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Solids and Plant Nutrient Content, and Settling Characteristics of Milking Centre Wastewater on a Grazing Dairy

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Large milking centre wastewater samples were collected from a storage pit on a grazing dairy farm in South Carolina on two different days. The samples were analysed to determine the concentrations of the following constituents: TS, VS, TKN, TAN (NH_4^+-N, + NH_3-N), Org-N, NO_3-N, total-P, total-K, Ca, Mg, S, Zn, Cu, Mn, and Na. The large amount of waste milk present on the second day (TS =7165 mg/L) resulted in significantly reduced solids and plant nutrient concentrations as compared to the sample collected on the first day (TS = 17,024 mg/L). Gravity settling and thickening experiments were performed for the two milking centre waste samples. Gravity settling was effective at removing a fraction of all of the defined constituents. The greatest mass removals were for TS, VS, Org-N, P_2O_5, S, Cu, and Zn. The mass removal efficiency for the soluble constituents, TAN, K_2O, and Na, was a consequence of the liquid contained in the layer of settled material and not a result of a change in concentration. The data provided in this study can be used for design of primary treatment for milking centre wastewater, development of nutrient management plans, and to provide loading data for secondary treatment of the supernatant or settled solids.

1. Introduction

On dairy farms that use barns to house cows the milking centre wastes are often included with manure from the animal housing areas for treatment, storage, and land application. On a grazing dairy, animals are kept on pastures and milking centre wastewater is often handled independent of other manure generated on the farm. Gravity settling can be used to concentrate solids and plant nutrients into the settled fraction to facilitate use of a portion of the N, P, and K as fertilizer on fields remote from the dairy. The liquid fraction can then be used to fertilize pastures close to the milking centre.

Very little data are available on the solids, and plant nutrients contained in milking centre wastewater, and little information is available concerning the performance of gravity settling that is sufficient to assist in settling basin design. Wright and Graves (1998) provided information that indicates that the TS concentration of milking centre waste can range from 6,000 to 15,000 mg/L with major plant nutrient concentrations of 899 mg N/L, 99 mg P/L and 399 mg K/L. No information was provided concerning the organic and soluble forms of nitrogen that are required to make reasonable estimates of plant available N. In addition, no data were provided concerning the concentrations of important minor plant nutrients such as S, Ca, and Mg. Martínez-Suller, et al. (2010) monitored the N, P, and K concentrations in wastewater on a dairy farm in Ireland that included milking centre waste, milk spillage, and runoff from outdoor cattle yards. All of the wastewater was collected in a storage tank and a portion of the solids were removed by a three-stage sediment trap. Effluent from the third settling chamber was sampled over a five month period and the average concentrations were 351 mg N/L, 44 mg P/L, and 415 mg K/L. No data were provided concerning the major plant nutrient contents prior to sedimentation and no information was provided on settling effectiveness. Martínez-Suller, et al. (2010) also provided a review of other dairy wastewater studies conducted in Ireland, England, and Wales. However, none of these studies isolated milking centre wastewater from other sources of wastewater such as runoff from outside lots, and feeding areas.

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The objectives of the study were to: (1) determine the concentrations of solids and plant nutrients in milking centre wastewater on a grazing dairy, and (2) determine the settling characteristics of the milking centre wastewater samples.

2. Methods

The samples used in this study were collected on two different days from the milking centre waste storage pit on a dairy farm located in Orangeburg, County, South Carolina (USA). The milking herd numbered 200 Holstein cows kept on 24.3 ha of intensively grazed pasture except when provided supplemental feed on a concrete lot immediately after milking.

The milking centre consisted of a single building that contained a covered holding pen, a herringbone milking parlour, milk room, office, and other support areas. The parlour and holding area floors were cleaned with water from a high-pressure hose and by scraping. All of the wastewater from washing the milk pipelines, bulk tank, milk room floors, and parlor and holding area floors were collected and transferred to an uncovered, belowground, concrete holding pit located adjacent to the milking centre. Waste milk from cows treated with antibiotics was also transferred and stored in the holding pit. The holding pit had a capacity of 53,000 L. This volume did not include the required freeboard, and depth for net rainfall and the 25-year, 24-hour storm. The wastewater was agitated and emptied approximately once each week. The actual frequency of emptying depended on the number of cows being milked, the amount of rainfall that occurred, and the amount of waste milk generated from treated cows. Milking centre wastewater removed from the holding pit was applied on pasture or cropland using a tank-type manure spreader.

