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AN EXPERIMENTAL STUDY OF THE
ANEROBIC DIGESTION OF MILK
PLANT BY-PRODUCTS

Thesis for the Degree of B. S.
MICHIGAN STATE COLLEGE
Kenneth S. MacPherson
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THESIS

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An Experimental Study of the
Anerobic Digestion of Milk Plant By-Products

A Thesis Submitted to

The Faculty of
MICHIGAN STATE COLLEGE
of
AGRICULTURE AND APPLIED SCIENCE

by
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The Statement of the Problem

To determine:

- (1) The feasibility of anaerobic digestion as a means of disposal for milk plant by-products.
- (2) The effect of seeding the by-products with sewage sludge on the rate of digestion.
- (3) The quantity and quality of gas produced and its possible use in the milk plant for power or heat.

Introduction

The effective disposal of dairy by-products is one of the most important and troublesome problems to be solved by sanitarians at the present time. Dairy by-products are important due to their common occurrence and to the fact that they are of such composition as to cause disruption of the ordinary processes of waste disposal unless in very dilute form.

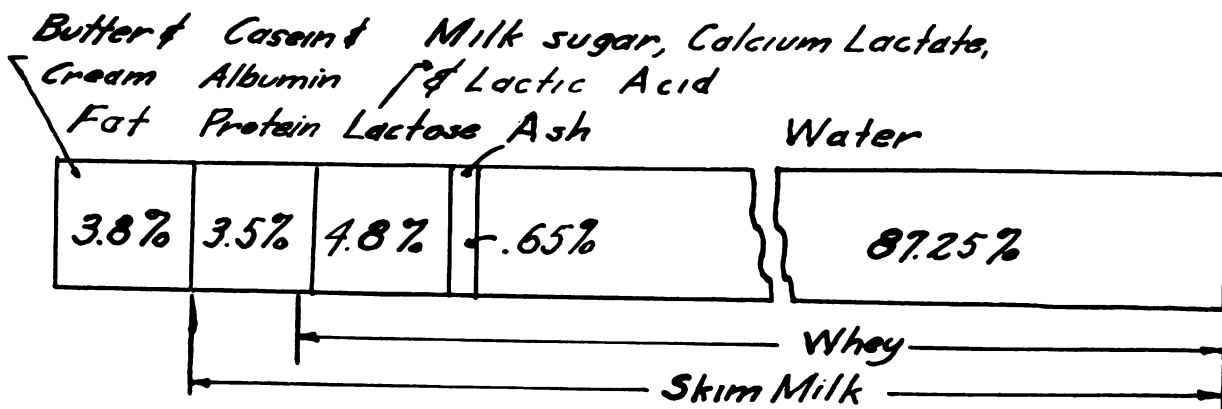
Before modern trends toward treatment of all industrial wastes, it was common practice to dispose of dairy by-products by merely discharging the waste into the nearest body of water. There were many complaints about the pollution in lakes and streams, but due to the fact that no other effective method of disposal was known little was done about this condition.

Dairy by-products are especially troublesome due mostly to their high organic solids content. In the eastern part of the United States there is not much trouble from dairy by-products because most of the dairy products

are used by the dense local population. In the middle west the situation is quite different. So great a volume of dairy products are produced that all are not used by the local population, and as a result there are many milk plants that produce cheese, butter, casein, etc., for other sections of the country. In the manufacture of these products there are by-products of pure milk, skim milk, whey, washings, etc., which are high in organic content and require treatment.

Many of these plants are located on small streams in which the flow of waste from the plant at certain times of the year is equal to, or greater than the stream flow. The resulting depletion of oxygen in the stream causes definite polluted conditions which must be corrected if present sanitary demands are to be satisfied. These plants, many of which are located in small towns, also tax the sewage disposal facilities of the town. Often the sewage disposal plant must be designed for a population several times greater than the actual population.

The composition of milk is shown in the following table:



- (1) Butter and cream are made from the fat.
- (2) Casein and albumin are made from the protein.
- (3) Milk sugar, calcium lactate and lactic acid are made from the lactose.
- (4) Whey contains all ash and lactose, and a small amount (about .5%) of protein.
- (5) Skim milk contains all the ash, protein, and lactose.

