

# EVOLUTION OF DAIRY MANURE IN A DUNGPIT WITH REGARD TO SOLID-LIQUID SEPARATION

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## 1 INTRODUCTION

Solid-liquid separation is a convenient pre-treatment that allows obtaining a manageable solid fraction rich in nutrients and a liquid fraction more easily treatable by anaerobic digestion. Powers et al (1995) found that in dairy manure 60% of DM, 86% of TKN and 94% of Total Phosphorous (TP) were contained in particles that passed through a 0.5 mm mesh sieve. Knowing the evolution with time of the organic matter and nutrients distribution regarding particle size would allow selecting the moment in time and the technology to employ for the separation of liquid and solid fractions.

The objective of the present work was to determine the evolution with storage time of the distribution of components in the solid and liquid separated fractions by physical-chemical processes.

## 2 MATERIALS: STORAGE OF MANURES

Raw manure (RM) was taken at the end of January in the cow house of a 500 cow dairy farm in Loreda (Cantabria). Four similar samples of 20 kg of RM were introduced in four 25 litre vessels. Storage period comprehended from the 30<sup>th</sup> of January, the day that samples were taken, to the middle of June when the sample 4.5M was separated. During storage, samples were exposed to temperature changes, with minimum and maximum values of 5°C and 25°C during this period of time. The days 0, 45, 90 and 135 samples in vessels 0M, 1.5M, 3M and 4.5M were separated into liquid and solid fractions. The first separation operation consisted in a static horizontal laboratory 0.8 mm mesh sieve which permitted obtain a screened solid fraction (SSF) and a screened liquid fraction (SLF). SLF was subsequently separated by means of a coagulation-flocculation process, employing a dose of 2% TS of solutions of FeCl<sub>3</sub> (50 g/l) and Fennopol K504 (1 g/l). Chemicals were mixed with the dairy cattle manure using two 1000 ml beakers, by the pouring method. When the optimal flocculation was achieved, by means of centrifugation liquid (CFSLF) and solid (CFSSF) flocculated fractions were separated. For each of the storage times TS, VS, TKN, TP and volatile fatty acids (VFA) were determined and their distributions in the separated fractions.

## 3 METHODS

### 3.1 Analytical methods

Volatile fatty acids (VFA) were determined using a HP 6890 GC equipment fitted with a 2m x 1/8 in. glass column, liquid phase 10% AT 1000, packed with the solid support Chromosorb W-AW 80/100 mesh. Gas composition was assayed on a 2m Poropak T column in a HP 6890 GC System with helium as carrier gas and TCD detector. All other analyses were performed following the Standard Methods for the Analysis of Waters and Wastewaters.

### 3.2 Calculations

To determine the percentage of mass or components in SSF from RM screening, the calculation was done from RM content. For the fractions obtained by flocculation-centrifugation and the global, the calculations were done taking as reference the mass and composition of the obtained fractions.

$$\% X_{CFSSF} = \frac{M_{CFSSF} X_{CFSSF}}{M_{SSF} X_{SSF} + M_{CFSSF} X_{CFSSF} + M_{CFSLF} X_{CFSLF}}$$

## 4 RESULTS AND DISCUSSION

### 4.1 Chemical composition of raw manure, solid and liquid fractions, evolution with age.

Since storage tanks were lab scale, the vessels were easily homogenised and samples presented almost constant composition, except from sample 4.5M whose VS and VFA concentration had diminished indicating the starting up of the methanization.

TABLE 1 Mass distribution and composition of RM and separated fractions

	ASSAY	Mass (g)	DM (g/kg)	VS (g/kg)	N-TKN (g/kg)	Pt (g/kg)	COD <sub>VFA</sub> (mg/kg)
RM	0 M	1000	118.4	96.51	4.770	1.094	4590
	1.5 M	1000	116.3	91.12	4.881	1.073	9112
	3 M	1000	118.5	95.37	4.817	1.094	10301
	4.5 M	1000	107.4	79.36	5.080	0.907	7780
SSF	0 M	354	154.7	115.04	5.286	1.332	
	1.5 M	302	186.6	160.8	6.005	1.662	
	3 M	343	175.9	162.8	4.928	1.506	
	4.5 M	353	160.3	129.8	5.524	1.060	
SLF	0 M	646	98.37	59.97	4.727	0.906	7105
	1.5 M	698	84.56	61.97	4.438	0.728	13047
	3 M	652	89.82	62.42	4.721	0.857	15677
	4.5 M	647	77.76	54.31	4.730	0.678	12019
CFSSF	0 M	334	132.1	77.14	5.340	1.450	
	1.5 M	454	96.62	73.70	4.153	0.887	
	3 M	392	111.8	78.71	4.463	0.947	
	4.5 M	400	96.94	72.19	4.800	1.060	
CFSLF	0 M	1302	14.93	9.969	0.976	0.062	2472
	1.5 M	1448	10.51	6.660	0.987	0.037	6547
	3 M	1311	11.26	6.550	0.947	0.014	7858
	4.5 M	1171	10.84	6.706	1.175	0.004	6641

Table 1 shows the results of the separation process, mass distribution and concentration of DM, VS, TKN and TP of each of the samples obtained from 1 kg of raw manure for the four assays carried out.