2.1 Sample Collection

Large manure samples were collected from the holding pit on two different days. On the first day, the holding pit was full with 7 days of milking centre wastewater. The entire contents of the holding pit were agitated prior to loading the manure spreader. Samples were collected during the beginning, middle, and end of the emptying and land application operations. The samples were combined in a 20 L container to yield a composite sample that was representative of the pit contents. On the second day, the holding pit was not completely full and the dairy producer indicated that waste milk from treated cows was disposed of in the pit. The pit contents were agitated and approximately 20 L of wastewater was removed for analysis of the composition and gravity settling characteristics. The 20 L samples were transported to Clemson University on the day they were collected, and were stored in large refrigerators prior to composition analysis and the gravity settling experiments.

2.2 Constituents Measured

Two well-mixed aliquots were taken from the milking centre wastewater samples collected on both days. The aliquots were analysed to determine the concentrations of the following constituents: total solids (TS), volatile solids (VS), total Kjeldahl nitrogen (TKN), total ammoniacal nitrogen (TAN = NH_4^+-N + NH_3-N), total phosphorus (expressed as P_2O_5), and total potassium (expressed as K_2O), calcium (Ca), magnesium (Mg), sulphur (S), zinc (Zn), copper (Cu), manganese (Mn), and sodium (Na) . Organic nitrogen (Org-N) was calculated as: Org-N = TKN - TAN. The Agricultural Service Laboratory at Clemson University provided plant nutrient analyses using standard methods (CUASL, 2017).

Measurements of TS, and VS, were obtained at Clemson University using standard oven drying and incineration methods (APHA, 1999). The samples were dried completely by holding them in a drying oven for 24 hours at a temperature of 105°C. The mass of TS was determined after the samples were allowed to cool in a desiccator. The mass of fixed solids (FS) was determined after incinerating the dried solids in a furnace maintained at 550 to 600°C for 1.5 to 2 hours, and allowing the samples to cool in a desiccator. The TS and FS concentrations were calculated by dividing the mass in mg by the sample volume in L. The VS concentration was the difference between TS and FS.

2.3 Settling Experiment

Experiments were performed in the laboratory to observe the settling characteristics of the two milking center wastewater samples. Graduated cylinders were used to facilitate the measurement of the settled material volumes with respect to time.

The gravity selling experiments were carried out using the following procedure: (1) a well-mixed sample of wastewater was extracted and poured into a graduated cylinder, (2) the initial volume of the sample was recorded, (3) the volume of the settled material was measured after 30 and 60 minutes of settling, and (4) the supernatant was decanted and well-mixed aliquots were analysed to measure the concentrations of the previously defined plant nutrients and solids.

In previous studies by the author (Chastain and Vanotti, 2003), the volume of the supernatant was observed to increase as settling time increased, but the constituent concentrations in the supernatant did not change significantly. Consequently, the supernatant concentrations at 30 min were assumed to be the same as at 60 min.

The volume of the settled material was normalized with respect to the initial volume poured into the graduated cylinder as follows:

$$SVF(t) = V_{SM}(t) / V_I. \tag{1}$$

Where,
$SVF(t)$= the settled volume fraction,
t = settling time (min),
$V_{SM}(t)$ = volume of the settled material (L), and
V_I = volume of wastewater poured into the cylinder (L).

A single settling experiment was performed for the sample collected on the first day using a 1000mL graduated cylinder. The wastewater sample collected on the second day was more dilute and it was anticipated that the volume of the settled material would be less than 100mL resulting in a relatively large error for the volume measurement. In order to reduce the error in volume measurement, the settling experiment for the second day was conducted using two 500mL graduated cylinders. This procedure provided supernatant samples for solids and plant nutrient analysis for wastewater samples collected on both days.

The equations used to describe the effectiveness of gravity settling were derived based on a mass balance for each of the defined constituents. Application of the law of conservation of mass on the graduated cylinder used for the settling experiments gave:

$$C_I V_I = C_{SUP} V_{SUP} + C_{SM} V_{SM}(t). \tag{2}$$

Where,
C_I = initial concentration of a constituent in the wastewater (mg/L),
C_{SUP} = concentration of a constituent in the supernatant (mg/L),
V_{SUP} = volume of the supernatant layer (L), and
C_{SM} = concentration of a constituent in the settled material (mg/L).