The first real development in sewage disposal was the development of septic and Imhoff tanks, which operate as anaerobic digestion chambers. However when milk by-products are put in these tanks, the whole system of digestion is upset. As can be seen from the above table, all milk by-products contain lactose, and this sugar causes the trouble. This is because lactose is readily oxidized to lactic acid, which quickly inhibits the growth of most of the anaerobic species so helpful in reducing the volume of precipitated solids. When the oxidation of lactose has been allowed to proceed to a point where the pH value of 4 or 5 is indicated, the action of most microorganisms is almost completely inhibited. Some attempts have been made to raise the pH into the alkaline range by the addition of lime or caustic soda, which are the only chemicals cheap enough to be of practical use, but the calcium or sodium reacts with the lactic acid to form a calcium or sodium lactate, which is a heavy precipitate, and almost as toxic to the growth of anaerobic bacteria as the free acid. These lactates have some commercial value which may be capitalized upon to pay for the treatment of the dairy by-products, but has not been done to any extent as yet. These deposits of lactate

in the digestion chambers of Imhoff tanks have been known to stay from 6 to 9 months with no apparent decomposition. Another uncesirable acid, butyric acid, is also formed in the Imhoff and septic tanks handling dairy by-products. Butyric acid is objectionable chiefly because of its disagreeable odor, which is noticeable when only small quantities of the acid are present.

Often in sewage plants an Imhoff tank is followed by a trickling filter. If dairy by-products are allowed in the tank, the resulting reactions give an acid effluent and the tank acts merely as a settling basin. The highly acid effluent is relatively stable due to the toxic effect of the acid and is in a poor state for efficient treatment on trickling filters. Treatment of dairy wastes by oxidation on a trickling filter to remove the acid producing material has had some success. Work on this phase has been done by Levine, Burke, Linton, Soppeland, Watkins, Galligan and Goresline at Iowa State College; Eldridge at Michigan State College; and Ruf and Warrick in Wisconsin. At the present time this method is accepted as the best developed although it has definite limitations. These are chiefly that the waste must not be over 2½ to 3 hours old before treatment, and that trouble is found with the filters clogging.

The present trend toward waste disposal by the activated sludge method offers a possibility of oxidizing the acid forming compounds. The activated sludge process has been found effective in destroying these compounds, but very large quantities of air were required, it was extremely

difficult to maintain the sludge in suitable conditions and the effluents were not stable.

It therefore can be said that the common methods of waste disposal by anaerobic processes are not applicable to dairy by-products because: --

(1) The milk sugar is rapidly decomposed with the production of acids that may curdle the waste.

(2) If the dairy by-products are sufficiently concentrated, the resulting acidity will inhibit or kill many of the protein digesting anaerobic bacteria.

(3) The effluents from such anaerobic tanks are in an undesirable state for most efficiency on filters.

(4) The high acidity and consequent relative sterility of the contents of the anaerobic tanks receiving milk by-products results in a relatively high stability of the effluents.

Anaerobic treatment has the most success in milk by-product disposal, but meets with the following difficulties:

(1) Trickling filters need very fresh influent and are bothered with clogging.

(2) The activated sludge method is expensive; uses a large amount of air; is hard to control the sludge formation; and gives an unstable effluent.

The object of this experiment is to determine:

(1) The feasibility of anaerobic digestion as a means of disposal for milk plant by-products which consist of whey, skim milk, washings, etc.

(2) The effect of seeding the by-products with sewage sludge, on the rate of digestion.

(3) The quantity and quality of gas produced and its possible use in the milk plant for power or heat.

To determine the above objects:

(1) Flasks were set up containing a known volume of whey and skim milk, and the digestion of each was measured.

(2) The by-products were seeded with sludge so that mixtures contained 25, 50, and 75% of sludge.

(3) The gas produced by each mixture was analysed in a gas analysis apparatus.

The methods used in determining the above objects will be discussed later.

The mixtures to be digested were first mixed in the desired proportions so that a volume of 1500 ml. of each mixture was available. This volume was then transferred to two liter flasks having a tight rubber stopper with a glass tube connecting the flask to the gas collection apparatus. The gas was collected in an inverted two liter graduate filled with water and standing in a reservoir of water. The apparatus is shown in Figure I.

The milk by-products were gathered at the Michigan State College Dairy in East Lansing, Michigan. The skim milk was 24 hours old but had been kept in a refrigerator to hold back

any bacterial growth. The whey was fresh and was the by-product from the casein removing process. The sludge was gathered at the Lansing sewage treatment plant, Lansing, Michigan. At this plant there are no grit chambers due to the comparative absence of large inorganic solids. In place of the grit chambers are two small sedimentation chambers which remove the large particles of solids in the sewage. These solids are then removed from the chambers and disposed on a dump. From the first chambers, the sewage goes to a large sedimentation basin where the finer settleable solids are removed. This is the sludge used in the experiment, and its analysis is given in Table I.

