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WILTED AND UNWILTED SILAGE IN THE FEEDING OF DAIRY CATTLE

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ETTALA, E., RISSANEN, H., VIRTANEN, E., HUIDA, L. & KIVINIEMI, J. 1982. **Wilted and unwilted silage in the feeding of dairy cattle.** *Ann. Agric. Fenn.* 21: 67—83. (Agric. Res. Centre, North Savo Exp. Sta., SF-71750 Maaninka, Finland.)

The feeding value of wilted precision-chopped and unwilted, flail-harvested grass silages was determined in four experiments in 1977—1979. Unwilted precision-chopped silages were also studied in two experiments. The silages were preserved in tower, clamp and bunker silos. AIV preservative consisting mainly of formic acid was added at the chopping stage at the rate of 4 to 6 litres per tonne. One wilted grass silage was prepared without preservative in a tower silo. 120 cows were used in the feeding experiments. The digestibility experiments were performed with wethers.

All silages prepared with preservative were of good quality. The wilted silages showed less fermentation than the unwilted silages. During feeding the wilted silages were susceptible to the effects of air. The silage made without preservative was of poor quality; its butyric acid content was enough to preclude its use in the feeding experiments.

The differences in the feeding values of wilted and unwilted silages were small. With wilted silages the average intake was slightly higher but the average milk yield slightly lower than with unwilted silages. The digestibility of some wilted silages was lower than that of the unwilted silages. The unwilted precision-chopped silages had the highest digestibility. Silages prepared in tower silos were very similar to those prepared in clamp and bunker silos. The differences in the fat and protein contents of milk produced with the various silages were small.

Index words: unwilted silage, wilted silage, dairy cattle feeding.

INTRODUCTION

Unwilted flail-harvested (single-chop) grass silage made with preservatives is widely used in cattle feeding in Finland. As a result of research, advisory work and frequent testing, silage quality is usually good. However, on the larger farms

faster ensiling would be desirable. Therefore a series of experiments on ensiling techniques, costs and silage quality was conducted as a joint programme between several research institutes. A more rapid technique for silage preparation

was realised by wilting the grass and using precision-chop forage harvesters and clamp and bunker silos.

Since the quality of silage must be maintained, and all methods should provide silage of high quality, palatability and feeding value, the ex-

perimental silages were compared using chemical analyses and feeding and digestibility experiments. Preservatives were used to ensure uniform treatment and the best possible quality. For comparison one wilted silage was ensiled without preservative.

MATERIAL AND METHODS

Preparation of silages

The silages were prepared in 1977 and 1978 at the Institute of Animal Husbandry (IAH) in Jokioinen, and at the North Savo Experimental Station (NSES) in Maaninka.

The experimental silages were as follows. The preservative used in each case was AIV 2. Bunker = low, walled silo; clamp = low, un-walled silo.

Experiment 1. Silage prepared in Maaninka on June 17—20, 1977.

1. Wilted precision-chopped silage, tower silo, preservative applied at 6 l/t.
2. Wilted, precision-chopped, clamp, 6 l/t.
3. Wilted, precision-chopped, tower, no preservative.
4. Unwilted precision-chopped, tower, 6 l/t.
5. Unwilted flail-harvested, tower, 6 l/t.
6. Unwilted flail-harvested, clamp, 6 l/t.

Experiment 2. Silage prepared in Jokioinen on June 8—11, 1977.

1. Wilted precision-chopped, tower, 4 l/t.
2. Wilted precision-chopped, clamp, 4 l/t.
3. Unwilted precision-chopped, tower, 4 l/t.
4. Unwilted precision-chopped, clamp, 4 l/t.
5. Unwilted flail-harvested, tower, 4 l/t.
6. Unwilted flail-harvested, clamp, 4 l/t.

Experiment 3. Silage prepared in Maaninka on June 17—20, 1978.

1. Wilted precision-chopped, tower, 5 l/t.

2. Wilted precision-chopped, bunker, 5 l/t.
3. Unwilted flail-harvested, tower, 5 l/t.
4. Unwilted flail-harvested, clamp, 5 l/t.

Experiment 4. Silage prepared in Jokioinen on July 19—21, 1978.

1. Wilted precision-chopped, tower, 4 l/t.
2. Wilted precision-chopped, bunker, 4 l/t.
3. Unwilted flail-harvested, tower, 4 l/t.
4. Unwilted flail-harvested, bunker, 4 l/t.

The dominant species in the silage swards were timothy (experiments 1 and 3), and timothy and cocksfoot (experiments 2 and 4). Meadow fescue and weeds were present in the swards. Fertilizer N had been applied at the rate of 100 kg/ha for each cut.

In order to obtain both wilted and unwilted silage from the same raw material each section of field was divided as closely as possible into two equal plots. Flail-type forage harvesters (Varsta and Tuhti/Gyro) and, during the first year, precision-chop forage harvesters (JF) were used for harvesting unwilted herbage. Grass for wilted silage was mown in the morning with a mower conditioner, and transferred to the silo with a precision-chop harvester (JF and Valtra) in the afternoon or evening of the same day (experiments 1, 2 and 4) or the following day (experiment 3). The weather was fine. The windrows were not teded. The wilting raised the dry matter (DM) content to 30—40 %.

AIV 2 solution, which contains 83 % formic acid and 2 % orthophosphoric acid by weight,

was added to the herbage from an applicator built into the harvester at the rate of 4–6 litres per tonne.

The tower silos in experiments 1 and 3 were made of fibreglass (diameter 3 m, height 7,5 m). In experiments 2 and 4 the inner surface of the towers (diameter 5,5 m, height 6,5 m) was sealed with fibreglass. In experiment 3 the walls of the bunker silo were made of formed plywood and the base of reinforced concrete (width 4,5 m, length 16 m, height 2,8 m), in experiments 2 and 4 both walls and base were of reinforced concrete (width 5 m, length 14 m, height 2 m).

The tower silos were filled straight from the multi-purpose trailer (experiments 2 and 4), or with an elevator (1 and 3); the herbage was levelled and consolidated by hand. Herbage in the clamp silos was consolidated by driving a tractor to and fro over it. In the bunker silos the herbage was consolidated with a tractor equipped with front loader. Filling of the bunker silos was started from the closed end, the front of the herbage pile being kept as steep as possible. Polythene sheet was used in all silos to cover the top of the silage, to cover the sides of the clamps (in experiment 2 the base as well), and in experiment 3 to line the walls of the bunker silos.

Herbage in the tower silos was weighted with water bags or concrete blocks (350 to 400 kg/m²); clamp and bunker silos were covered with a 15–20 cm layer of sawdust.

Animal trials

120 Ayrshire cows were used in the feeding experiments (Table 6), and wethers in the digestibility experiments in a Latin square design.

The experimental period of 70–91 days (Fig. 1–4) was preceded by a preliminary period of 2 to 4 weeks and a transition period of 1 week. In experiments 1, 2 and 4 there was also a follow-up period of 2 to 3 weeks (Fig. 1, 2 and 4).

During the preliminary and follow-up periods the animals were given the same type of silage: unwilted flail-harvested (in experiments 1, 2 and 4) or wilted precision-chopped silage (in experiment 3). Transition to the experimental silages was gradual. The animals were grouped so as to minimise inter-group differences as determined from performance data (milk yield, live weight, milk fat content, calving date and silage intake) recorded during the preliminary period.

The silages were fed *ad libitum* to the cows in weighed portions. In experiment 1 hay was fed at 1 kg/cow/day and in the other experiments 2 kg. The concentrates fed consisted of barley alone or, in experiments 1 and 3, whey meal (0,5 or 0,8 kg/cow/day) plus barley. The amount of concentrates was 0,4 kg for each kg of 4 % fat-corrected milk (FCM) exceeding 5 kg. Minerals were supplied by mixing them with the concentrates. Unconsumed feed was weighed daily.

The yield of each cow at each milking was recorded. Fat and protein contents were determined with the Infra Red Milk Analyser at 1 to 2-week intervals. The cows were weighed on two consecutive days at the beginning of the preliminary, transition and experimental periods, during the experimental period at 28-day intervals, and at the end of the experiment.

Feed analyses

Grass samples were collected from each trailer load; each combined sample represented 2 or 3 loads. Silage scheduled for feeding during the following fortnight was sampled thoroughly in the silo and the material combined and mixed for analysis. Samples of hay, barley and whey meal were collected daily and combined and analysed at fourweek intervals.

Ash, crude protein and crude fat (ether extract) were determined by standard methods. The DM content of silage, determined by drying

at 105 °C, was corrected for volatiles: DM (corrected) = DM % (105 °C) + butyric acid % + propionic acid % + 0,8 × acetic acid % (JARL and HELLEDAY 1948, NORDFELDT 1955). Crude fibre was determined by a modification of the Weende method (HIRSJÄRVI 1954). For the other analyses, fresh silage plus cold water (1 + 10) was blended (Braun blender) for 5 minutes, cooled, and blended for a further 5 minutes. The filtrate was analysed for water soluble nitrogen by the Kjeldahl method and volatile fatty acids by gas chromatography (HUIDA 1973); lactic acid (BARKER and SUMMER-

SON 1941), ammonium nitrogen (McCULLOUGH 1967) and sugars (SOMOGYI 1945) were determined colorimetrically. For the determination of sugars the silage extract (30 ml) was shaken with 4 g regenerated Duolite A 7 ion exchange resin and 4 g Duolite C 20 for 1 hour in a conical flask and filtered. 10 ml of the filtrate in a glass-stoppered tube (18 × 150 mm) was hydrolysed with 2 ml 0,12 N sulphuric acid for 30 min at 100 °C, then rapidly cooled and neutralised with 2 ml 0,12 N NaOH. The reducing sugars in the solution were determined photometrically and the results are given as glucose.

RESULTS AND DISCUSSION

Silage composition

The average DM content of fresh-harvested grass was 19,1 % (range 15,0—21,9 %, Table 1). After wilting, average DM contents of 27,5—

33,4 % were reached during the first day (experiments 1, 2 and 4), and more than 36 % in material harvested the day after mowing (experiment 3, Table 1). The overall mean DM content of the wilted grass was 31,7 %.

Table 1. Quantity and composition of herbage ensiled; rate of application of preservative.

| Silage | Herbage kg | DM % | % in DM | | | | | AIV 2 l/t |
|----------------------------------|------------|------|---------|---------------|-----------|-------------|----------------|-----------|
| | | | Ash | Crude protein | Crude fat | Crude fibre | N-free extract | |
| Experiment 1 | | | | | | | | |
| Wilted precision-chop., tower | 23 750 | 28,8 | 8,1 | 18,7 | 3,4 | 26,6 | 43,2 | 6,1 |
| Wilted precision-chop., clamp | 33 510 | 31,0 | 8,3 | 18,5 | 3,5 | 26,0 | 43,7 | 6,2 |
| Wilted precision-chop., tower | 21 350 | 30,0 | 8,1 | 19,4 | 3,0 | 26,4 | 43,3 | 0 |
| Unwilted precision-chop., tower | 37 900 | 19,0 | 8,3 | 18,6 | 3,7 | 27,0 | 42,4 | 5,8 |
| Unwilted flail-harvested, tower | 35 950 | 19,8 | 8,5 | 18,4 | 3,7 | 26,6 | 42,9 | 6,0 |
| Unwilted flail-harvested, clamp | 59 050 | 20,0 | 8,8 | 19,8 | 3,8 | 25,9 | 41,7 | 5,7 |
| Experiment 2 | | | | | | | | |
| Wilted precision-chop., tower | 37 000 | 31,6 | 10,1 | 17,5 | 3,4 | 20,5 | 48,5 | 3,9 |
| Wilted precision-chop., clamp | 38 000 | 33,4 | 10,0 | 17,8 | 3,7 | 20,3 | 49,2 | 4,1 |
| Unwilted precision-chop., tower | 65 000 | 21,1 | 10,2 | 18,3 | 4,0 | 19,8 | 47,7 | 3,9 |
| Unwilted precision-chop., clamp | 59 000 | 19,4 | 9,8 | 18,4 | 4,1 | 19,9 | 47,8 | 3,8 |
| Unwilted flail-harvested, tower | 65 000 | 20,1 | 11,1 | 19,0 | 4,2 | 19,4 | 46,4 | 4,2 |
| Unwilted flail-harvested, clamp | 65 000 | 21,3 | 10,1 | 19,3 | 4,2 | 19,4 | 47,1 | 4,0 |
| Experiment 3 | | | | | | | | |
| Wilted precision-chop., tower | 22 800 | 36,6 | 8,7 | 16,4 | 3,1 | 26,3 | 45,4 | 4,9 |
| Wilted precision-chop., bunker | 73 000 | 36,9 | 8,9 | 15,8 | 3,1 | 26,7 | 45,5 | 5,1 |
| Unwilted flail-harvested, tower | 34 700 | 17,9 | 8,3 | 17,0 | 3,5 | 25,3 | 45,8 | 5,2 |
| Unwilted flail-harvested, clamp | 71 250 | 21,9 | 8,6 | 17,2 | 3,4 | 25,3 | 45,5 | 5,2 |
| Experiment 4 | | | | | | | | |
| Wilted precision-chop., tower | 64 000 | 27,9 | 11,8 | 18,5 | 3,7 | 25,9 | 40,1 | 4,1 |
| Wilted precision-chop., bunker | 64 000 | 27,5 | 12,0 | 18,6 | 3,9 | 26,3 | 39,3 | 4,3 |
| Unwilted flail-harvested, tower | 120 000 | 15,0 | 11,0 | 20,2 | 4,0 | 25,1 | 39,8 | 4,0 |
| Unwilted flail-harvested, bunker | 119 000 | 15,0 | 11,0 | 19,6 | 4,0 | 26,2 | 39,2 | 3,9 |

The average DM content of the unwilted silages was 22,0 % (Table 2). During storage the DM content rose by 2,9 percentage units as a result of the discharge of effluent. Effluent in experiment 1 from unwilted flail-harvested tower silage totalled 22,8 % of the weight of the grass ensiled, from unwilted precision-chopped tower silage 24,5 %, and in experiment 3 from flail-harvested tower silage 30,5 %. There was negligible effluent from the other unwilted silages. Apparently the quantity of effluent produced from unwilted silage in clamp and bunker silos was lower than that in tower silos, since the increase in DM content averaged 2,2 and 3,5 percentage units for bunker/clamp and tower silos, respectively.