The mass balance for gravity settling was written on a unit volume basis by dividing through Eq(2) by V_I and applying the definition of the settled volume fraction, Eq(1), to yield:

$$C_I = C_{SUP} (1 - SVF(t)) + C_{SM} SVF(t). \tag{3}$$

The only quantity in Eq(3) that was not measured was C_{SM}. However, the concentration of a constituent in the settled material was calculated by rearranging Eq(3) as:

$$C_{SM} = [\, C_I - C_{SUP} (1 - SVF(t)) \,] / SVF(t). \tag{4}$$

The mass of any constituent, C, in the settled material was simply [$C_{SM}\ SVF(t)$]. Solving Eq(3) for [$C_{SM}\ SVF(t)$] indicates that the mass removal efficiency (MRE_C) of a constituent can be calculated from the data as:

$$MRE_C = 100 \times [\, C_I - C_{SUP} (1 - SVF(t)) \,] / C_I. \tag{5}$$

The concentration reduction of any constituent (CR_C) by gravity settling was calculated as:

$$CR_C = 100 \times [(C_I - C_{SUP}) / C_I]. \tag{6}$$

3. Results

The plant nutrient and solids content of milking centre wastewater was determined for samples collected on two different days. The average solids and plant nutrient concentrations are shown as the initial concentrations, C_I, in Tables 1 and 2. It is believed that the data shown for day 1 (Table 1) are more representative of the typical milking centre wastewater composition on this farm containing normal amounts of cleaning chemicals, milk, and manure. The sample collected on the second day (Table 2) also contained a large amount of waste milk from treated cows. The waste milk and extra water used to wash the waste milk into the holding pit resulted in a reduction in the TS content from 17,024 mg/L to 7165 mg/L. As would be expected, the concentrations of the volatile solids, and all major and minor plant nutrients were all reduced by the dilution provided by the disposal of waste milk. The only exception was the 33% increase in the Na concentration that was believed to be from the milk.

Table 1. Milking center wastewater composition and settling results for a typical day (Sample from first day)

Constituent	Initial C_I (mg/L)	Supernatant C_{SUP} (mg/L)	SD_C [1] (mg/L)	CR_C [2] (%)	Settling Time = 30 min $SVF(30)$=0.279 [3] MRE_C [4] (%)	Settling Time = 30 min C_{SM} (mg/L)	Settling Time = 60 min $SVF(60)$=0.254 [3] MRE_C [4] (%)	Settling Time = 60 min C_{SM} [5] (mg/L)
TS	17,024	8960	221	47	62	37,894	61	40,658
VS	13,373	6049	122	55	67	32,328	66	34,838
TAN	756	785	28	-4 NS	28 [6]	771 [7]	25 [6]	771 [7]
Org-N	452	167	19	63	73	1190	73	1288
TKN	1207	951	35	21	43	1869	41	1957
P_2O_5	402	296	16	26	47	676	45	713
K_2O	917	961	40	-5 NS	28 [6]	939 [7]	25 [6]	939 [7]
Ca	321	286	14	11	36	413	34	425
Mg	133	102	5.6	23	45	214	43	224
S	65	47	2.9	28	48	112	46	118
Zn	4.2	3.0	0.4	29	48	7.3	47	7.7
Cu	1.2	0.6	0.4	50	64	2.7	63	3.0
Mn	3.0	2.4	0.3	20	42	4.5	40	4.8
Na	161	167	8	-4 NS	28 [6]	164 [7]	25 [6]	164 [7]

[1] Pooled standard deviation using all concentration data
[2] Concentration Reduction calculated using Eq(6).
[3] Mean settled volume fraction for defined settling time.
[4] Mass removal efficiency calculated using Eq(5).
[5] Concentration of the settled solids calculated using Eq(4).
NS = Concentration reduction not significantly different from zero based on calculation of uncertainty using Eq(7).
[6] Actual mass removal efficiency if CR_C was not significantly different from zero = 100 x $SVF(t)$.
[7] Mean of initial and supernatant concentrations was used for C_I and C_{SUP}. in Eq(4).