During the processing of milk for casein removal, the casein is precipitated by the addition of hydrochloric acid and heat, and the resulting by-product is distinctly acid, usually having a pH of 4.2 to 5.3. In the case of the whey used in this experiment, it was found necessary to add 1.05 grams of $\text{Ca}(\text{OH})_2$ per ml. of the whey to raise the pH to about 7.4. This caused a precipitate of calcium lactate, but it was necessary to have this rather than have the pH around 5 because as has been previously discussed, the anaerobic bacteria are almost totally inhibited when the medium in which they are living has a pH in this range.

The gas was removed from the gas collecting apparatus by means of leveling tubes so that the gas was at atmospheric pressure. The carbon dioxide in the gas was dissolved in potassium hydroxide contained in the gas analysis apparatus. The amount of methane was then assumed to be the remaining

gas. This is not an accurate assumption due to the fact that the gas is apt to contain a few percent of oxygen or nitrogen. However, these other gasses occur in such small volumes that they may be considered as negligible.

The gas collection apparatus caused some error in the results. Water was used to fill the inverted graduated cylinders that collected the gas. Carbon dioxide in the gas evolved from the flasks will dissolve in the water and will therefore give high results for the percentage of methane in the evolved gas. However, the temperature of the room in which the experiment was run, remained fairly constant, so after a few days the water should be sufficiently saturated and very little more should be dissolved.

On the first day of the experiment, the various mixtures were made up and the pH of the mixtures were obtained. During the running of the experiment the pH of the mixture in each flask was determined at intervals as shown in Table 2. Also, on the first day, the by-products and the sludge were analysed for total and volatile solids; and the pH of the whey was raised by the addition of $\text{Ca}(\text{OH})_2$. When sufficient gas had been collected in the gas collector, it was analysed. This data is shown in Table 3. The experiment was allowed to run for 40 days and at the end of this time the mixture in each flask was analysed for total and volatile solids. The experiment was run at room temperature of approximately 22 C.

The use of anaerobic digestion to dispose of milk plant by-products has been tried many times and, except under unusual circumstances, is not practical. Flasks #1 and #2, containing

the whey or skim milk exactly as they would come from a milk plant except that the pH of #1 has been raised by the addition of $\text{Ca}(\text{OH})_2$, and were allowed to digest for 40 days. The reduction of solids in both flasks was very small and therefore proves again that simple anaerobic digestion is not a practical method of disposal for milk by-products. As compared to #3, containing only sludge, #1 produced only 20% as much gas and had a reduction of total solids of 33% that of #3, while #2 produced about 33% as much gas and had a reduction of total solids of about 25% that of #3. It should be noticed that both #1 and #2 had almost completely stopped producing gas which is a good measure of the bacterial activity going on. From Table 3 it may be seen that the gas evolved from #1 and #2 was low in methane gas compared to the gas from #3. This gas from #1 and #2 had a B.T.U. value of about 450 which is considerably lower than is commonly used for heating or power.

Flasks #'s 4, 6, and 8, contained 25, 50, and 75% sludge respectively, and the remainder of whey. After a few days of running it was discovered that #4 was digesting aerobically due to an error in laboratory procedure and the results were useless. The data taken on this flask was discarded. Flask #6 showed a definite increase in volatile solids reduction from #1, the ratio being about 3 to 1. #6 showed an increase of 46% in gas production from #1, but the gas evolved was even lower in methane content. #8 produced a large volume of gas between the first and fifth days, but the gas production stopped almost completely after this initial spurt. The gas evolved was at first of high methane content, which may be explained

by the absorption of CO₂ by the water. The total gas evolved from #8 was greater than that from #1, per gram of volatile solids, by 166%. This leads to the conclusion that the seeding of whey with sludge gives greater B.T.U. value. The initial spurt of #8 cannot be explained and the same mixture of whey and sludge should not be expected to act the same again under similar conditions.

#'s 5, 7, and 9 contain 25, 50, and 75% sludge respectively and the remainder of skim milk. There is no relation between the amount of seeding and the rate of digestion in these flasks. The gas production remains about the same as for pure skim milk and also the B.T.U. value of the gas evolved is little changed from the gas produced in #2.

From a study of the daily gas production, Table I, and the pH values of the mixtures during the experiment, Table 2, there is noted that the gas produced always occurred when the pH was above 5.6. In these mixtures this value seems to be the limiting pH of anaerobic activity. It can also be observed that the closer the pH is to a neutral or slightly alkaline solution, the faster the evolution of gas and therefore the faster the rate of digestion. In #8 and #9 the 25% of milk by-product acidifies the whole mixture so quickly that most of the gas evolution occurs before the seventh day. This emphasizes the fact that only relatively small quantities of lactose under proper bacteriological conditions, are necessary to so acidify it's surrounding media in order to kill off or inhibit all anaerobic bacteria. Control of the pH value is the most important factor to be overcome in the anaerobic digestion of

milk by-products.