The mean DM content of wilted silage was 30,3 %. During storage the DM content decreased by 1,4 percentage units (Tables 1 and 2). Part of this reduction may be due to experimental

error, i.e. the difficulty of obtaining samples from the unevenly-drying windrows, which were properly representative of the total harvest. Part may be due to water produced during fermentation. Effluent production in wilted silages in tower silos in experiments 1 and 3 was measured. The quantities were small: 2,7 % and 1,4 %, respectively, of the total weight of the grass.

The ash content of unwilted silages in tower silos decreased during ensiling by an average of 1,1 percentage units (from 9,6 % to 8,5 % in DM), that of unwilted silages in horizontal silos remained constant (9,7 %), and that of wilted forage increased by 0,7 percentage units (from 9,7 % to 10,4 %). The ash content of unwilted silages in tower silos differed significantly from that of most wilted silages (Table 2). Loss of minerals is closely related to the volume of effluent (ETTALA and KOSSILA 1980). Since the wilted silages produced no effluent and part of

Table 2. Composition of silages used during the experimental period.

| Silage | DM % | % in DM | | | | |
|--|--------------------|---------------------|--------------------|-------------------|--------------------|--------------------|
| | | Ash | Crude protein | Crude fat | Crude fibre | N-free extract |
| Experiment 1 | | | | | | |
| Wilted precision-chop., tower | 28,6 ^b | 9,2 ^b | 18,6 ^a | 4,6 ^{ab} | 26,6 ^a | 40,9 ^a |
| Wilted precision-chop., clamp | 27,1 ^b | 9,4 ^b | 18,2 ^a | 4,4 ^a | 27,4 ^a | 40,7 ^a |
| Unwilted precision-chop., tower | 22,3 ^a | 7,3 ^a | 18,3 ^a | 5,4 ^c | 28,6 ^a | 40,4 ^a |
| Unwilted flail-harvested, tower | 22,1 ^a | 7,4 ^{ab} | 18,2 ^a | 5,5 ^c | 28,9 ^a | 40,0 ^a |
| Unwilted flail-harvested, clamp | 21,4 ^a | 8,3 ^{ab} | 18,6 ^a | 5,1 ^{bc} | 27,7 ^a | 40,3 ^a |
| Experiment 2 | | | | | | |
| Wilted precision-chop., tower | 31,8 ^c | 11,5 ^c | 18,8 ^{ab} | 4,6 ^{ab} | 22,6 ^{ab} | 42,4 ^a |
| Wilted precision-chop., clamp | 32,9 ^c | 10,5 ^{abc} | 19,8 ^b | 3,8 ^a | 21,5 ^a | 44,4 ^a |
| Unwilted precision-chop., tower | 23,9 ^{ab} | 9,0 ^a | 18,6 ^{ab} | 5,3 ^b | 24,2 ^{bc} | 42,9 ^a |
| Unwilted precision-chop., clamp | 22,3 ^a | 10,2 ^{abc} | 18,4 ^{ab} | 4,7 ^b | 23,5 ^{bc} | 43,2 ^a |
| Unwilted flail-harvested, tower | 24,7 ^b | 9,8 ^{ab} | 18,0 ^a | 5,4 ^b | 24,3 ^c | 42,5 ^a |
| Unwilted flail-harvested, clamp | 23,6 ^{ab} | 10,9 ^{bc} | 18,1 ^a | 4,7 ^b | 23,4 ^{bc} | 43,0 ^a |
| Experiment 3 | | | | | | |
| Wilted precision-chop., tower | 33,4 ^b | 9,6 ^b | 16,1 ^a | 3,8 ^a | 27,1 ^a | 43,4 ^{ab} |
| Wilted precision-chop., bunker | 33,6 ^b | 9,3 ^b | 16,7 ^{ab} | 3,8 ^a | 25,5 ^a | 44,7 ^b |
| Unwilted flail-harvested, tower | 20,2 ^a | 7,5 ^a | 15,8 ^a | 5,3 ^b | 30,3 ^b | 41,0 ^a |
| Unwilted flail-harvested, clamp | 21,2 ^a | 8,4 ^{ab} | 17,9 ^b | 4,6 ^{ab} | 27,2 ^a | 41,9 ^a |
| Experiment 4 | | | | | | |
| Wilted precision-chop., tower | 28,6 ^b | 11,6 ^b | 18,1 ^a | 5,1 ^a | 26,7 ^a | 38,3 ^b |
| Wilted precision-chop., bunker | 26,7 ^b | 12,3 ^b | 19,0 ^a | 4,7 ^a | 26,0 ^a | 37,9 ^b |
| Unwilted flail-harvested, tower | 20,5 ^a | 10,1 ^a | 19,9 ^a | 7,0 ^c | 30,2 ^b | 32,7 ^a |
| Unwilted flail-harvested, bunker | 20,1 ^a | 10,6 ^a | 20,3 ^a | 6,4 ^b | 29,6 ^b | 33,1 ^a |

The significance of the differences within experiments was tested by means of analyses of variance and the differences between means with Tukey's test (STEEL and TORRIE 1960). Values in the same column and group of experiments without the same superscript letter differ significantly a-c: P < 0,05.

the organic matter was lost through fermentation the relative proportion of minerals increased.

There was only a slight difference in the average crude protein content of the grass and the corresponding unwilted silages (18,7 % and 18,4 % in DM). The crude protein content of the wilted silages increased during ensiling by an average of 0,5 percentage units (from 17,7 % to 18,2 %). The increase indicates that the loss of crude protein was lower than the loss of other organic material.

During ensiling the content of N-free extract showed a clear decrease in both types of silage: unwilted silages 4,1 percentage units (from 44,2 % to 40,1 % in DM) and wilted silages 2,8 percentage units (from 44,4 % to 41,6 %). In three cases out of the four, the differences in the contents of N-free extract in unwilted and wilted silages were statistically significant in experiments 3 and 4 (Table 2).

Due to fermentation the ether extract of unwilted silages increased more than that of wilted silages (from 3,9 % to 5,4 % in DM, and from 3,5 % to 4,2 %, respectively). The differences between unwilted and wilted silages were significant in most cases (Table 2).

In most silages the crude fibre content increased during ensiling, as the proportion of fermentable and readily soluble components decreased. In unwilted tower silages the increase was 3,9 percentage units (from 23,9 % to 27,8 % in DM), in unwilted clamp and bunker silages 2,9 percentage units, in wilted tower silages 0,9 percentage units and in wilted clamp and bunker silages 0,3 percentage units (from 24,8 % to 25,1 %). With the exception of experiment 1 some of differences in crude fibre content between unwilted and wilted silages were significant (Table 2).

Silage quality

On average, the quantities of preservatives actually used in the preparation of the silages

were reasonably close to those planned (p. 68, Table 1). The consumption of preservative was recorded for each load and the loads were weighed.

All silages prepared with preservative were of very good quality (Table 3). Fermentation was usually slight, particularly in the wilted silages. Butyric acid was either present in very small quantities or was not detected. In wilted silages ammonium nitrogen averaged 4,7 % of the total nitrogen and in unwilted silages 3,6 %. Silage quality analyses were made on the same portions of silage as those used in the feeding experiments.

The wilted, precision-chopped, no-preservative tower silage (experiment 1) was of poor quality, differing significantly from the other silages (Table 3). In particular the butyric acid and ammonium nitrogen contents were high, even though the silage was prepared very carefully and the fibreglass silo was airtight. Owing to its high content of butyric acid the silage was excluded from the feeding experiment, as it was feared that the milk produced, which was intended for processing in a cheese dairy factory, would be contaminated with butyric acid bacteria. The silage was analysed, however, and its digestibility determined.

MARSH (1979) has reviewed many studies comparing wilted and unwilted silages and concluded that wilting restricts the decomposition of the soluble carbohydrate more efficiently than that of the protein. This was apparent in the present series of experiments, though the preservative, too, had a decisive effect on the fermentation.

When the daily consumption of wilted silage was small in relation to the exposed surface area the quality of the surface layer deteriorated during the feeding period. There was a rise in temperature or a growth of mould. These effects were observed in wilted clamp silage (experiment 1) and tower silage (experiment 2). No such problems occurred when the rate of consumption was higher, for example in experi-

Table 3. Silage quality.

| Silage | % in DM | | | | | % of total N | |
|--|--------------------|-------------------|--------------------|--------------------|--------------------|-------------------|--------------------|
| | Sugars | Lactic acid | Acetic acid | Butyric acid | Propionic acid | NH ₄ N | Soluble N |
| Experiment 1 | | | | | | | |
| Wilted precision-chop., tower | 12,2 ^b | 0,6 ^a | 0,42 ^a | 0,00 ^a | 0,00 ^a | 1,3 ^a | 51,4 ^{ab} |
| Wilted precision-chop., clamp | 9,3 ^b | 1,5 ^{ab} | 0,98 ^{ab} | 0,06 ^{ab} | 0,01 ^a | 3,7 ^a | 50,4 ^{ab} |
| Unwilted precision-chop., tower | 9,3 ^b | 2,9 ^{bc} | 0,78 ^a | 0,01 ^a | 0,01 ^a | 2,1 ^a | 55,8 ^b |
| Unwilted flail-harvested, tower | 10,4 ^b | 1,7 ^{ab} | 0,76 ^a | 0,15 ^{ab} | 0,03 ^{ab} | 2,4 ^a | 54,5 ^b |
| Unwilted flail-harvested, clamp | 9,5 ^b | 1,5 ^{ab} | 1,08 ^{ab} | 0,01 ^a | 0,00 ^a | 2,2 ^a | 42,8 ^a |
| Wilted precision-chop., tower | 2,8 ^a | 4,4 ^c | 1,77 ^b | 1,41 ^b | 0,22 ^b | 12,9 ^b | 70,3 ^c |
| no preservative | | | | | | | |
| Experiment 2 | | | | | | | |
| Wilted precision-chop., tower | 16,3 ^{bc} | 4,3 ^{ab} | 2,12 ^b | 0,01 ^a | 0,00 ^a | 5,0 ^a | 61,9 ^a |
| Wilted precision-chop., clamp | 19,1 ^c | 1,9 ^a | 1,07 ^a | 0,04 ^a | 0,01 ^a | 4,5 ^a | 58,4 ^a |
| Unwilted precision-chop., tower | 5,4 ^a | 6,2 ^b | 2,49 ^b | 0,00 ^a | 0,03 ^a | 3,2 ^a | 53,3 ^a |
| Unwilted precision-chop., clamp | 6,2 ^a | 5,0 ^b | 2,30 ^b | 0,10 ^a | 0,02 ^a | 3,1 ^a | 49,0 ^a |
| Unwilted flail-harvested, tower | 9,1 ^a | 4,4 ^b | 2,42 ^b | 0,01 ^a | 0,03 ^a | 3,1 ^a | 54,3 ^a |
| Unwilted flail-harvested, clamp | 10,2 ^{ab} | 4,3 ^{ab} | 2,41 ^b | 0,00 ^a | 0,04 ^a | 3,1 ^a | 48,9 ^a |
| Experiment 3 | | | | | | | |
| Wilted precision-chop., tower | 14,9 ^b | 1,7 ^a | 0,92 ^a | 0,02 ^a | 0,00 ^a | 5,7 ^a | 62,5 ^a |
| Wilted precision-chop., bunker | 15,0 ^b | 1,1 ^a | 1,00 ^a | 0,01 ^a | 0,04 ^a | 5,4 ^a | 55,9 ^a |
| Unwilted flail-harvested, tower | 5,0 ^a | 4,5 ^b | 1,52 ^a | 0,31 ^a | 0,04 ^a | 6,2 ^a | 60,6 ^a |
| Unwilted flail-harvested, clamp | 12,0 ^{ab} | 2,2 ^a | 1,04 ^a | 0,01 ^a | 0,01 ^a | 3,1 ^a | 51,3 ^a |
| Experiment 4 | | | | | | | |
| Wilted precision-chop., tower | 7,7 ^b | 7,0 ^a | 1,53 ^a | 0,00 ^a | 0,00 ^a | 5,5 ^a | 55,6 ^a |
| Wilted precision-chop., bunker | 8,3 ^b | 5,9 ^a | 1,44 ^a | 0,00 ^a | 0,00 ^a | 6,2 ^a | 55,4 ^a |
| Unwilted flail-harvested, tower | 1,6 ^a | 5,1 ^a | 1,67 ^a | 0,01 ^a | 0,01 ^a | 5,5 ^a | 52,6 ^a |
| Unwilted flail-harvested, bunker | 3,0 ^a | 5,6 ^a | 1,85 ^a | 0,01 ^a | 0,03 ^a | 5,6 ^a | 49,1 ^a |

Tests and levels of significance as given in Table 2.

ment 4 (12 animals/silo), or when the silage was fed to non-test animals as well. The deleterious effect of air on wilted silage was observed, too, when the polythene cover was torn. Unwilted silages did not show mould growth or any rise in temperature.

Digestibility and feeding value of silages

The average digestibility of the organic matter of wilted and unwilted silage was 71,4 and 72,8 %, respectively (Table 4). The difference was greatest for wilted silage in experiment 3, and for wilted clamp silage versus unwilted precision-chopped silages in experiment 2. The digestibility of the organic matter was highest in unwilted precision-chopped silages; the mean

was 74,8 %, being significantly higher than that of the other silages in experiment 2 and that of the untreated wilted silage in experiment 1.

