal (ANR) is the combination of partial nitritation (PN) and ANAMMOX. It was suggested as the post-treatment for the removal of nitrogen from liquid fraction of digestate originating from livestock [30]. However, no full-scale ANR applied for liquid fraction of digestate originating from manure up to date [30]. The effectiveness and energy requirement of ANR depends on the liquid fraction of digestate composition [30]. In order to avoid inhibition by phosphates, $NH_3$ and sulfides, the important factor that needs to be taken into account is the dilution [24]. ANR is more competitive than other alternative methods due to lower specific energy requirement compared to traditional biological nitrogen removal, steam ammonia stripping/$(NH_4)_2SO_4$ absorption, air ammonia stripping/$(NH_4)_2SO_4$ absorption, struvite crystallization, concentration by vacuum evaporation and concentration by reverse osmosis particularly at concentrations of up to 2 kg $NH_4^+$-N $m^3$ [24], [30]. However, other methods can be more advantageous at higher concentrations [30]. The disadvantage of ANAMMOX is that heavy metals, phosphorus or antibiotics in liquid fraction of digestate can negatively affect the process of ANAMMOX. Therefore, phosphorus should be precipitated before the ANR stage [30].

**Direct contact membrane distillation process**

Membrane distillation is an integration of membrane separation with thermal distillation in order to combine the comparative advantages of both approaches [31]. In a study, direct contact membrane distillation process was applied to liquid digestate after lab-scale centrifugation originating from livestock wastewater. The treatment has successfully removed up to 99% for both COD and total phosphorus while the removal of total nitrogen was from 85 to 96% and it depends on the extent of cake layer formed on the membrane surface [32].

**Constructed wetland system**

A study of pilot hybrid constructed wetland by Maucieri et al [33] to evaluate the performance of combined subsurface flow line (SSL) with floating treatment wetland line (FTWL) has shown a promising results of organics and nutrients removal. The removal efficiency obtained in that study for total nitrogen, ammonium-nitrogen, nitrate-nitrogen, total phosphorus and phosphorus is 64.6%, 65.1%, 35.6%, 49.2% and 45.1%, respectively after treatment by SSL. Combination with FTWL has successfully removed 90%, 89%, 93.8%, 50.3% and 49.9% total nitrogen, ammonium-nitrogen, nitrate-nitrogen, total phosphorus and phosphorus, respectively. Liquid fraction of digestate used in that study was originated from slurry, corn silage and residues from agriculture, which was mechanically separated into solid and liquid fraction after anaerobic digestion process.

**Vapor pressure membrane contactor (VPMC)**

A study of the removal of ammonia from chicken manure digestate through vapor pressure membrane contactor (VPMC) via Polytetrafluoroethylene (PTFE) membrane was investigated [34]. In the study, 93.6% ammonia removal efficiency was achieved with a concentration of 231 mg N/L from 3644 mg N/L. Almost complete ammonia removal was achieved with an additional polishing step by phytoremediation via Lemna minor species.

## 3. Recovery of Nutrients from Liquid Fraction of Digestate

**Vacuum evaporation**

Vacuum evaporation is a process which consists in boiling a liquid sample at a lower temperature than boiling temperature at atmospheric conditions under negative pressure [35]. Vacuum evaporation of liquid fraction of digestate has been studied using 0.100 and 0.025$m^3$ pilot scale plants [35]. In this study, liquid

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fraction of digestate after screw press (originating from swine manure, corn silage and other biomasses) was used. Two-stage vacuum evaporation with acidification was observed to effectively concentrate 1688%, 1850% and 1527% of total solids (TS), volatile solids (VS) and total kjeldahl nitrogen (TKN), respectively. The two-stage system has successfully removed up to 94.4% of mass which contained 2.5% of the TKN mass which can either be used as dilution water for the input of digester or discharged into surface water after purification [35]. The reduction of pH to 5 is important to prevent from ammonia vaporization to form condensate and therefore, remained in the solid concentrate [35]. In another study of vacuum evaporation of liquid fraction of digestate originating from pig manure has shown that at optimum pH 6, there were only 114% and 224.8% increase in $NH_4^+$-N and soluble phosphorus concentrations respectively [29]. In a study performed by Vondra et al [36], three different types of industrial evaporators were evaluated and compared for their energy performance for liquid fraction of digestate thickening. It was observed that the multi-stage flash evaporator was the most efficient evaporator where it requires the least heat transfer area in terms of consumption of energy and cooling duty compared to the forced-circulation evaporator and the falling-film evaporator.