When considering the following conclusions, take into account that the scope of this experiment is very limited because of the following factors:

(1) Many errors enter into these results because of the difficulty in getting representative samples of these mixtures.

(2) The gas analysis is not strictly accurate.

(3) The temperature was not constant through out the experiment.

(4) No corrections were made in the results for the removal of small quantities (10 ml.) for pH determinations.

(5) The number and volumn of experiments run is so small, due to limited apparatus and time, that too much weight should not be put on any of the following conclusions except #1.

Conclusions

(1) The anaerobic digestion of milk plant by-products is not feasible due to the acid conditions which arise.

(2) Removal of lactose from milk plant by-products is necessary before anaerobic digestion will successfully dispose of them.

(3) Seeding the by-products with fresh sludge will increase the rate of digestion and gas evolution but not in direct proportion to the amount of seeding added.

(4) The gas evolved is of some value for heat or power but it is doubtful that enough gas is produced to warrant the expense of utilizing it.

Table 1.

The Volume of Gas from Each Flask in Ml.

(Totals corrected for vacume of collecting apparatus.)

Days	Flask							
	1	2	3	4	5	6	7	8
0-1		20	720	500	610	570	300	300
1-2		20	40	70	80	250	275	25
3-4		5	50	25	80	50	75	325
4-5		25	390	120	120	10	150	200
5-6			190		85	210	80	1400
6-8		40	680		275	1460	200	20
8-9		50	865		250	1260	580	
9-11		5	445		100	25	280	
11-12			390		80	5	280	20
12-14		45	670		200	10	680	10
14-15	40	110	550		90	20	340	55
15-17	410	580	880		255	40	540	25
17-18	230	510	440		170	10	550	95
18-20	280	1100	940		225	10	850	420
20-21	160	940	800		280	20	10	10
21-22	30	450	440		105		10	40
22-24	210	1450	1830		510	50	50	50
24-26	120	550	2040		755	5		10
26-27	360	70	1500		550	15	10	
27-28	700	500	2010		500			
28-29	270		1000		250	10		
29-31	350		1710		440			
31-32	90		730		540			5
32-33	140		610		250		90	
33-34	150		610		560	40	20	10
34-34	70		440		200	10		5
35-38	500		1150		840	150	50	20
38-40	30		410		110	250	10	
Total	4048	7490	21854		7260	4552	5816	2862

Table 2.

pH of Material in Each Flask

Days	Flask #							
	1	2	3	5	6	7	8	9
0	7.4	6.9	7.3	7.2	7.3	7.2	7.2	7.2
3	7.6	6.2	7.6	6.3	6.4	6.1	7.0	5.9
11	7.2	5.3	6.8	6.0	5.9	5.9	5.9	5.9
20	7.2	5.2	6.9	5.3	5.8	5.4	5.2	5.7
31	6.6	5.3	6.8	5.3	5.9	5.4	5.2	5.5
40	5.7	5.2	6.8	5.2	5.8	5.4	5.2	5.5

Table 3.

Gas Analysis in % Methane

(Carbon dioxide equals 100% minus the % methane.)

Day	Flask #							
	1	2	3	5	6	7	8	9
0							77.6	
5			78.0			67.6	74.4	
8						71.6		
10			74.2	75.6	57.2			55.8
11			73.9					
14			77.9					
15						64.6		
18		69.0	64.0			45.3		
20	81.5	54.4	60.8					
21				65.9				
22	52.2							
24		51.8	81.5					
26			83.1	54.4	42.2			
28	69.2		80.6					
31			82.2					
33			84.4					
38	44.0	49.2	83.2	55.8	40.2	42.1	40.4	

Table 4.