With wilted silages the digestibility of the crude protein averaged 69,8 % and with unwilted silages 70,9 %. The difference between wilted and unwilted silages was highest in experiment 3 and for untreated, wilted silage versus unwilted precision-chopped silage in experiment 1 (Table 4).

The average crude fat digestibility of wilted and unwilted silages was 65,4 and 69,4 %, respectively; certain inter-silage differences in experiments 1, 2 and 4 were significant (Table 4).

The mean crude fibre digestibility of wilted silages was 70,3 %; that of unwilted silages averaged 74,2 %. The difference between wilted and unwilted silages in experiment 3 was sig-

Table 4. Digestibility of silages and nitrogen balance of wethers.

| Silage | Digestibility, % | | | | | N-balance g/day |
|--|---------------------|--------------------|--------------------|---------------------|--------------------|--------------------|
| | Organic matter | Crude protein | Crude fat | Crude fibre | N-free extract | |
| Experiment 1 | | | | | | |
| Wilted precision-chop., tower | 71,2 ^{ab} | 73,8 ^{ab} | 58,3 ^a | 70,4 ^{ab} | 71,7 ^{bc} | 3,3 ^{ab} |
| Wilted precision-chop., clamp | 71,7 ^{ab} | 74,7 ^{ab} | 62,7 ^b | 72,3 ^{ab} | 71,3 ^{bc} | 3,5 ^{ab} |
| Unwilted precision-chop., tower | 72,6 ^b | 76,3 ^b | 66,4 ^b | 71,7 ^{ab} | 72,0 ^c | 4,2 ^b |
| Unwilted flail-harvested, tower | 71,1 ^{ab} | 74,4 ^{ab} | 62,3 ^{ab} | 69,5 ^a | 71,8 ^{bc} | 4,0 ^{ab} |
| Unwilted flail-harvested, clamp | 70,7 ^{ab} | 72,9 ^a | 65,2 ^b | 72,7 ^b | 69,1 ^{ab} | 2,1 ^{ab} |
| Wilted precision-chop., tower no preservative | 69,9 ^a | 72,8 ^a | 63,3 ^b | 71,6 ^{ab} | 68,2 ^a | 0,5 ^a |
| Experiment 2 | | | | | | |
| Wilted precision-chop., tower | 73,5 ^{abc} | 63,0 ^a | 72,4 ^{bc} | 73,3 ^{abc} | 78,0 ^{bc} | 4,0 ^a |
| Wilted precision-chop., clamp | 71,4 ^a | 64,8 ^a | 61,1 ^a | 68,9 ^a | 76,4 ^{ab} | 4,9 ^a |
| Unwilted precision-chop., tower | 76,6 ^c | 68,2 ^a | 72,1 ^{bc} | 78,6 ^d | 79,6 ^c | 3,1 ^a |
| Unwilted precision-chop., clamp | 75,3 ^{bc} | 65,6 ^a | 73,7 ^c | 77,8 ^{cd} | 78,2 ^{bc} | 4,5 ^a |
| Unwilted flail-harvested, tower | 73,4 ^{ab} | 65,0 ^a | 72,3 ^{bc} | 75,0 ^{bcd} | 75,8 ^{ab} | 4,1 ^a |
| Unwilted flail-harvested, clamp | 71,2 ^a | 63,4 ^a | 66,5 ^{ab} | 72,3 ^{ab} | 74,2 ^a | 3,3 ^a |
| Experiment 3 | | | | | | |
| Wilted precision-chop., tower | 71,1 ^a | 69,1 ^a | 68,6 ^a | 69,5 ^a | 72,9 ^a | 1,6 ^{ab} |
| Wilted precision-chop., bunker | 71,9 ^a | 69,7 ^{ab} | 66,4 ^a | 70,6 ^a | 74,1 ^{ab} | 2,9 ^b |
| Unwilted flail-harvested, tower | 74,2 ^b | 71,7 ^b | 71,2 ^a | 75,0 ^b | 74,8 ^b | 0,4 ^a |
| Unwilted flail-harvested, clamp | 75,6 ^b | 74,8 ^c | 68,7 ^a | 77,2 ^b | 75,7 ^b | 2,5 ^{ab} |
| Experiment 4 | | | | | | |
| Wilted precision-chop., tower | 69,7 ^a | 71,1 ^a | 64,2 ^a | 66,5 ^a | 72,0 ^b | 3,2 ^a |
| Wilted precision-chop., bunker | 70,6 ^a | 72,0 ^a | 69,5 ^{ab} | 70,9 ^a | 69,9 ^b | 5,1 ^a |
| Unwilted flail-harvested, tower | 69,6 ^a | 73,0 ^a | 72,9 ^b | 73,8 ^a | 62,8 ^a | 3,3 ^a |
| Unwilted flail-harvested, bunker | 70,7 ^a | 74,4 ^a | 72,6 ^b | 72,3 ^a | 66,7 ^{ab} | 3,3 ^a |

Tests of significance as given in Table 2. a-d: $P < 0,05$

nificant. A significantly higher crude fibre digestibility was also recorded with the unwilted precision-chopped silages in experiment 2 (Table 4).

The digestibility of the N-free extract (NFE) of wilted silages averaged 73,3 % and that of unwilted silages 72,8 %. The differences were not consistent with treatment. In experiment 3 a significantly lower digestibility was recorded with the wilted tower silage than that with either of the unwilted silages, whereas in experiment 4 the NFE digestibility of the wilted tower silages was higher than that of the unwilted tower silage (Table 4).

MARSH (1979) has summarised the results of a number of digestibility trials: wilting quite often reduced the digestibility. A similar effect was observed in the present study, especially when

the herbage was wilted overnight and its DM content rose to 35-40 %.

The mean digestibility of wilted silages prepared in tower silos was the same as that of wilted silages prepared in other types of silo; the same was found with unwilted silages.

The average nitrogen balance of wethers fed wilted silages was 3,6 g/d, that with unwilted silages being 3,2 g/d. The nitrogen balance with wilted untreated silage was significantly lower than that with unwilted precision-chopped silage in experiment 1 (Table 4). In contrast, unwilted flail-harvested tower silage gave a significantly lower nitrogen balance in experiment 3 than did wilted precision-chopped bunker silage.

The energy value of wilted silages averaged 1,37 kg DM per feed unit (f.u. = 0,7 kg starch equivalent), and that of unwilted silages 1,34 kg

Table 5. Feeding value of experimental silages.

| Silage | kg feed/f.u. | kg feed DM/f.u. | Digestible crude protein | |
|--|-------------------|---------------------|--------------------------|-------------------|
| | | | % in DM | g/f.u. |
| Experiment 1 | | | | |
| Wilted precision-chop., tower | 4,8 ^a | 1,37 ^b | 13,8 ^a | 189 ^a |
| Wilted precision-chop., clamp | 5,1 ^a | 1,36 ^b | 13,7 ^a | 186 ^a |
| Unwilted precision-chop., tower | 6,0 ^b | 1,32 ^a | 13,9 ^a | 184 ^a |
| Unwilted flail-harvested, tower | 6,2 ^b | 1,35 ^{ab} | 13,5 ^a | 182 ^a |
| Unwilted flail-harvested, clamp | 6,5 ^b | 1,36 ^b | 13,5 ^a | 185 ^a |
| Experiment 2 | | | | |
| Wilted precision-chop., tower | 4,1 ^a | 1,32 ^{abc} | 14,9 ^a | 189 ^a |
| Wilted precision-chop., clamp | 4,2 ^a | 1,39 ^{bc} | 12,7 ^a | 176 ^a |
| Unwilted precision-chop., tower | 5,4 ^b | 1,28 ^a | 12,6 ^a | 161 ^a |
| Unwilted precision-chop., clamp | 5,9 ^c | 1,31 ^{ab} | 11,9 ^a | 156 ^a |
| Unwilted flail-harvested, tower | 5,4 ^{bc} | 1,34 ^{abc} | 11,7 ^a | 156 ^a |
| Unwilted flail-harvested, clamp | 5,9 ^c | 1,40 ^c | 11,4 ^a | 159 ^a |
| Experiment 3 | | | | |
| Wilted precision-chop., tower | 4,1 ^a | 1,35 ^c | 11,3 ^a | 153 ^a |
| Wilted precision-chop., bunker | 4,0 ^a | 1,33 ^b | 11,8 ^a | 157 ^{ab} |
| Unwilted flail-harvested, tower | 6,2 ^b | 1,25 ^a | 11,7 ^a | 146 ^a |
| Unwilted flail-harvested, clamp | 5,9 ^b | 1,26 ^a | 13,6 ^b | 171 ^b |
| Experiment 4 | | | | |
| Wilted precision-chop., tower | 5,0 ^a | 1,43 ^{bc} | 13,0 ^a | 187 ^a |
| Wilted precision-chop., bunker | 5,3 ^a | 1,43 ^c | 13,7 ^{ab} | 195 ^{ab} |
| Unwilted flail-harvested, tower | 6,9 ^b | 1,42 ^a | 14,5 ^b | 206 ^{ab} |
| Unwilted flail-harvested, bunker | 7,0 ^b | 1,40 ^{ab} | 15,0 ^b | 211 ^b |

Tests and levels of significance as given in Table 2.

DM/f.u. (Table 5). The energy value of silages wilted to higher DM contents was significantly lower than that of unwilted flail-harvested silages (experiment 3). In the other experiments the differences between extremes were significant (Table 5). In experiment 2 the energy values of unwilted flail-harvested and wilted silages in clamps were lower than those of the other silages. Unwilted precision-chopped silages had the highest energy values.

The concentration of digestible crude protein was the same (13,1 % in DM) in both wilted and the corresponding unwilted silages (Table 5). The highest concentration of digestible crude protein was found in unwilted flail-harvested silages in low silos in experiments 3 and 4; the protein digestibility of these silages was high (Table 4).

Silage intake by cows and utilization of nutrients

Of the total of 120 cows in the feeding trials, 52 received wilted precision-chopped silages, 52 unwilted flail-harvested silages and 16 unwilted precision-chopped silages (Table 6).

The average daily intake of wilted precision-chopped silages was slightly higher than that of unwilted flail-harvested silages, namely 8,5 and 8,2 DM/cow/day, respectively. In experiment 4 the difference between wilted and unwilted silages was marked and significant for wilted tower silage (Table 6, Fig. 4). The palatability of wilted clamp silage and unwilted precision-chopped tower silage was significantly higher than that of the others in experiment 2.

When the consumption of wilted and unwilted

Table 6. Intake of silages by cows and utilization of nutrients.

| Silage | Feed/cow/day | | | | | | | | | | | |
|--|--------------------|------|-------------------|------|--------------------|---------------------|----------------------------|------|-------|--------------------------|--------------------|----------------------|
| | Silage | | | | | | Concentrates ¹⁾ | | | Total Feed ²⁾ | | |
| | kg | | kg DM | | f.u. | DCP g | kg DM | f.u. | DCP g | kg DM | f.u. | DCP g |
| | \bar{x} | s.d. | \bar{x} | s.d. | | | | | | | | |
| Experiment 1 (5×4 = 20 cows) | | | | | | | | | | | | |
| Wilted precision-chop., tower | 28,8 ^a | 0,8 | 8,5 ^a | 0,3 | 6,2 ^a | 1 221 ^a | 5,5 | 6,4 | 595 | 14,8 ^a | 13,1 ^a | 1 872 ^a |
| Wilted precision-chop., clamp | 31,4 ^{ab} | 2,9 | 8,6 ^a | 0,8 | 6,3 ^a | 1 181 ^a | 5,1 | 5,9 | 546 | 14,5 ^a | 12,7 ^a | 1 783 ^a |
| Unwilted precision-chop., tower | 35,0 ^{bc} | 1,3 | 7,8 ^a | 0,3 | 5,9 ^a | 1 121 ^a | 5,2 | 6,0 | 561 | 13,8 ^a | 12,4 ^a | 1 738 ^a |
| Unwilted flail-harvested, tower | 37,7 ^c | 2,3 | 8,5 ^a | 0,5 | 6,3 ^a | 1 157 ^a | 5,5 | 6,3 | 590 | 14,7 ^a | 13,1 ^a | 1 803 ^a |
| Unwilted flail-harvested, clamp | 37,9 ^c | 2,5 | 8,1 ^a | 0,5 | 5,9 ^a | 1 085 ^a | 5,3 | 6,0 | 563 | 14,0 ^a | 12,4 ^a | 1 704 ^a |
| Experiment 2 (6×6 = 36 cows) | | | | | | | | | | | | |
| Wilted precision-chop., tower | 25,7 ^a | 3,7 | 8,2 ^a | 1,2 | 6,2 ^a | 1 119 ^{ab} | 4,7 | 5,1 | 481 | 14,6 ^a | 12,1 ^a | 1 705 ^{ab} |
| Wilted precision-chop., clamp | 30,7 ^a | 3,2 | 10,2 ^b | 1,1 | 7,3 ^{bc} | 1 280 ^b | 5,4 | 5,8 | 550 | 17,0 ^b | 13,9 ^b | 1 921 ^c |
| Unwilted precision-chop., tower | 41,4 ^b | 1,8 | 10,0 ^b | 0,4 | 7,8 ^c | 1 257 ^b | 5,2 | 5,6 | 528 | 16,4 ^{ab} | 14,1 ^b | 1 866 ^{bc} |
| Unwilted precision-chop., clamp | 42,7 ^b | 7,1 | 9,6 ^{ab} | 1,6 | 7,3 ^{abc} | 1 151 ^{ab} | 5,2 | 5,6 | 526 | 16,1 ^{ab} | 13,6 ^{ab} | 1 765 ^{abc} |
| Unwilted flail-harvested, tower | 38,1 ^b | 3,9 | 9,4 ^{ab} | 1,0 | 7,0 ^{abc} | 1 101 ^{ab} | 5,1 | 5,5 | 522 | 15,9 ^{ab} | 13,3 ^{ab} | 1 710 ^{abc} |
| Unwilted flail-harvested, clamp | 38,7 ^b | 4,2 | 9,2 ^{ab} | 1,0 | 6,5 ^{ab} | 1 042 ^a | 5,1 | 5,5 | 518 | 15,8 ^{ab} | 12,8 ^{ab} | 1 657 ^a |
| Experiment 3 (4×4 = 16 cows) | | | | | | | | | | | | |
| Wilted precision-chop., tower | 20,4 ^a | 4,3 | 6,9 ^{ab} | 1,4 | 5,1 ^a | 778 ^a | 5,0 | 5,7 | 571 | 13,3 ^a | 11,7 ^a | 1 515 ^a |
| Wilted precision-chop., bunker | 21,6 ^a | 1,7 | 7,3 ^{ab} | 0,6 | 5,5 ^{ab} | 868 ^a | 4,8 | 5,5 | 549 | 13,6 ^a | 11,9 ^a | 1 580 ^a |
| Unwilted flail-harvested, tower | 32,7 ^b | 2,4 | 6,7 ^a | 0,4 | 5,4 ^{ab} | 786 ^a | 4,7 | 5,4 | 537 | 12,8 ^a | 11,7 ^a | 1 484 ^a |
| Unwilted flail-harvested, clamp | 34,6 ^b | 3,9 | 7,4 ^b | 0,8 | 5,9 ^b | 1 013 ^b | 5,0 | 5,7 | 576 | 13,9 ^a | 12,6 ^a | 1 751 ^a |
| Experiment 4 (4×12 = 48 cows) | | | | | | | | | | | | |
| Wilted precision-chop., tower | 32,0 ^a | 4,1 | 9,1 ^b | 1,2 | 6,4 ^a | 1 175 ^b | 5,2 | 5,9 | 476 | 15,5 ^a | 12,9 ^a | 1 773 ^a |
| Wilted precision-chop., bunker | 33,4 ^a | 4,1 | 9,0 ^{ab} | 1,1 | 6,3 ^a | 1 228 ^a | 5,0 | 5,6 | 456 | 15,2 ^a | 12,6 ^a | 1 812 ^{ab} |
| Unwilted flail-harvested, tower | 38,9 ^b | 5,6 | 8,0 ^a | 1,2 | 5,6 ^a | 1 159 ^a | 5,1 | 5,8 | 468 | 14,3 ^a | 12,1 ^a | 1 751 ^{ab} |
| Unwilted flail-harvested, bunker | 41,5 ^b | 4,5 | 8,3 ^a | 0,9 | 5,9 ^a | 1 257 ^a | 5,6 | 6,3 | 513 | 15,2 ^a | 13,0 ^a | 1 900 ^b |