### Struvite recovery

Precipitation via struvite formation (combination of ammonium, magnesium and phosphate ions ($MgNH_4PO_46H_2O$)) is a promising method for the recovery of high concentrations of ammonium, magnesium and phosphate from liquid fraction of digestate due to the method that is simple but high efficiency and environmental friendly [37]–[39]. Struvite precipitation processes can be effected by the source of $Mg^{2+}$, $PO_4^{3-}$, effluent solid content, pH and $Mg:NH_4:PO_4$ molar ratio [37], [39]. However, few parameters that limits to efficiency of struvite process include high $Ca^{2+}$ concentration, high suspended solids concentration, high alkalinity, high ionic strength and complex chemical composition [39]. Liquid fraction of digestate originating from manure which is rich in nitrogen, ammonium and orthophosphates can give high market potential for the recovery of struvite as well as ammonia ($(NH_4)_2SO_4$) [39]. For an example, Tampio et al. studied combined ammonia stripping with $H_2SO_4$ scrubbing to recover ammonium sulfate $(NH_4)_2SO_4$ which is a chemically pure product with no TS, VS, phosphorus and potassium [40]. The same author also combined studied combined ammonia stripping (with $H_2SO_4$ scrubbing) with reverse osmosis and succeeded to obtain $(NH_4)_2SO_4$ and remove TS, VS, total nitrogen, $NH_4$-N, potassium and phosphorus from liquid fraction of digestate [40]. Besides, precipitation of struvite can also be used for the removal of ammonium from liquid fraction of digestate. A study obtained a removal of up to 95% from initial $NH_4^+$ concentration of 2.5 g/L in only 30 seconds after the addition of $Na_3PO_4 \cdot 12H_2O$ and $MgCl_2 \cdot 6H_2O$ with molar ratio of 1:1:1 for $Mg:NH_4:PO_4$ without an adjustment of pH[37].

### Vacuum thermal stripping with acid absorption

Vacuum thermal stripping with acid absorption allows the recovery of ammonia at higher flow rate in a recirculation line of a mesophilic anaerobic digester compared to the thermal stripping with higher temperature [41]. Ammonia is stripped out and later absorbed to a sulfuric acid solution which later forms ammonium sulfate crystal which has a high market value. At an optimum pressure of 25.1 kPa and boiling point of 65°C, more than 95% of ammonia could be stripped out from liquid digestate in one and half hour [41].

### Combined evaporation and reverse osmosis

A combination of evaporation and reverse osmosis was studied by Tampio et al for the recovery of nutrients from liquid fraction of digestate. In the study, up to 99.7, 99.1, 100 and 100% of total nitrogen, $NH_4^+$-N, phosphorus and potassium, respectively were successfully recovered [40]. The same author also combined stripping before evaporation and reverse osmosis and obtained 100% recovery of total nitrogen, $NH_4^+$-N, phosphorus and potassium [40].