The Composition and Reduction of Mixtures

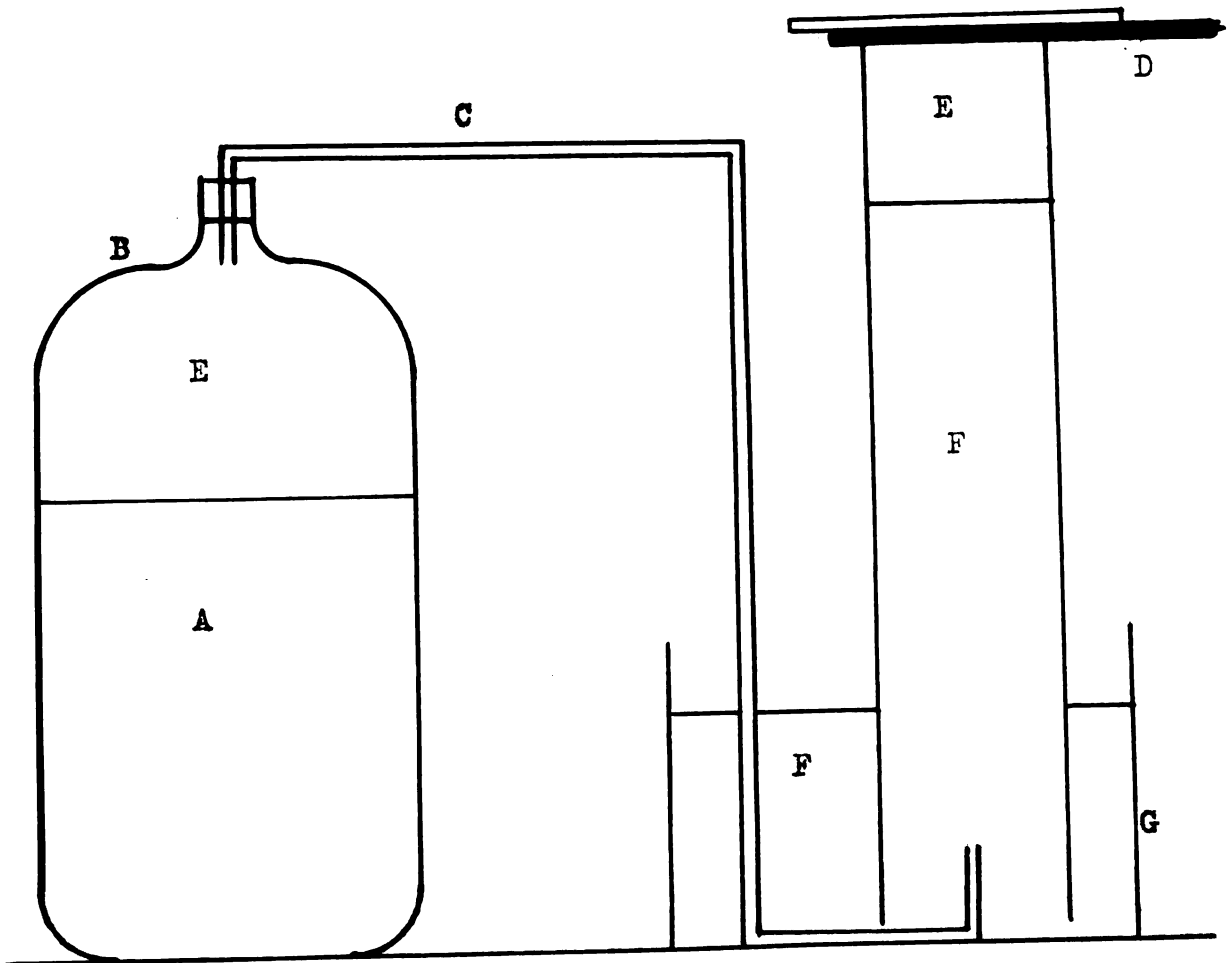
#	Compo- sition	pH at start	--Start--		--Finish--	
			Total Solids % dry wt.	Volume Solids % dry wt.	Total Solids	Volume Solids
1	Whey + Ca(OH) ₂	7.4	6.32	5.68	5.61	5.12
2	Skim Milk	6.9	8.87	7.68	7.93	6.97
3	Sludge	7.3	4.41	3.74	2.93	1.96
5	25% Sludge 75% Sk. Mlk.	7.2	7.76	6.45	7.06	5.92
6	50% Sludge 50% Whey	7.3	5.36	4.21	4.21	3.22
7	50% Sludge 50% Sk. Mlk.	7.2	6.64	5.21	5.23	4.27
8	75% Sludge 25% Whey	7.2	4.86	3.84	3.91	3.02
9	75% Sludge 25% Sk. Mlk.	7.2	5.52	3.98	4.27	3.49

Table 4 Continued

The Composition and Reduction of Mixtures

#	Reduction of Total Solids % dry wt.	Reduction of Volital Solids % dry wt.	Gas in ml. per gm. Volitile Solids at start.
1	.71	.49	46.6
2	.94	.71	65.2
3	1.48	.78	582.5
5	.70	.53	72.2
6	1.15	.99	67.2
7	1.41	.94	69.9
8	.97	.82	124.0
9	.75	.49	46.7

Apparatus



- A- mixture
- B- flask
- C- glass tube
- D- support
- E- gas
- F- water
- G- reservoir

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