The differences within experiments in intake of silage and total feed DM, f.u. and DCP (digestible crude protein) were assayed by means of least-squares analyses of variance, and the differences between means with Tukey's test. In order to eliminate differences between animals, the intakes were corrected by linear regression analysis, for live weight, milk yield and intake recorded during the preliminary period. Values in the same column and group of experiments without a common superscript letter differ significantly a-c: $P < 0,05$.

¹⁾ In experiments 1 and 3, respectively, half of the animals were given 0,5 kg or 0,8 kg whey meal/cow/day in addition to barley. The other half received only barley.

²⁾ The DM, f.u. and DCP supplied by the hay ration can be obtained by subtracting the Silage plus Concentrates quantities from the Total.

silages was considered together for each type of silo it was observed that the average consumption of clamp silage was slightly higher than that of tower silage, namely 8,6 and 8,3 kg DM/cow/d, respectively. In experiment 3 the palatability of unwilted flail-harvested clamp silage was significantly higher than that of corresponding tower silage (Table 6, Fig. 3); there was a small but distinct difference in the quality of the two silages (Table 3). There was a significant difference in palatability between wilted clamp silage and wilted tower silage in experiment 2 (Table 6, Fig. 2). The difference in quality appeared only in the top layer as the surface of the silage in the tower was attacked by mould

and warmed up, as a result of the low rate of consumption. In experiment 1, the newly-exposed surface of wilted clamp silage suffered from heating and caused considerable variation in intake (Fig. 1). The results indicate that cows are very sensitive to silage quality, as was observed in earlier studies (ETTALA and LAMPILA 1978).

One of the conclusions of MARSH's (1979) review is that wilting usually increases the intake of silage. The increase is greater when concentrates are not fed. In some of the experiments reviewed, however, the increase was very small. Apparently the variation in intake depends on the quality of the silage, whether wilted or un-

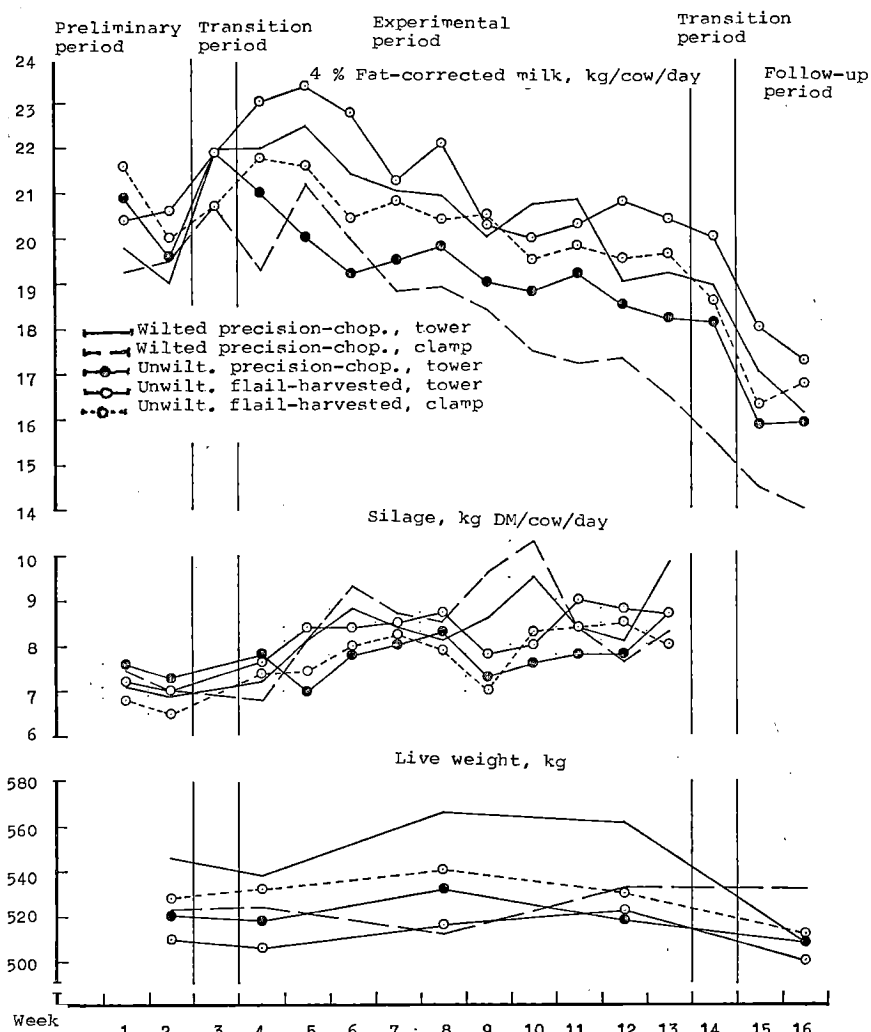


Figure 1. Milk yields, silage intakes and live weight changes in experiment 1 (Maaninka 1977-1978).

wilted. CASTLE and WATSON (1973) observed that the application of formic acid has a positive effect on the intake of wilted silage.

According to calculations, the cows obtained on the average almost equal amounts of energy from wilted and unwilted flail-harvested silages, namely 6,2 and 6,1 f.u. per day, respectively (Table 6). There was also a negligible difference between the average energy intake from tower silage and the corresponding bunker/clamp silages (6,0 and 6,2 f.u.). On average, slightly

more energy was obtained from the unwilted precision-chopped silages than from the corresponding unwilted flail-harvested silages (7,0 f.u. and 6,6 f.u.). The differences in the average intakes of protein were very small. Significant differences in the intake of energy and protein between the most and the least palatable silages in experiments 2 and 3 were recorded.

The average ration of concentrates given in these experiments varied from 4,7 to 5,6 kg DM/cow/day (Table 6) and was in accordance

with milk yield (p. 69). The hay ration was 1 kg (experiment 1) or 2 kg (experiments 2 to 4) per cow per day. In experiments 2 and 4 the cows did not consume all the hay given.

The mean total DM intake of cows on wilted silages was slightly higher than with unwilted flail-harvested silages: 14,8 and 14,6 kg DM/day, respectively (Table 6). The average energy intake was the same in both groups (12,6 f.u./day). Cows on bunker/clamp silages consumed on average slightly more DM than cows on tower silages (15,0 and 14,6 kg/day, respectively). The corresponding f.u. intakes were 12,7 and 12,5, and the protein intakes 1 760 and 1 700 g. Significantly lower intakes of total DM and energy were obtained for wilted tower silage in experiment 2 (Table 6).

The proportion of wilted silage in the total dry matter intake was on average 56,9 %, in the

total energy intake 48,9 % and in the protein intake 63,4 %. The respective values for unwilted flail-harvested silage were 56,2 %, 48,0 % and 62,5 %.

Milk yield and change in live weight of cows

The milk yield of the 52 cows on wilted silages was, on average, slightly lower than that of the 52 cows on unwilted flail-harvested silages (17,0 and 17,4 kg/day). The corresponding yields of 4 % fat-corrected milk (FCM) were 18,9 and 19,7 kg/day. The only significant difference observed was in the FCM yield of cows on wilted and unwilted bunker silages in experiment 4 (Table 7). In this experiment the higher intake of wilted silages did not lead to a higher milk

Table 7. Live weight changes and yields of milk, protein and fat.

| Silage | Milk yield/cow/day | | | | Milk | | | | Live weight change kg |
|--|--------------------|------|--------------------|------|-------------------|------|-------------------|------|-----------------------|
| | kg | | 4 % FCM kg | | fat % | | protein % | | |
| | \bar{x} | s.d. | \bar{x} | s.d. | \bar{x} | s.d. | \bar{x} | s.d. | |
| Experiment 1 (5 × 4 = 20 cows) | | | | | | | | | |
| Wilted precision-chop., tower | 18,4 ^a | 4,8 | 20,8 ^a | 4,7 | 5,10 ^a | 0,57 | 3,89 ^a | 0,42 | +25 |
| Wilted precision-chop., clamp | 16,7 ^a | 5,4 | 18,2 ^a | 4,8 | 4,81 ^a | 0,57 | 3,86 ^a | 0,43 | +14 |
| Unwilted precision-chop., tower | 17,1 ^a | 2,4 | 19,4 ^a | 2,9 | 5,07 ^a | 0,29 | 3,74 ^a | 0,12 | + 2 |
| Unwilted flail-harvested, tower | 18,2 ^a | 5,3 | 21,6 ^a | 5,9 | 5,54 ^a | 0,51 | 3,85 ^a | 0,41 | +15 |
| Unwilted flail-harvested, clamp | 18,5 ^a | 6,7 | 20,3 ^a | 7,2 | 4,78 ^a | 0,43 | 3,79 ^a | 0,19 | + 4 |
| Experiment 2 (6 × 6 = 36 cows) | | | | | | | | | |
| Wilted precision-chop., tower | 15,7 ^a | 2,7 | 18,4 ^a | 2,9 | 5,41 ^a | 0,37 | 3,80 ^a | 0,26 | - 0,5 |
| Wilted precision-chop., clamp | 18,2 ^a | 4,5 | 20,1 ^a | 5,0 | 4,87 ^a | 0,54 | 3,71 ^a | 0,28 | +24 |
| Unwilted precision-chop., tower | 18,4 ^a | 3,5 | 20,8 ^a | 4,1 | 5,05 ^a | 0,40 | 3,67 ^a | 0,32 | +33 |
| Unwilted precision-chop., clamp | 17,3 ^a | 2,8 | 19,4 ^a | 3,0 | 4,99 ^a | 0,46 | 3,92 ^a | 0,36 | +41 |
| Unwilted flail-harvested, tower | 17,8 ^a | 2,9 | 20,5 ^a | 3,3 | 5,25 ^a | 0,37 | 3,78 ^a | 0,13 | ± 0 |
| Unwilted flail-harvested, clamp | 16,9 ^a | 3,0 | 20,0 ^a | 3,7 | 5,50 ^a | 0,66 | 3,91 ^a | 0,44 | +15 |
| Experiment 3 (4 × 4 = 16 cows) | | | | | | | | | |
| Wilted precision-chop., tower | 16,8 ^a | 6,7 | 19,1 ^a | 6,2 | 5,28 ^a | 0,90 | 3,76 ^a | 0,29 | - 5 |
| Wilted precision-chop., bunker | 16,5 ^a | 3,5 | 18,3 ^a | 2,7 | 4,97 ^a | 0,59 | 3,80 ^a | 0,43 | + 8 |
| Unwilted flail-harvested, tower | 16,0 ^a | 3,0 | 18,1 ^a | 3,1 | 5,05 ^a | 0,37 | 3,65 ^a | 0,25 | + 5 |
| Unwilted flail-harvested, clamp | 16,9 ^a | 5,9 | 19,1 ^a | 6,3 | 5,08 ^a | 0,48 | 3,75 ^a | 0,37 | +15 |
| Experiment 4 (4 × 12 = 48 cows) | | | | | | | | | |
| Wilted precision-chop., tower | 17,2 ^a | 3,3 | 18,5 ^{ab} | 3,3 | 4,69 ^a | 0,69 | 3,65 ^a | 0,38 | + 8 |
| Wilted precision-chop., bunker | 16,3 ^a | 1,9 | 17,7 ^a | 2,3 | 4,70 ^a | 0,52 | 3,65 ^a | 0,31 | +12 |
| Unwilted flail-harvested, tower | 16,5 ^a | 3,3 | 18,2 ^{ab} | 2,6 | 4,90 ^a | 0,68 | 3,68 ^a | 0,37 | - 2 |
| Unwilted flail-harvested, bunker | 18,0 ^a | 3,1 | 19,7 ^b | 3,3 | 4,79 ^a | 0,40 | 3,54 ^a | 0,26 | + 6 |

Milk yield and composition data were analysed as described in Table 6. Using linear regression analysis, corrections were made according to milk yield and composition recorded during the preliminary period and interval from last calving.