## 4. Nutrients Reuse from Liquid Fraction of Digestate

### Cultivation

In a review by Koutra et al and Stiles et al [42], [43], microalgal biomass cultivated in liquid fraction of digestate could be used to produce biofuels products such as biogas, biodiesel and bioethanol. Besides, added-value products such biofertilizers, bioplastics, forage and feed supplements as well pharmaceuticals products can also be produced from microalgal biomass. For example, in a study of microalgae cultivation, Chlorella sp. was cultivated in the liquid fraction of digestate originating from chicken manure after treated with membrane ultra filtration [44]. The study showed great potential of biocrude oil production via hydrothermal liquefaction from cultivated Chlorella sp. In another study, Chlorella 1067 was cultivated in chicken manure based liquid digestate after ultra filtration treatment in a 400 L open raceway pond [45]. The cultivated Chlorella 1067 biomass after separation was co-digested with chicken manure for biogas production and obtained 239 mL $CH_4$/g VS fed. Other cultivation study investigate two freshwater microalgae (Botryococcus braunii and Tetradesmus obliquus), a photosynthetic cyanobacterium (Arthrospira maxima) and a marine diatom (Phaeodactylum tricornutum) for their ability to grow on three different liquid digestates originating from zootechnical, vegetable biomass and organic fraction of municipal solid waste, respectively [46]. It was observed that cultivation in liquid digestate from vegetable biomass

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showed the same growth as respective standard media. Cultivation in liquid digestate from zootechnical was only efficient for growth of Tetradesmus obliquus and Botryococcus braunii while cultivation in liquid digestate originating from organic fraction of municipal solid waste (OFMSW) had the poorest growth medium for all the strains. The study also observed that Tetradesmus obliquus and Arthrospira maxima were the best for Ammonium-Nitrogen removal from liquid digestate with 98.9 to 99.8% removal compared to 88.5% and 79.0% removal for Botryococcus braunii and Phaeodactylum tricornutum, respectively [46]. Similar study also investigated microalgae cultivation in an agro-zootechnical ultrafiltered digestate [47]. In that study, Chlorella sp. and Phaeodactylum tricornutum were able to grow and their growth in ultrafiltered digestate were similar to those obtained using synthetic media. It was also observed in that study that Chlorella sp. and Phaeodactylum have successfully removed 92% and 71%, respectively nitrogen in filtered digestate.

Tao et al [48] studied cultivation of Scenedesmus acuminatus in a liquid fraction of digestate originating from mesophilic and thermophilic anaerobic digesters treating pulp and paper industry biosludge with and without thermal pretreatment. It was observed that higher Scenedesmus acuminatus biomass yields were obtained in thermophilic digestates (without and with pretreatment prior to anaerobic digestion: 10.2 ± 2.2 and 10.8 ± 1.2 g $L^{-1}$, respectively) compared to the ones in pretreated mesophilic digestates (7.8 ± 0.3 g $L^{-1}$). This was likely due to the differences in iron, sulfate, and/or other minor nutrients concentration. Cultivation of Scenedesmus acuminatus removed 99.9% of phosphate and sulfate and over 97.4% ammonium from the digestates. In addition, the soluble COD and colour of the digestates were reduced up to from 29 to 39% and from 74 to 80%, respectively. In that study, it was concluded that different anaerobic digestion processes resulted in different methane yields, digestate compositions, and microalgal yields [48]. Another study by Fran chino [49] used diluted digestate originating from pig slurry and corn for cultivation of green algae Chlorella vulgaris. The result obtained was very promising with a removal of 90% total nitrogen, ammonia and phosphate. Beside microalgae cultivation, other study used liquid fraction of digestate originating from biomass (Syzygium cumini, Tectona Grandis and Ficus aurea leaves) which was filtered at 50 μm and autoclaved for nutrient supplement and maintaining moisture content for the cultivation of mushroom (Pleurotus florida) [50]. The study had shown to increase 66 to 100% yield of mushroom growth. An increase of 20% N supply was observed to increase 40% of mushroom yields [50]. Similar study investigated the possibility of cultivation of hydroponic baby leaf lettuce (Lactuca sativa L.) in liquid fraction of digestate combined with agriperlite [51]. The results showed that liquid fraction of digestate could be used as a sustainable and alternative growing media as well as nutrient solution for the cultivation in hydroponic system.