It must be taken into account that in the first fraction DM content passed from 10-12% in RM to values between 15-19% for SSF, which means that it still had an important moisture content and may buckle the results of particle size distribution in Table 2. The same can be said for the CFSSF with a DM between 9.5-11.5%.

In Table 1 it can be observed that from 1000 g of RM the mass percentage of SLF obtained ranged between 64-70%. Screening operation resulted in an increase of DM content of SSF respect RM between 1.3 and 1.6 and between 1.4 and 1.7 in VS. This bigger increment in the ratio of VS respect DM indicates that SSF retains a higher percentage of VS than fixed solids and then the SLF will be more mineralized. The increases in DM concentration for SSF were lower than the values referred by Møller *et al.* (2002) for RM with 55 g DM/kg. Similar increase values to which Møller *et al.* (2002) were obtained when the separation was done with an industrial screw-press separator with raw manure taken from the dung pit of the farm.

The objective of the coagulation-flocculation process is to remove the smaller size solids that can not be separated nor by screening neither by centrifugation. In flocculation followed by centrifugation the ratio of the concentration of DM of CFSSF with respect to SLF, the starting fraction was comprehended between 1.1 and 1.3, whereas VS content ranged between 1.2 and 1.4. This result corroborates the mineralization of CFSLF respect the original SLF in flocculation process. From Table 1 it can be deduced the percentage of VS in DM of RM and the

rest of fractions. The solid fraction increased the percentage of VS respect RM whereas that value decreased in the separated liquid fraction. The percentage of VS ranged from 58% in CFSLF 3M to 93% SSF 3M. In this sample the VFA content was the highest of the four samples which means that in that sample the losses of volatile matter during DM and VS determination by drying was the highest (Derikx *et al.*, 1994). The percentage of VS resulted in the diminishing following order SSF, RM, CFSSF, SLF y CFSLF.

After screening operation TKN concentration of SSF respect to RM was comprehended between 1.0-1.2 and between 0.91-1.0 for the liquid fraction. These results indicate that TKN is distributed almost equally between particles bigger and smaller than 0.8 mm, being in accordance with the results reported by Møller *et al.* (2002). In the fraction CFSSF, the concentration of TKN respect SLF was comprehended between 0.91-1.1 and for CFSLF that ratio was 0.20-0.25. Coagulation-flocculation followed by centrifugation allowed to obtain a CFSSF whose TKN was four times higher than that of the CFSLF. Møller *et al.* (2002) reported a mean value of 2.4, which indicates that coagulation-flocculation transfers to the solid fraction a part of the nitrogen in colloidal form that could not be separated only by centrifugation. It can be stated that anaerobic digestion of CFSLF will have less ammonia inhibition problems since its concentration is 4.4 times lower than that in RM.

Regarding the distribution of TP after screening, the concentration in SSF was 1.3-1.6 higher than that in RM, whereas in the SLF this ratio ranged between 0.72-0.83. The separation of the SLF by coagulation-flocculation and subsequent centrifugation gave a CFSSF with a TP concentration 1.4-1.7 times higher than that of the SLF. For the CFSLF this ratio varied from 0.005 to 0.07, diminishing as storage time increased. The TP concentration ratio in CFSSF/CFSLF was 24 for the sample 0M and 300 for the sample 4.5M. Møller *et al.* (2002) also reported this increase with storage time obtaining the following values: 13 and 25 for 2 and 16 weeks in storage channels respectively. The higher transference of TP to the CFSSF is due to the addition as coagulant of  $\text{FeCl}_3$  that precipitates dissolved phosphates as  $\text{FePO}_4$ .

TABLE 2      **Distribution of VFA in CFSLF**

(mg/L)	0M	1.5M	3M	4.5M
AcH	1564	3511	3775	3236
PrH	295	841	1017	830
IBuH	0	149	195	189
BuH	172	546	843	661
IVaH	12	80	111	119
VaH	11	66	92	74
DQO <sub>AGV</sub>	2472	6574	7858	6641

Table 2 shows the composition of the VFA of the CFSLF for each sample. VFA concentrations increased from sample 0M to 3M, decreasing in the sample 4.5M. The majority VFA were in this order acetic, propionic and butyric. The highest increase in VFA concentrations occurred when passing from sample 0M to sample 1.5M, 2,66 times higher.

VFA concentrations were not too high may be due to two factors. The first, because manure was stored in open tanks at ambient temperature, allowing the oxidation of the VFA located in the upper part of the vessels.

There again, the maximum and minimum temperatures during storage period comprehended between the 30<sup>th</sup> of January when samples were taken and the middle of June when sample 4.5M was separated were 12-5°C; 19-6°C and 25-7°C for samples 1.5M, 3M, 4.5M, respectively. VFA concentration increased until sample 3M. The sample 4.5M had started the methanization stage, reaching a pH of 7.2, decreasing VFA concentration.