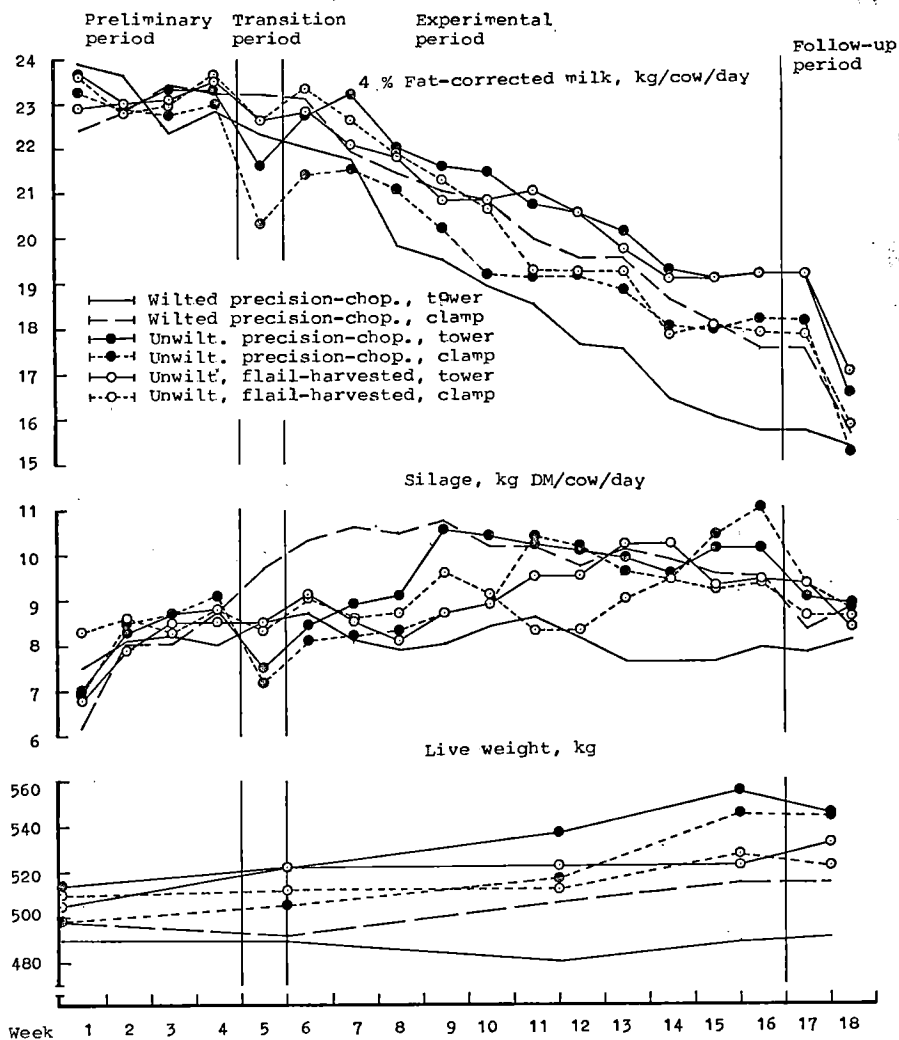


Figure 2. Milk yields, silage intakes and live weight changes in experiment 2 (Jokioinen 1977-1978).

yield (Fig. 4). Similar results were obtained by GORDON (1979) and WEISS et al. (1979). Opposite effects have also been recorded, but in several experiments the rise in yield was small in relation to the increase in silage intake (MARSH 1979).

The average milk yield of cows on tower silages was slightly lower than with bunker/clamp silages (17,1 and 17,3 kg/day), but slightly higher as FCM (19,4 and 19,2 kg/day).

In experiment 1 less milk, but in experiment 2 more milk, was produced from unwilted precision-chopped silage than from the corresponding flail-harvested silage. These results are in line with the measured silage intakes. The differences in milk yield were not significant (Table 7).

The fall in the yield of FCM from the level during the transition period to that at the end

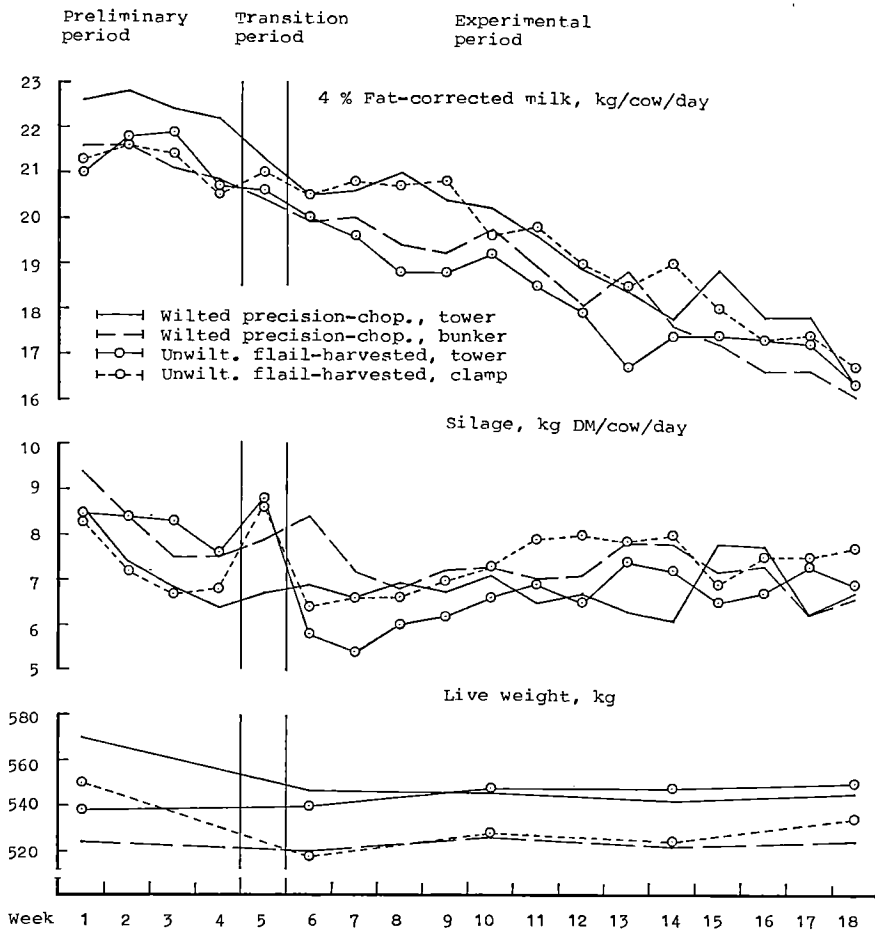


Figure 3. Milk yields, silage intakes and live weights changes in experiment 3 (Maaninka 1978—1979).

of the experimental period of cows on wilted tower and bunker/clamp silages, respectively, was 58 and 65 g/day, and of cows on unwilted flail-harvested tower and bunker/clamp silages 42 and 46 g/day (Fig. 1—4).

In several cases the decrease in yield followed the changes in intake and quality of the silage quite closely. For example, in experiment 2 the consumption of wilted tower silage was the lowest, apparently due to the heat production in the surface layer, and the yield was also lower than that in the other groups (Fig. 2). In experiment 3 the consumption of flail-harvested

tower silage at the beginning of the experiment was low due to inferior quality, and the milk yields dropped correspondingly (Fig. 3). On the other hand, in experiment 1 the fall in the yield of cows on wilted clamp silage was considerably greater than the yield decrease of the others, even although the intake was adequate (Fig. 1). Apparently the variation in intake caused a decrease in production, and possibly the nutritive value of the silage decreased along with the thermal spoilage of the surface layer.

There was no significant difference between feeds in milk fat content in any of the experi-

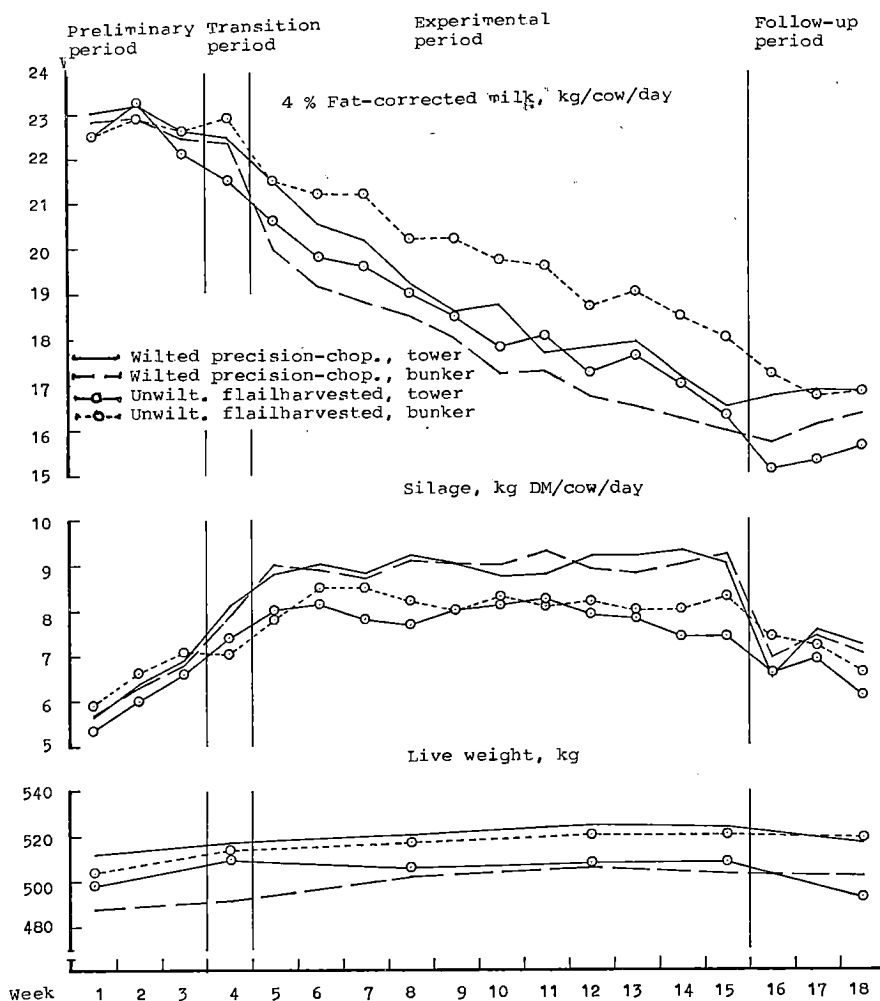


Figure 4. Milk yields, silage intakes and live weight changes in experiment 4 (Jokioinen 1978—1979).

ments (Table 7). However, a slight trend, could be observed. The milk fat content of the 52 cows on wilted silage was on average slightly lower than that of the 52 cows unwilted flailharvested feed (4,97 and 5,11 %, respectively). Similarly, the average milk fat content of cows on bunker/clamp silages was lower than that of cows on tower silages (4,93 and 5,15 %, respectively), and lower with unwilted precision-chopped silages than with the corresponding flailharvested silages (Table 7).

No significant differences were recorded in milk protein contents (Table 7). The group average concentrations were very close to each other, ranging from 3,74 to 3,77 %.

The live weight of the cows remained the same or increased in most groups (Table 7, Fig. 1—4). The average live weight gain during the experimental period of cows on wilted silages was slightly higher than that of cows on unwilted flailharvested silage (10 and 8 kg), and somewhat higher for the cows on bunker/clamp

silages than for those on tower silages (12 and 6 kg).

Differences in live weight gain arise from variations in the utilization of feed energy. The mean energy intake of cows on wilted and unwilted flail-harvested silages was the same, but the FCM yield of cows on wilted silages was slightly lower, so that slightly more energy re-

mained to raise the live weight. The energy intake of the groups on bunker/clamp silage was slightly higher but the FCM yield was slightly lower than in the tower silage groups. Therefore the increase in live weight in the bunker/clamp silage groups was greater than in the tower silage groups.

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SELOSTUS

Esikuivatun ja tuoreen säilörehun vertailu lypsykarjan ruokinnassa

ELSI ETTALA, HEIKKI RISSANEN, ERKKI VIRTANEN, LEA HUIDA ja JAAKKO KIVINIEMI

Maatalouden tutkimuskeskus ja Valtion maatalouskonéiden tutkimuslaitos

Esikuivattuja tarkkuussilputtuja ja tuoreita kelasilputtuja ruohosäilörehuja verrattiin toisiinsa neljässä ruokintakokeessa vuosina 1977—79. Kahdessa kokeessa oli myös tuoreita tarkkuussilputtuja rehuja. Rehut säilöttiin torni- ja tasosäilöihin. Pääasiassa muurahaishappoa sisältävää säilöntäainetta (AIV 2) lisättiin silppuroinnin yhteydessä 4—6 l/tn. Yksi esikuivattu tornirehu valmistettiin myös ilman säilöntäainetta. Ruokintakokeissa oli yhteensä 120 lehmää. Sulavuuskokeet tehtiin päseillä.