Table 2. Milking center wastewater composition and settling results for the sample that contained large amounts of waste milk (Sample from second day)

Constituent	Initial C_I (mg/L)	Supernatant C_{SUP} (mg/L)	SD_C [1] (mg/L)	CR_C [2] (%)	Settling Time = 30 min $SVF(30)$= 0.105 [3] MRE_C [4] (%)	Settling Time = 30 min C_{SM} (mg/L)	Settling Time = 60 min $SVF(60)$= 0.093 [3] MRE_C [4] (%)	Settling Time = 60 min C_{SM} [5] (mg/L)
TS	7165	4645	221	35	42	28,554	41	31,800
VS	4621	2690	122	42	48	21,011	47	23,498
TAN	354	375	28	-6 NS	11 [6]	365 [7]	9 [6]	365 [7]
Org-N	252	153	19	39	46	1092	45	1220
TKN	607	528	35	13	22	1278	21	1379
P_2O_5	255	146	16	43	49	1180	48	1321
K_2O	508	533	40	-5 NS	11 [6]	521 [7]	9 [6]	521 [7]
Ca	237	195	14	18	26	593	25	648
Mg	77	48	5.6	38	44	323	43	361
S	39	25	2.9	36	43	158	42	176
Zn	2.1	1.2	0.4	50	55	10.5	55	11.8
Cu	0.4	0.2	0.4	50	55	2.1	55	2.4
Mn	1.7	1.1	0.3	50	55	10.5	55	11.8
Na	214	228	8	-7 NS	11 [6]	221 [7]	9 [6]	221 [7]

[1] Pooled standard deviation using all concentration data
[2] Concentration Reduction calculated using Eq(6).
[3] Mean settled volume fraction for defined settling time.
[4] Mass removal efficiency calculated using Eq(5).
[5] Concentration of the settled solids calculated using Eq(4).
NS = Concentration reduction not significantly different from zero based on calculation of uncertainty using Eq(7).
[6] Actual mass removal efficiency if CR_C was not significantly different from zero = 100 x $SVF(t)$.
[7] Mean of initial and supernatant concentrations was used for C_I and C_{SUP}. in Eq(4).

The volume of milking centre wastewater was measured on day 1 based on the number of manure spreader loads removed and land applied. It was calculated that 52,990 L of wastewater were removed from a pit that held 7 days of waste for a milking herd of 200 Holstein cows. Therefore, it was estimated that, on a typical day, 37.9 L of milking centre wastewater were per produced per cow on this farm. Large amounts of waste milk also increased the volume of wastewater produced. However, there was no way to estimate the actual amount of milk added prior to sample collection on day 2.

Gravity settling experiments were performed in the laboratory using graduated cylinders for both of the milking centre wastewater samples collected and the results including the settled volume fractions, supernatant concentrations, and measures of removal are shown in Tables 1 and 2.

The settled volume fraction, as defined by Eq(1), was the volume of the settled material normalized to the initial volume placed in a graduated cylinder. The average value of $SVF(t)$ decreased from 0.279 at 30 min to 0.254 at 60 min for the sample with a TS of 17,024 mg/L (Table 1). The average $SVF(t)$ for 7165 mg TS/L (Table 2) was 0.105 at 30 min and 0.093 at 60 min. Therefore, increasing the settling time from 30 to 60 min decreased the settled volume by only 9% to 11% depending on the initial TS concentration. These results imply that the settled volume fraction was a function of the initial TS content of the wastewater and that the majority of the settling occurred for both waste samples after 30 min of settling.

Milking centre wastewater is a non-homogeneous mixture which leads to unavoidable errors associated with obtaining consistent representative samples, and small errors in the laboratory techniques used to measure the solids and plant nutrients. These errors are accentuated when the means are used to calculate the difference in concentration of a particular constituent (ΔC). Calculation of the difference in concentrations was the controlling error in the calculation of the concentration reduction, Eq(6), and the mass removal efficiency, Eq(5). Previous experience by the author has shown that the variability in sampling is often the greatest source of error in field studies. The uncertainty in taking a difference in two concentrations, $U_{\Delta C}$, was used to evaluate the significance of the difference in the initial and supernatant concentrations and was computed as (Holman, 1978):

$$U_{\Delta C} = [U_{C\text{-}INITIAL}{}^{2} + U_{SUP}{}^{2}]^{0.5} = [\,2\, SD_C{}^{2}\,]^{0.5}. \tag{7}$$

Where,

$U_{C\text{-}INITIAL}$ = the estimate of the uncertainty of the initial concentration,

U_{SUP} = the estimate in the uncertainty of the supernatant concentration, and

SD_C = the pooled standard deviation calculated from all initial and supernatant concentrations.