#### 4.2      **Components transference efficiency on solid-liquid separation processes. Evolution with storage time**

In Table 1 the mass of each of the obtained fractions after the separation processes are represented. Starting from 1000 g of RM, the mass of the SLF varied between 65-70% of the initial mass in RM. In coagulation-flocculation centrifugation, the mass of the CFSLF ranged between 75-80% of the total mass involved in the process (CFSSF+CFSLF). The mass of the FSF varied between 34-39%.

TABLE 3 Distribution of components in the different separated fractions

	SAMPLE	DM (%)	VS (%)	TKN (%)	TP (%)
SSF $\phi > 0.8\text{mm}$	0 M	46.3	49.4	38.0	45.5
	1.5 M	48.8	52.8	36.9	49.0
	3 M	50.7	58.6	36.1	48.3
	4.5 M	52.3	55.5	37.1	53.0
CFSSF $0.8\text{mm} > \phi > 25\mu\text{m}$	0 M	37.3	37.0	36.2	46.7
	1.5 M	38.0	36.6	37.2	45.0
	3 M	36.9	32.4	37.4	50.0
	4.5 M	35.9	35.0	36.6	46.5
CFSLF $\phi < 25\mu\text{m}$	0 M	16.4	13.6	25.8	7.8
	1.5 M	13.2	10.6	26.0	6.1
	3 M	12.4	9.0	26.5	1.7
	4.5 M	11.8	9.5	26.2	0.5

In Table 3 the percentages of components retained in three fractions regarding particle size are represented. The percentage of DM separated by screening increased with storage time from 46.3% in sample 0M to 52.7% for sample 4.5M. For fractions CFSSF and CFSLF the DM content also increased with storage time from 69.4 to 75.3 %. From results in Table 3 it can be deduced that the percentage of intermediate size particles diminished softly with time. That means that the flow of particles that enter into the size  $0.8\text{mm} > \phi > 25\mu\text{m}$  is compensated with those that pass to a lower size ( $\phi < 25\mu\text{m}$ ). The DM content of the smallest particles decreased clearly with storage time. The DM content of the final solid fraction (FSF=SSF+CFSSF) was 83.6% in sample 0M and increased with time up to 88.2% for sample 4.5M. With regard to the percentage of VS transferred to the solid fractions, their values were similar to those found for DM. The retained percentage in the FSF was 86.4% for sample 0M and increased with storage time, reaching the 91% for sample 4.5M.

The percentage of TKN in the SSF ranged between 35.1-39.2%, which indicates that TKN is for the most part in particles smaller than 0.8 mm. By coagulation-flocculation and centrifugation, the CFSLF contained a percentage of TKN between 41.1-41.7% of that present in CFSSF and CFSLF. Now TKN is in a major proportion in the fraction CFSSF, in particles with a size  $0.8\text{mm} > \phi > 25\mu\text{m}$ . In the SSF the percentage of TKN retained ranged from 73.5-74.2%. Since CFSLF only contains 25.8-26.5% of the original TKN present in manure, then after an anaerobic digestion process it would only be necessary a surface four times smaller than raw manure to be applied to the land according to the normative (170 Kg TKN/ha/year; Board(Directive) 91/676/CEE, RD 261/1996).

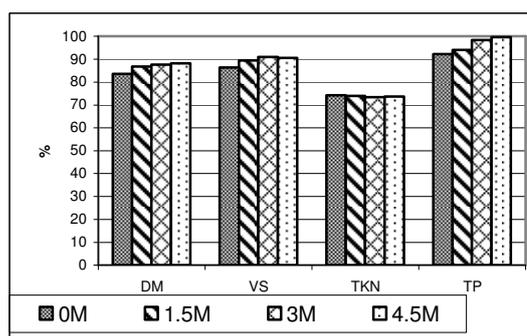


FIGURE 1 % of DM, VS, TKN and TP in the FSF

Regarding TP, after screening the SSF retained 44.4-53.2% of the TP present in RM. This value increased with storage time. In the CFSSF obtained by the physical-chemical process the percentage of TP transferred to the solid fraction increased with storage time and supposed a value between 85.8 and 99.0 of the TP present in both separated fractions. As mentioned before,  $\text{Fe}^{3+}$  has an important role in the efficiency of TP separated. In the FSF, the percentage of TP separated increased from 92.2% in sample 0M to 99.5% in sample 4.5M.

— In Figure 1 are shown the evolution of percentages of DM, VS, TKN and TP which were transferred to the FSF for all samples.

## **5 CONCLUSIONS**

Separation of dairy manure in solid and liquid fractions by means of mechanical and physicochemical procedures allows obtaining a FSF to which it has been transferred up to 88, 90, 74 and 99 % of DM, VS, TKN and TP, respectively of the content of the pre-treated manure. With increasing manure storage time, the content of all the components transferred to the FSF increased.

## **ACKNOWLEDGEMENTS**

The authors would like to thank the Council Environment of Cantabria (Spain) for financially supporting this research.

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