Kaikki säilöntäainella säilötyt rehut olivat hyvälaatuisia. Esikuivattujen rehujen käyminen oli vähäisempää kuin tuoreiden. Esikuivatut rehut olivat käytön aikana arkoja ilman vaikutukselle. Ilman säilöntäainetta tehty

rehu oli heikkolaatuista ja se jouduttiin voi happopitoisuuden vuoksi jättämään pois ruokintakokeesta.

Esikuivattujen ja tuoreiden säilörehujen ruokinnalliset erot olivat pieniä. Keskimäärin lehmät söivät vähän enemmän esikuivattuja kuin tuoreita säilörehuja, mutta keskimääräinen maitotuotos oli esikuivatuilla vähän pienempi. Joidenkin esikuivattujen rehujen sulavuus oli heikompi kuin tuoreiden. Paras sulavuus oli tuoreilla tarkkuussilputuilla säilörehuilla. Torni- ja tasosäilörehut olivat keskenään hyvin tasavertaisia. Erilaisilla säilörehuilla tuotetun maidon rasva- ja valkuaispitoisuuserot olivat pieniä.

LOSS OF SELENIUM FROM PLANT MATERIAL DURING DRYING,
STORAGE AND DRY ASHING

TOIVO YLÄRANTA

YLÄRANTA, T. 1982. Loss of selenium from plant material during drying, storage and dry ashing. *Ann. Agric. Fenn.* 21: 84—90. (Agric. Res. Centre, Inst. Soil Sci., SF-31600 Jokioinen, Finland.)

The loss of selenium from Italian rye grass (*Lolium multiflorum* Lam.) during drying and storage, and the loss of selenium from rye grass and barley (*Hordeum vulgare* L.) shoots during dry ashing were investigated. The radioactive isotope ^{75}Se was used in the study.

No selenium was lost from the rye grass during drying for four days at 50 °C in an oven, whereas drying at 100 °C caused a loss of about 4 %. When drying was carried out at room temperature (20—22 °C) for 20 days, more than 3 % of the selenium was released from the rye grass. No loss of selenium was observed when dried rye grass was stored for 13 months at room temperature.

When rye grass dried for four days at 50 °C was placed in an oven for 24-hour periods at temperatures of 100, 150 and 200 °C the selenium loss was clearly greatest at the highest temperature, when the plant material started to carbonize. A total of 20 % of the selenium was released from the rye grass at 200 °C. During dry ashing of the rye grass at 450 °C for 16 hours 42 % of the selenium was lost. Dry ashing of barley shoots for six hours at 500 °C using 8 ml of $\text{Mg}(\text{NO}_3)_2$ solution (650 g $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ in 1 litre of water) per 1 g of sample caused a selenium loss of about 13 %.

Index words: selenium loss, ^{75}Se , drying, storage, dry ashing, Italian rye grass, barley.

INTRODUCTION

Some of the selenium contained in plant material has been reported as being released during drying and storage (WATKINSON 1966, ASHER et al. 1967, EHLIG et al. 1968, GISSEL-NIELSEN 1970 and BISBJERG 1972). EHLIG et al. (1968) found that some 20 plant species, including alfalfa, red clover, timothy, oats, cabbage and maize, lost 0—5 % of their selenium during 30 hours'

drying at 70 °C. ASHER et al. (1967) measured selenium losses of 0,5—3 % from alfalfa during 48 hours' drying at 70 °C. According to BISBJERG (1972) plant samples should be dried immediately after cutting, in a well-ventilated oven at the lowest possible temperature in order to avoid loss of selenium. He considered a drying temperature of 50 °C suitable.

In view of the fact that several months may elapse between sampling and selenium analysis, it is important to know how much selenium is lost from plants during storage. According to GISSEL-NIELSEN (1970) almost 14 % of the selenium contained in red clover was released in 9 months. The alfalfa and rye grass also included in the experiments showed smaller selenium losses. The amount of selenium lost was about the same, irrespective of whether plants were given selenium in the form of selenite or selenate. During storage, more selenium is lost from ground plant material than from plant material compressed into briquettes. BISBJERG (1972) found that in most cases less than 5 % of the selenium was lost from plants during storage for one year. Some of the selenium in the plant material may be released during drying and storage, irrespective of the original selenium content of the sample (ASHER et al. 1967, BISBJERG 1972).

The hydride method for the determination of selenium developed by SIEMER and HAGEMANN (1975) has been widely adopted in Finland during recent years. The hydride method is a simple, sensitive and rapid means of determination using atomic absorption spectrometry. It can be used to obtain reliable values even when the plant sample contains very low amounts of selenium ($\text{Se} < 20 \mu\text{g}/\text{kg}$ of dry material). Dry ashing is an excellent way of decomposing the plant sample in preparation for the hydride method. However, the formation of reducing conditions in the mixture during ashing causes loss of selenium. Selenium losses can be reduced by using $\text{Mg}(\text{NO}_3)_2$ as an ashing aid (GORSUCH 1970).

The aim of this study was to further investigate the loss of selenium from plant material during drying, storage and dry ashing, using the radioactive isotope ^{75}Se .

MATERIAL AND METHODS

Selenium loss from plant material during drying and storage

The loss of selenium-75 from Italian rye grass (*Lolium multiflorum* Lam.) was studied after drying at 50 °C, 100 °C and room temperature. Rye grass containing selenium-75 was grown on a fine sand soil in a 4,5 litre polythene pot. The pot contained four litres of soil fertilized as follows (mg/l of soil): N 275 (NH_4NO_3), K 200 (KCl), P 80 ($\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$), Mg 60 ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$), S 60 ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$), B 0,5 (H_3BO_3), Mo 0,5 ($\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$), Cu 2 (CuCl_2), Mn 2 (MnCl_2), Fe 1 (FeNa-EDTA) and Zn 1 ($\text{ZnNa}_2\text{-EDTA}$). The fertilizer solutions were prepared from analytical grade reagents. The solutions and $\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$ powder were mixed with the soil together

with 5,4 g of $\text{Ca}(\text{OH})_2$ powder as liming agent. The soil pH(CaCl_2) after cutting the rye grass was 5,9. The pH(CaCl_2) was measured from soil solution suspensions (25 ml of soil + 62,5 ml 0,01 M CaCl_2 solution) 2 hours after the addition of the CaCl_2 solution.

Fifty rye grass seeds (variety Romo) were planted in the pot and the soil watered daily to 60 % of the water holding capacity. The grass was grown outdoors under a growing frame. One week after planting, 50 ml of aqueous Na_2SeO_4 solution containing 200 μg Se and 1,9 MBq (50 μCi) ^{75}Se sodium selenate (The Radiochemical Centre, Amersham, England) was added to the soil in the pot. About 5 weeks after planting, a crop corresponding to 12 g dry weight was cut. The selenium content of the plant material was about 3 mg/kg, since accord-

ing to ^{75}Se measurements the crop had taken up almost 20 % of the selenium added to the soil.

For radioactivity measurements the fresh plants were cut into 2-cm pieces and 300–400 mg were placed in 10 ml glass measuring tubes. The tubes were placed in an Automatic Gamma Sample Counter Wallac GTL 500 (Scaler/Timer SC-23, Pulse High Analyser AS-12, Programmer PP-67) with a 2×2 NaI(Tl) well crystal (SCDA-4) detector. The measurement range of 70–190 keV (photopeak of 136 keV) was chosen to give a high counting rate for a background of 70 counts per minute. No differences in counting efficiencies were observed between ^{75}Se measurements carried out on dry rye grass and those carried out on fresh rye grass. With the measurement geometry used in this study the counting efficiency was constant for samples of 5 ml or less. The 2-cm pieces of grass used occupied a volume of about 2.5 ml at the bottom of the measuring tube.

In order to reduce the statistical counting error at least 100 000 impulses were measured using sample tubes and tubes containing a standard solution. The measuring time varied between 5 and 30 minutes. Five parallel tubes were measured for each sample. The samples were dried at 50 °C or 100 °C in an oven (Memmert Tv80uL) or at room temperature in the laboratory. The results of the activity measurements taken during drying and storage were compared throughout with the results obtained from measurement of the five standards of known ^{75}Se activity, in order to correct for the drop in the counting rate due to radioactive decay. This meant that each measurement could be compared with the selenium content measured before drying was started.

In this study the same samples were measured throughout the 13-months storage period in an attempt to avoid any possible errors due to lack of homogeneity in the sample or to changes in the measurement geometry. After drying, the sample tubes were allowed to stand open in the

laboratory, exposed to normal light and temperature (20–22 °C) conditions.

Loss of selenium from plant material during dry ashing

The loss of selenium-75 from rye grass was studied after 16 hours dry ashing at 450 °C, and the loss from barley (*Hordeum vulgare* L.) shoots after dry ashing with $\text{Mg}(\text{NO}_3)_2$ using the method of SIEMER and HAGEMANN (1975). The $\text{Mg}(\text{NO}_3)_2$ dry ashing method used in this study was as follows: 1 g of finely ground plant sample was weighed into a 100 ml beaker and 8 ml of $\text{Mg}(\text{NO}_3)_2$ solution (650 g $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, Merck, product number 5853, in one litre of water) was added. After thorough mixing the mixture was allowed to stand overnight. The suspension was carefully dried on a sand bath at 220 °C. The dry ashing was then performed in a muffle furnace for 6 hours at 500 °C. The residue was moistened with 5 ml of deionized water and dissolved in 5 ml of 12 M HCl and 10 ml of 6 M HCl by heating the solution to boiling point on a hot-plate.

For the dry ashing of rye grass, 150–200 mg (3 ml) of rye grass dried at 50 °C and finely ground, was weighed into five 10 ml glass sample tubes of the Automatic Gamma Sample Counter Wallace GTL 500. The samples were ashed in a muffle furnace (Naber L 47 T) for 16 hours at 450 °C. The temperature was raised to 450 °C over a period of 3 hours. The 265 keV photopeak of ^{75}Se was measured from the samples and the standard tubes using 50 000–100 000 impulses both before and after ashing. The measurement period varied from 40 to 60 minutes. The background was about 45 cpm. The half-life correction was made using four standards.

Dry ashing with $\text{Mg}(\text{NO}_3)_2$ was used to study the effects of ashing time, ashing temperature and volume of $\text{Mg}(\text{NO}_3)_2$ solution on the loss of selenium. The barley containing selenium-75

was obtained from pot experiment number four in the study of PAASIKALLIO (1981). The selenium content of the barley was found to be 0,015 mg/kg d.m. using the hydride method of SIEMER and HAGEMANN (1975). The selenium-75 had been applied to the soil in the pot in the form of sodium selenite.

1,00 g (5 ml) of dried, ground barley shoots were weighed into 10 ml glass sample tubes. The measurement range was 70—190 keV, providing about 700 cpm. Since a minimum of 10 000 impulses were measured, the measuring times at the start of the test were 20—30 minutes. The test involved four parallel tubes. After dry ashing in the muffle furnace the residue was dissolved in hydrochloric acid and all the solution transferred in 5 ml aliquots to four measuring tubes. The beaker was finally rinsed twice with 5 ml of 6 M HCl. This gave six measuring tubes, each of which was measured for 60 minutes. Most of the radioactivity was found in the first four tubes, from which more than 10 000 impulses were measured.

The measurements before and after ashing were compared with four ^{75}Se standards, which had been measured using an average of 50 000 impulses. Because the matrix in the measure-

ment tube had changed during ashing the counting efficiency was investigated in barley and in 6 M HCl solution. For this purpose 1,00 g (5 ml) of barley was moistened with 2,5 ml of 0,01 % NaN_3 in an aqueous solution containing 4 mg/l of Se in the form of sodium selenite and 550 Bq (15 nCi) of ^{75}Se (moistening solution). After careful mixing, the five parallel samples were allowed to dry in 100 ml polythene beakers in the laboratory for about one week. In addition, 2,5 ml of the moistening solution was volatilized in 5 polythene vessels. The aim was to investigate any loss of selenium by volatilization during drying.

After careful mixing the dry barley was returned to the measuring tube and the radioactivity measured. The residue was removed by rinsing the beakers four times with 5 ml of 6 M HCl and transferring the washings to measuring tubes. The results were compared with the moistening solution used in the test diluted with 2,5 ml of 12 M HCl. The samples were measured for 60 minutes, giving more than 30 000 impulses. No loss of selenium was found during drying, and nor were there any significant differences between the counting efficiencies in barley and 6 M HCl.

RESULTS AND DISCUSSION

Loss of selenium during drying

At the 1 % risk level no selenium was lost from the rye grass during drying for four days at 50 °C or at room temperature (Table 1 and Table 3). After four days at 100 °C almost 4 % of the selenium had been released from the rye grass. The results are in accordance with the investigations of EHLING et al. (1968) and WATKINSON (1966). No loss of selenium from some twenty plant species, including alfalfa, red clover, timothy, oats, cabbage and maize were found by EHLING et al. (1968) after drying at 50 °C. In four plant samples drying at 100 °C

caused a 6 % loss of selenium after 18 hours WATKINSON (1966).

When all the samples had reached constant weight after twenty days, those dried at 100 °C and at room temperature had lost 3—4 % of their selenium. It may be that during the slow drying at room temperature some of the selenium in the plant forms volatile selenium compounds. This would explain why very little selenium is released during four days at room temperature, but more is released over a longer period.

When it was found that no more than 3—4 % of the selenium in rye grass was released at 100 °C, and experiment was carried out in which

Table 1. Effect of drying temperature and drying time on selenium loss. Selenium contents given as a percentage of the selenium content of fresh rye grass. Figures not marked with the common letter are significantly different at the 1 % risk level (DUNCAN 1955). Comparison made at each measurement time for the different drying temperatures.

| | 1 d | 4 d | 20 d |
|-----------------|-------------------|-------------------|----------------------|
| 50 °C | 99,2 ^b | 99,0 ^l | (99,0 ^s) |
| 100 °C | 97,5 ^a | 96,3 ^k | (96,1 ^r) |
| room temp. | 99,8 ^b | 99,2 ^l | 96,8 ^r |

five measuring tubes containing rye grass dried for four days at 50 °C were placed in an oven for one day at a time at temperatures of 100, 150 and 200 °C (Table 2). Although the plant material started to show signs of carbonization at 150 °C there was little loss of selenium. The release of selenium was considerably greater at 200 °C, when the rye grass started to carbonize.