### Recycling nutrients back to digester

Recycling liquid fraction of digestate which contains high nutrients into a low nitrogen mono-digestion of straw has shown to increase methane production in a reactor. For an example, supplementary of co-digestion with sewage sludge or addition of macronutrient (N and P), it was observed to increase process performance with higher methane production with low VFAs accumulation and stable pH [52]. Li et al [53] studied recirculation of liquid fraction of the digestate from the second-stage reactor into the first-stage reactor in completely stirred tank reactors (CSTRs) connected in series for corn stover anaerobic digestion. It was observed that liquid fraction of digestate recirculation increased 2.3% and 10.8% methane and biogas production, respectively. In addition, recirculation of liquid fraction of digestate increased alkalinity and pH as well as decreased volatile fatty acids (VFAs) concentrations and the ratio of VFAs to alkalinity which indicates a significant increase in the stability of the anaerobic digestion system [53].

### Soil application

A three year field experiment was performed by Sigurnjak et al [54] to evaluate the impact of liquid digestate as a partial substitute for synthetic N fertilizer. Liquid digestate originating from mixture of pig manure, organic waste from food industry and energy maize was obtained via a sieve band press separator. In the study, it was observed that there was no significant difference in crop yield and soil quality when synthetic N fertilizer was substituted by liquid digestate indicating the efficiency of liquid digestate as soil fertilizer.

### Subsurface injection into soils

A study by Riva et al and Orzi et al [55], [56] on the injection of liquid fraction of digestate into the soils has successfully reduced odours and ammonia volatilization into the air and preserving fertilizer value compared to the use of untreated biomass such as cow manure and pig slurries. In the study, the injection of liquid fraction of digestate into the soils was observed to be correct method compared to surface application; making it the best substitute for urea without reducing crop yields.

## 5. Conclusions

The common practice for digestate management is through solid-liquid separation. Whilst the solid fraction of digestates are commonly applied for land spreading, liquid fraction of digestates still contain high concentration of nutrients. High nutrients concentration limits land application of 60 kg/ha/y of phosphate and 100kg/ha/y of potassium. Therefore, the removal of these nutrients is important before land application or disposal. Moreover, these nutrients could be marketed to regions

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with a high demand for nutrients or to the non-agricultural sector. The opportunities for nutrients marketing from digestate are not largely exploited and the strategies of marketing is still immature. The current technologies on nutrients removal reviewed in this paper include ammonia stripping, anaerobic ammonium oxidation (ANNAMOX), direct contact membrane distillation, constructed wetland system and vapor pressure membrane contactor. Nutrients recovery technology discussed in this paper include vacuum evaporation, struvite recovery, vacuum thermal stripping with acid absorption, combined evaporation and reverse osmosis. Meanwhile, the current technology on nutrients reuse include cultivation with microalgal for biomass production, nutrients recycling back to digester, soil application and subsurface injection into soils. The summary of the post-treatments for nutrients removal, recovery and reuse are shown in Figure 1.

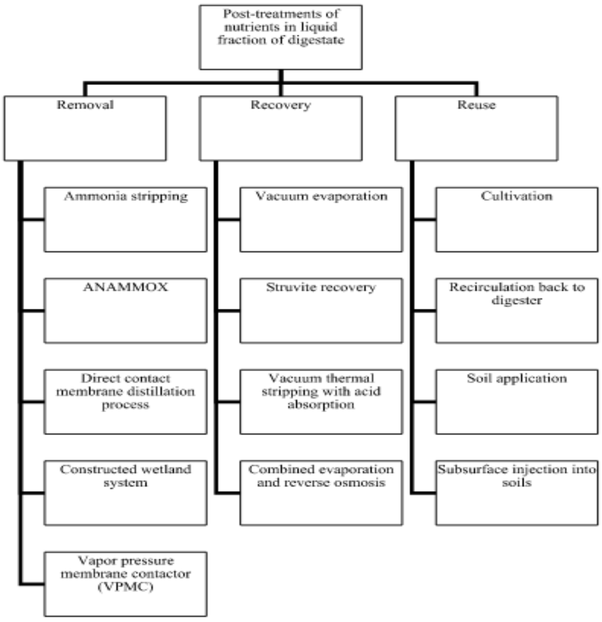


Figure 1: Types of post-treatments for nutrients removal, recovery and reuse from liquid fraction of digestates

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