The pooled standard deviations used to estimate in the uncertainty in the measurement for each of the defined constituents are also shown in Tables 1 and 2. The values in the tables are identical because all data in the study was used to provide the best estimate of SD_C possible for each of the constituents measured.

The concentration reduction and mass removal efficiencies were calculated for all solids and plant nutrients using the defining equations. Sedimentation reduced the concentration of all of the solids and plant nutrients except for TAN, K_2O, and Na since these are soluble in water and the concentration reductions were not different from zero within the estimates of uncertainty.

Overall, gravity settling was effective at removing a portion of all of the defined solids and plant nutrients. The mass removal efficiency for the soluble constituents, TAN, K_2O, and Na, was a consequence of the liquid in the settled material and not a change in concentration. Gravity settling provided the greatest removal for TS, VS, Org-N, P_2O_5, S, Cu, and Zn. The best performance was for the milking centre wastewater with the highest initial concentrations (Table 1). This was due to two factors, a greater change in constituent concentrations, and a greater settled volume fraction. A similar increase in settling performance with an increase in TS was observed by Chastain and Vanotti (2003) for liquid swine manure. In all cases, the MRE_C was greater than CR_C and provided the best measure of settling performance.

Primary treatment by gravity settling divides wastewater into two effluent streams; the supernatant and the settled material. Further treatment or utilization requires design values of the composition of both effluents. The constituent concentrations of the settled material were calculated from the data using Eq(4) following 30 and 60 min of gravity settling for both wastewater samples (Tables 1 and 2). As expected, the concentrations of solids and plant nutrients in the settled material were much higher for all solids and non-soluble plant nutrients. However, extending the settling time from 30 to 60 min only provided a small increase in the settled concentrations. Comparison of the concentrations of the settled material to the initial concentrations (C_{SM} / C_I) indicated that the concentration of TS in the settled layer averaged 2.3 times greater than the initial value for the sample with an initial TS of 7165 mg/L, and to 4.2 times greater than the initial value for the wastewater with C_I = 17,024 mg TS/L. Similar increases in concentration can also be observed for VS (2.5 to 4.8), Org-N (2.7 to 4.6), and P_2O_5 (1.7 to 4.9).

4. Conclusions

The composition and settling characteristics were determined for two large samples of milking centre wastewater collected from the storage pit on a grazing dairy. The concentrations of solids and plant nutrients varied greatly depending on the amount of waste milk added to the storage pit (Tables 1 and 2). The dilution provided by the waste milk reduced the solids and plant nutrient concentrations significantly, and provided some insight into the variability in wastewater composition that actually occurs on a dairy farm.

The design of anaerobic lagoons or digesters for treatment of animal manure typically use volatile solids as a measure of the organic load instead of COD due to the high solids content (*e.g.* ASABE, 2011; Hill, 1991). In addition, a measure of the TS production is also needed to estimate sludge accumulation in a lagoon (ASABE, 2011). Based on data from the more typical sampling day (Table 1, with 37.9L/cow/day)) it was estimated that the solids production for this milking center was 645 g TS/cow/day and 507 g VS/cow/day.

An environmentally sound nutrient management plan requires measurements of the annual production of major plant nutrients (N, P_2O_5, K_2O), and measurement of organic and soluble N concentrations (Chastain and Camberato, 2003). The major plant nutrient production values from the milking centre in this study that can be used for planning purposes were calculated from the data in Table 1 and were 10.4 kg TAN/cow/yr, 6.2 kg Org-N/cow/yr, 5.6 kg P_2O_5/cow/yr, and 3.5 kg K_2O/cow/yr.

The gravity settling experiments indicated that sedimentation was very effective at moving a substantial portion of the solids and plant nutrients from the wastewater to the settled material. Sedimentation of milking centre wastewater was able to remove 41% to 61% of the TS, 47% to 67% of the VS, 45% to 73% of the Org-N, and 45% to 49% of the P_2O_5 depending on the TS content of the initial wastewater. The majority of the settling occurred in 30 min, and the solids content of the settled material was increased by a factor of 2.3 to 4.2 as compared to the initial TS concentration of the wastewater. Therefore, sedimentation would be an effective means to concentrate VS into a smaller volume prior to anaerobic digestion or to concentrate a large fraction of the plant nutrients to facilitate transportation to fertilize remote cropland.

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