Table 2. Loss of selenium at different temperatures. Selenium contents given as a percentage of the selenium content of fresh rye grass. Samples first dried at 50 °C for four days, followed by one day at 100 °C, one day at 150 °C and one day at 200 °C. Figures not marked with the common letter are significantly different at the 1 % risk level (DUNCAN 1955).

| 50 °C | 100 °C | 150 °C | 200 °C |
|-------------------|-------------------|-------------------|-------------------|
| 4 d | +1 d | +1 d | +1 d |
| 99,2 ^c | 96,5 ^b | 95,4 ^b | 79,7 ^a |

Loss of selenium during storage

After drying, there was no significant change in the selenium content during storage for 13 months (Table 3). This rye grass was not ground, it was in contact with the air and was under normal laboratory lighting at a temperature of 20—22 °C.

It is, of course, possible that selenium is retained differently in different plants and parts of plants. Direct sunlight, heat, poor ventilation, excessive humidity etc. may give rise to situations in which 13 % of the selenium can be lost

during storage for 9 months, as reported by GISSEL-NIELSEN (1970). BISBJERG (1972) did not observe any significant differences in the loss of selenium between different plants or for different initial selenium contents.

Table 3. Loss of selenium from plant material during drying and during 400 days' storage. Selenium contents given as a percentage of the selenium content of fresh rye grass. Figures not marked with the common letter are significantly different at the 1 % risk level (DUNCAN 1955). Comparison made at each drying temperature for the different storage times.

| | 1 d | 4 d | 20 d | 170 d | 400 d |
|-----------------|-------------------|-------------------|-------------------|-------------------|-------------------|
| 50 °C | 99,2 ^a | 99,0 ^a | 99,0 ^a | 99,4 ^a | 99,1 ^a |
| 100 °C | 97,5 ^k | 96,3 ^k | 96,0 ^k | 96,6 ^k | 96,5 ^k |
| room temp. | 99,8 ^s | 99,2 ^s | 96,8 ^r | 96,8 ^r | 96,9 ^r |

Loss of selenium during dry ashing

Dry ashing of rye grass for 16 hours at 450 °C caused a loss of (42 ± 2) % of the selenium at the 1 % risk level.

There were no significant differences in the loss of selenium from barley at 500 °C whether dry ashing with $Mg(NO_3)_2$ was carried out for 3, 6 or 18 hours (Table 4, point A). In each case the loss of selenium amounted to 13—15 %. On the other hand, the ashing

Table 4. Effect of (A) ashing time, (B) ashing temperature and (C) volume of $Mg(NO_3)_2$ solution (650 g $Mg(NO_3)_2 \cdot 6H_2O$ in one litre of water) on the loss of selenium during dry ashing with $Mg(NO_3)_2$. The figures show the amounts of selenium lost as percentages of the sample content prior to ashing. Figures in points A, B and C not marked with the common letter are significantly different at the 1 % risk level (DUNCAN 1955).

| | | | |
|---|-------------------|-------------------|--|
| A. Ashing time | | | |
| Ashing temperature 500 °C, 8 ml $Mg(NO_3)_2$ solution | | | |
| 3 h | 6 h | 18 h | |
| 13,0 ^a | 13,3 ^a | 14,5 ^a | |
| B. Ashing temperature | | | |
| Ashing time 6 h, 8 ml $Mg(NO_3)_2$ solution | | | |
| 300 °C | 500 °C | 700 °C | |
| 12,3 ^k | 13,3 ^k | 37,1 ^l | |
| C. Volume of $Mg(NO_3)_2$ solution | | | |
| Ashing temperature 500 °C, ashing time 6 h | | | |
| 4 ml | 8 ml | 16 ml | |
| 25,6 ^s | 13,3 ^r | 11,5 ^r | |

temperature should not exceed 500 °C, since at 700 °C more than 37 % of the selenium is released from the barley (point B). The combustion of barley at 300 °C was incomplete and the solution became brown in colour, although the selenium loss was about the same as that at 500 °C. The small amount of Mg(NO₃)₂ solution (4 ml) was not sufficient to oxidize the entire sample, resulting in incomplete combustion and the loss of about a quarter of the selenium content of the barley (point C). On the other hand, an unnecessarily large amount of Mg(NO₃)₂ solution increases the cost of the analysis, the impurity level due to the reagents used and the difficulties involved in dissolving the combustion mixture. In most cases about 10 ml of Mg(NO₃)₂ solution is probably about right. Care must be taken with different ashing conditions, different materials and quantities, and test must be carried out to ensure that the ashing method used causes the minimum loss of selenium.

More selenium was lost in this study than it were reported by REAMER and VEILLON (1981).

They investigated several wet and dry ashing procedures for biological materials for subsequent selenium determinations by hydride generation atomic absorption spectrometry. Samples were spiked with, or endogenously labeled with, ⁷⁵Se to monitor losses during the procedures. Dry ashing at 450 °C in a muffle furnace with Mg(NO₃)₂ as an ashing aid was satisfactory. Recoveries of ⁷⁵Se ranged from 95,4 to 100,5 %. SIEMER and HAGEMANN (1975) and TAM and LACROIX (1979) used a dry ashing procedure with Mg(NO₃)₂ and/or MgO as an ashing aid with no loss of added or endogenous selenium.

This investigation and later analytical work show that as far as selenium analysis is concerned, the optimum conditions for dry ashing plant material are 500 mg of dry, finely ground sample, 10 ml Mg(NO₃)₂ solution (650 g Mg(NO₃)₂ · 6H₂O in one litre of water), an ashing temperature of 500 °C and an ashing time of 6 hours. Under these conditions the selenium loss during ashing is about 13 %.

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SELOSTUS

Seleenin haihtuminen kasviaineksesta kuivatuksen, säilytyksen ja kuivapolton aikana

TOIVO YLÄRANTA

Maatalouden tutkimuskeskus

Seleenin haihtuminen kasviaineksesta kuivatuksen, säilytyksen ja erilaisten kasviaineksen hajotusmenetelmien aikana on ollut tunnettua jo pitkään. Tiedot seleenitappioiden suuruudesta ovat kuitenkin vaihdelleet suurella määrällä.

Tutkimuksessa, jossa käytettiin hyväksi seleenin radioaktiivista isotooppia ^{75}Se , ei raiheinästä neljän vuorokauden kuivatuksessa haihtunut seleeniä $50\text{ }^{\circ}\text{C}$ lämpötilassa. Seleenitappio $100\text{ }^{\circ}\text{C}$:ssa oli noin 4% . Kun kuivatus tapahtui huoneenlämpötilassa $20\text{--}22\text{ }^{\circ}\text{C}$, haihtui seleenistä 20 d aikana runsaat 3% . Kuivatusta raiheinästä ei 13 kuukauden säilytyksessä huoneenlämpötilassa havaittu seleenin haihtumista.

Kun $50\text{ }^{\circ}\text{C}$ lämpötilassa kuivattua raiheinää pidettiin

lämpökaapissa vuorokausi kerrallaan 100 , 150 ja $200\text{ }^{\circ}\text{C}$:ssa, suurenevät seleenitappiot selvästi korkeimmassa lämpötilassa, jossa kasviaineksen hiiltymisen oli alkanut. Raiheinän seleenistä oli $200\text{ }^{\circ}\text{C}$:ssa haihtunut yhteensä 20% .

Viime vuosina on seleenin määritysmenetelmistä hydridimenetelmä yleistynyt Suomessa. Kuivapoltto sopii erinomaisesti kasvinäytteen hajottamiseen hydridimenetelmää varten, kun valitaan oikeat poltto-olosuhteet. Raiheinän kuivapoltoissa $450\text{ }^{\circ}\text{C}$:ssa haihtui 16 h aikana 42% seleenistä. Kun ohran versojen kuivapoltto suoritettiin $500\text{ }^{\circ}\text{C}$:ssa 6 h käyttäen 8 ml $\text{Mg}(\text{NO}_3)_2$ -liuosta (650 g $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ litrassa vettä) 1 g kohti näytettä, olivat seleenitappiot noin 13% .

COMPOSTING OF SEWAGE SLUDGE AND
OTHER WASTES FROM A FOOD PROCESSING PLANT IN FINLAND

HEIKKI KALLIO and ELISA TIKANMÄKI

KALLIO, H. & TIKANMÄKI, E. 1982. Composting of sewage sludge and other wastes from a food processing plant in Finland. *Ann. Agric. Fenn.* 21: 91—102. (Saarioinen Ltd, SF-36420 Sahalahti, Finland.)

The basic physical and chemical parameters for the industrial-scale composting of the by-products of a convenience food factory were determined. The sludge from the effluent treatment plant of the factory, broiler chicken feathers from a slaughterhouse and peat-bound broiler breeder manure were composted together with pine bark.

Changes in the temperature, pH, dry matter, conductivity, nitrogen balance and carbon content were observed in compost windrows built up in the winter, spring, and autumn. The variables were: type of aeration and mixing; covering of the piles; urea addition; and stabilization with CaO.

It is possible to carry out an optimized and controlled composting process in an industrial-scale out-of-doors system throughout the year in Finland.

The only technical limitation was the air temperature when it dropped below $-10\text{ }^{\circ}\text{C}$ during the mixing and piling of the compost. The recorded maximum temperatures of individual piles (24 trial units) ranged from $65\text{ }^{\circ}\text{C}$ to $81\text{ }^{\circ}\text{C}$ ($81\text{ }^{\circ}\text{C}$ to $86\text{ }^{\circ}\text{C}$ when CaO was added). The thermophilic phase of the compost lasted from one to two months. During the first weeks of the process the change in the dry matter of the compost humus material depended particularly on the air temperature and precipitation. During a six-month period the conductivity of the compost humus increased up to between $30\text{ } \text{S} \cdot \text{m}^{-1}$ and $50\text{ } \text{S} \cdot \text{m}^{-1}$ and the pH was stabilized to around neutral (pH 6 to 8) or, with CaO addition, to pH 11. The nitrate nitrogen continued to increase during the whole period. Significant nitrogen losses occurred when CaO was used.

Index words: composting process, food industry wastes, broiler manure, sewage sludge, broiler feathers, pine bark loosener, out-of-door composting.

INTRODUCTION

Composting as a controlled biotechnical process on an industrial scale is becoming common in Finland. The central objective is to stabilize and refine community and industrial waste with

minimum cost into a harmless and usable form. The local conditions and the requirements set for the end-product must always be borne in mind when choosing the procedure. The north-

ern location and the great variation between the different seasons in Finland are the most prominent features concerning outdoor composting.

During the past decade numerous fundamental experiments have been carried out in Finland in order to discover and to test the procedures suited to the Finnish environment. The bark of coniferous trees has been found to be an excellent siccative, carbon source, and loosener for composting (HALLENBERG 1966, ISOMÄKI 1969, HOWARD 1972, VIRKOLA 1972, RAMBERG 1975, SUNDMAN and HUHTA 1977, HUHTA et al. 1978, PAATERO 1978 a, b, 1979, KEMPPAINEN 1979). A stabilized humus product suitable for use as soil has in several cases been the end-product of a relatively short process (ISOMÄKI 1969, 1974, SUNDMAN and HUHTA 1977, KEMPPAINEN 1979, PAATERO 1979); the humus quality depends significantly on the quality of the wastes composted and on the conditions of composting (BAADER et al. 1975, 1976, 1977, SCHUCHARDT 1977, KEMPPAINEN 1979). Poor conditions, from a technical point of view, have hampered the adjustment and control of the process; most often the process has been actively carried out only in the warm half of the year (SUNDMAN and HUHTA 1977, HUHTA et al. 1978, KEMPPAINEN 1979, MÄKELÄ et al. 1981). If the process is to

take place out of doors and throughout the year, the correct timing of raw material servicing and mechanical work phases is vital.

The utilization of by-products and wastes from food production and the prerequisite farming is diverse and unclear. The processed food industry could, however, be a pioneer in this area. Since, from an ecological point of view, the raw material is clean, there is potential for high-quality and usable end-products. At least one domestically manufactured, self-moving composting station has been commercially available; this apparatus can mix, crush and pile wastes, nutrients and carrier substances and, when needed, turn over the piles (NIIRTYMÄKI et al. 1979, PAATERO 1979).

The purpose of this study was to use composting to solve the waste and by-product disposal problems of a food processing plant so that the end-product would be standardized, high-quality humus material. The prerequisites of the system were: Feasible around the year; complete waste service; reasonable processing cost; a useful and valuable end-product safe to use for defined purpose. On the basis of the test series it has been possible to optimize the process as a year-round activity. The essential chemical and physical parameters are reported in this article.

MATERIAL AND METHODS

Raw materials for the compost

Sludge from an effluent treatment plant was used as the basic raw material in the compost. The plant treats the effluents from the Saarioinen Ltd convenience food factory, 61° 28' N, 24° 20' E (HIRN 1980), and the sewage of the rural Saha-lahti community (population 1 000). In 1980 the food processing plant used 11 000 metric tonnes of vegetables and 1 000 metric tonnes of meat. The factory also has a chicken slaughterhouse. The chemical and hygienic cleaning effect of the

effluent treatment plant has been reported earlier (HIRN 1980). The main properties of the sludge are: dry matter 10 to 13 %, pH 6 to 7, conductivity 10 to 17 S · m⁻¹, total nitrogen 0,7 % (fresh weight), phosphorus 0,2 % (f.w.) and potassium 0,075 % (f.w.). During particularly cold conditions (temperature below -10 °C) the sludge stored in basins was covered with a thin layer of peat to prevent freezing.

The wet fresh feathers (dry matter 20 %) from the broiler chicken slaughterhouse were used as such in the compost. Peat-bound manure from

the two broiler breeding houses was the third industrial waste.

Pine bark (*Pinus silvestris* L.) was recovered from saw mills. The bark was crushed into pieces not longer than 5 cm. The moisture of the bark was about 70 % and the density 450 kg · m⁻³.

Urea, limestone, calcium oxide, potassium sulphate, copper sulphate and fertilizing borate were commercial products.

Process

The composting process took place outdoors throughout the year. The test piles were prepared during three seasons: winter (between January 21 and February 14, 1980); spring (between May 5 and 14, 1980); and autumn (between September 1 and 3, 1980). The compost windrows, 1,8 m high, 3,2 m wide, and 10 m long, were built up loosely by mixing the pine bark and the waste mixture of sludge, feathers and manure in a special mixing and piling station (NIRTTYMÄKI

et al. 1979, PAATERO 1979) on an asphalt surface. After mixing, additional fertilizers and limestone were added before piling. The total number of trial units was 11 with two or three replicates each.

The two process variables in the winter (C₁) and spring (C₂) were (i) aeration of piles (B₁ = turning the piles mechanically with a front loader, or B₂ = aerating the piles for half an hour by blowing air through a pipe, diameter 30 cm with 1 cm slits, installed under the piles), and (ii) protection of piles against rain and snow (A₁ = with no cover, or A₂ = with cover, air space above the piles about 0,5 m). Thus, there were four trial units in the winter and in the spring, with two replicates each. All formulae were identical (formula 1). In the autumn the variables were three different formulae (2, 3 and 4) with neither cover nor aerating pipe and with two or three replicates.

The design of the composting procedure is shown in Table 1 and that of the waste treatment system in Figure 1.

Table 1. Scheme of the composting procedure.

| Process week | Type of aeration | | | | | | | | | | | | | | | | | | | | | | | | | | | |
|--------------|--------------------------------|----------|---------------------------|-----------|-----------------------------|----------|----------------|---------|--------------------------|---------|----------------|---------|----------------|---------|----------------|---------|--|---------|---------|-----------|---------|---------|---------|-----------|---------|---------|---------|----|
| | Formula 1 | | | | | | | | | | | | | | | | Autumn (C ₃) | | | | | | | | | | | |
| | Winter (C ₁) | | | | | | | | Spring (C ₂) | | | | | | | | Open (A ₁), without pipe (B ₁) | | | | | | | | | | | |
| | Without pipe (B ₁) | | | | With pipe (B ₂) | | | | B ₁ | | | | B ₂ | | | | Formula 2 | | | Formula 3 | | | | Formula 4 | | | | |
| | Open (A ₁) | | Covered (A ₂) | | A ₁ | | A ₂ | | A ₁ | | A ₂ | | A ₁ | | A ₂ | | I | | II | | III | | I | | II | | III | |
| | I | II | I | II | I | II | I | II | I | II | I | II | I | II | I | II | I | II | I | II | I | II | I | II | I | II | I | II |
| (23. 1.) | (13. 2.) | (21. 1.) | (7. 2.) | (4-5. 2.) | (14. 2.) | (21. 1.) | (6. 2.) | (5. 5.) | (14. 5.) | (7. 5.) | (12. 5.) | (5. 5.) | (14. 5.) | (7. 5.) | (12. 5.) | (1. 9.) | (1. 9.) | (2. 9.) | (2. 9.) | (2. 9.) | (3. 9.) | (3. 9.) | (3. 9.) | (3. 9.) | (3. 9.) | (3. 9.) | (3. 9.) | |
| 1 | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — |
| 2 | — | — | — | — | — | — | — | a | a | a | a | b | b | b | b | a | a | a | a | a | a | a | a | a | a | a | a | a |
| 3 | a | — | a | — | — | — | — | a | a | a | c | b | b | b | b | — | — | a | a | a | a | a | a | a | a | a | a | a |
| 4 | — | a | — | a | b | b | b | a | a | a | c | b | b | b | b | — | — | a | a | a | a | a | a | a | a | a | a | a |
| 5 | a | — | a | — | — | — | — | — | — | — | — | — | — | — | — | a | a | — | — | — | — | — | — | — | — | — | — | — |
| 6 | — | a | — | a | b | b | b | a | c | c | a | — | c | c | — | — | — | — | a | a | — | — | — | — | — | — | — | — |
| 7 | a | a | a | — | — | — | — | a | — | — | — | — | a | — | c | — | — | — | — | — | — | — | — | — | — | — | — | — |
| 8 | — | — | — | a | a | a | a | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — |

* date of piling in brackets
a = turning with a front loader
b = aerating by blowing air through a pipe
c = turning and mixing with the mixing and piling station

Formulae

The formula was standardized (formula 1) in all winter and spring piles. The ratio of pine bark to waste base was 2 : 1 (v/v). The waste base consisted of sludge : feather : peat-manure in ratios of 10 : 7 : 4 (v/v), respectively. Limestone ($14,3 \text{ kg/m}^3$), and urea (10 kg/m^3) were added to the piles.

In formula 2 sludge and feathers (10 : 7) were mixed and treated with calcium oxide (6 t of $\text{CaO} + 17 \text{ m}^3$ of waste mixture). The temperature of the heap exceeded $95 \text{ }^\circ\text{C}$. After 24 hours peat-manure (4 m^3) was added to the warm mixture and this final mixture piled with pine bark (1 : 2).

In formula 3 the sludge, feathers and peat-manure (10 : 7 : 4, respectively) were mixed (the waste base), the pine bark and the base were mixed (2 : 1), and limestone, potassium sulphate, fertilizing borate, and copper sulphate (12 kg, 2 kg, 40 g and 80 g/m^3 , respectively) were added to the mixture, which was then piled.

Formula 4 was identical to formula 3, but 10 kg of urea/ m^3 was added before piling.

Samples

Samples were taken from 50 to 60 cm below the surface at five points around the pile. All five samples (1 l each) were taken with a clean shovel, pooled and mixed in a plastic bag, chilled, and prepared the next day for analysis.

Analyses and measurements

For the conductivity and pH measurements a 0,5 l sample was stirred in 1 l of distilled water. After standing overnight, conductivity and pH were measured (Chemtrix type 70, Chemtrix Inc., Hillsboro, Oregon).

Dry weights of the compost and raw materials were determined by drying a 200-g sample at $105 \text{ }^\circ\text{C}$ for 14 to 16 hours.

The temperature of each compost windrow was measured at a depth of 80 cm from two standard points, during the first weeks every day and later less frequently. Microchanges in the temperature were observed in some piles with a continuously recording thermometer.

The carbon content was analyzed by burning 3 g of the sample in dichromate-sulphuric acid. The excess dichromate was reduced with potassium iodide, and iodine was titrated with sodium thiosulphate. Total nitrogen was determined by the Kjeldahl method. For the analysis of soluble nitrogen a 50-ml sample was extracted with 250 ml of 0,2 N K_2SO_4 for one hour. The soluble nitrogen was reduced with Devarda's alloy to ammonia, distilled, and titrated with 0,1 N HCl. Ammonium nitrogen was determined as the total soluble nitrogen with no reduction. The difference between the amounts of soluble and ammonium nitrogen gave the nitrate content of the samples. The nitrogen analyses were carried out in Viljavuuspalvelu Oy, Helsinki.

RESULTS AND DISCUSSION

Composting procedure

The design of the investigation (Fig. 1) describes an actual industrial-scale composting process at a convenience food processing factory. The schedule of the composting procedure of this study is presented in Table 1. The piling station was a self-contained, mobile, independent unit

with its own diesel engine; it consisted of silos for raw material, a hammer mill, conveyors, and a piling unit. The station was completed with four automatic nutrient rationers. The investigation began in January 1980 and was completed in March 1981. The main wastes (sludge, feathers and manure) were used in the ratios produced by the factory. Only about 5 % of the sludge

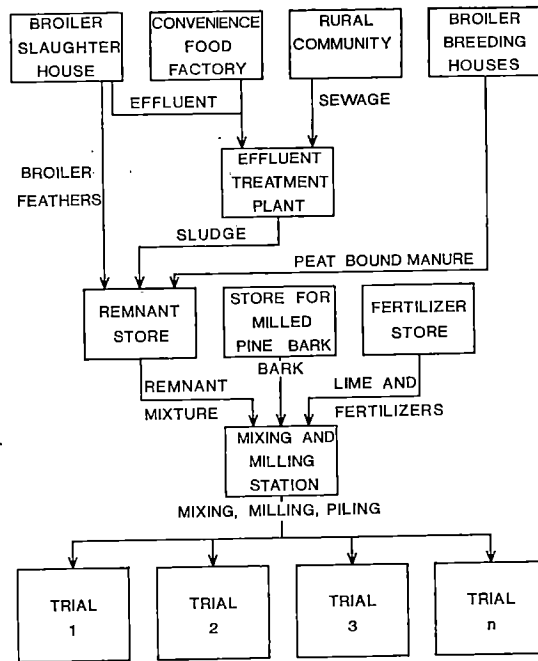


Figure 1. Design of the waste treatment system.

(from communal sewage to the treatment plant), pine bark, lime and nutrients added at the piling stage were brought from outside to the almost enclosed waste system of the factory.

Because of the industrial-scale outdoor process, some variations caused by environmental conditions, such as air temperature, precipitation, irregular aeration schedule of the replicate piles, etc., could not be avoided (cf. PAATERO 1978, KEMPPAINEN 1979, MÄKELÄ et al. 1981). The aim was to standardize the intervals between the aerations to two weeks but exceptions had to be permitted because of technical limitations. Variations in the proportions of the components did not exceed $\pm 10\%$.

It was technically possible to carry out the composting throughout the year, as reported by PAATERO (1979). When the air temperature fell below $-10\text{ }^{\circ}\text{C}$ the use of the piling station was seen to be technically unsafe. This was the only weather-induced obstacle during the year of this experiment. Because of the heavy front

loader and the mobile piling station an asphalted composting area was needed. The public health requirements of the region also emphatically warranted the covering of the area.

Temperature of the windrows

The temperature profiles of the winter, spring and autumn windrows are summarized in Figures 2 A, B, and C, respectively. In the winter and spring the covering did not influence the change in temperature, because there was slight precipitation during the first six-month period (Fig. 4). For this reason the covered and uncovered piles in Figures 2 A and B have not been distinguished as separate curves. The results from the winter and spring windrows have been divided into two temperature profiles according to the aeration systems (mechanical turning or piped aeration). In the autumn piles only mechanical turning (Fig. 2 C) was used on the uncovered piles.

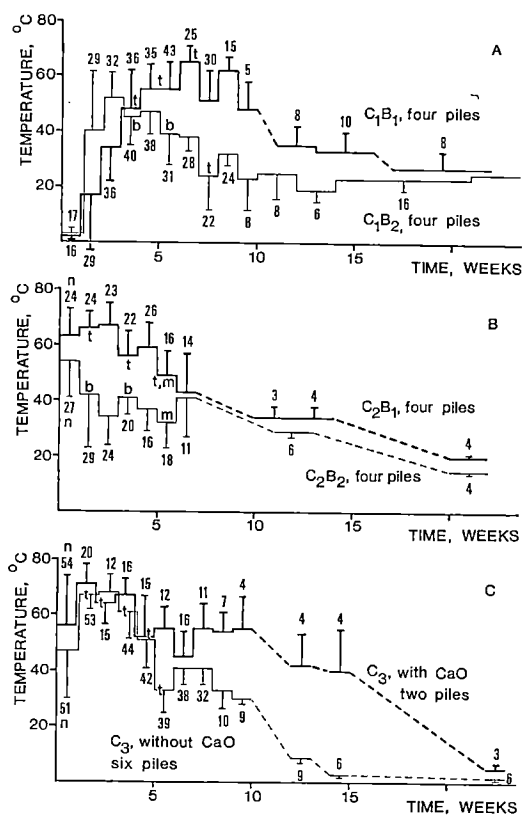


Figure 2. Temperature profiles of the windrows set up in the winter (A), spring (B), and autumn (C). C_1B_1 = winter, mechanically turned, C_1B_2 = winter, pipe aerated, C_2B_1 = spring, mechanically turned, C_2B_2 = spring, pipe aerated, C_3 = autumn, mechanically turned.

t = turning with a front loader

b = aerating by blowing air through a pipe under the heap

m = turning and mixing with the mixing and piling station

In Figure 2 each step in the curve represents the mean of the measurements from a certain period (in the beginning at one-week intervals) and from several windrows. The typical, relatively large standard deviations in the temperature curves are caused by the 'interval' calculations and by the industrial-scale arrangements.

Both in the winter and in the spring there was a very clear difference between the pipe aerated and mechanically turned piles. The mechanically turned piles clearly reached higher temperatures. This shows that the cooling effect of the piped air was greater than the heat production by

aerobic microbes. Blowing was usually followed by an immediate reduction in the temperature. The corresponding mechanical turning induced a rise in the temperature during the following week. In the latter system, the re-orientation of the particles in raw compost also promoted the thermophilic microbial activity. Demand for oxygen decreases in the thermophilic region (SNELL 1957, ARDITTI 1967, SCHUCHARDT 1977), which is defined as beginning at 45–50 °C (NIESE 1969). The rate of respiration also depends on the composition of the mixture and the stage of the composting process (SNELL 1957, SCHULZE 1960, MOTZ 1971, JERIS and REGAN 1973 a, b, c).

The cold weather effectively retarded the development of mesophilic and thermophilic stages in the winter but did not, however, prevent the warming. The winter piles with pipes warmed up faster than the others (cf. KEMPPAINEN 1979). The pipe ends were closed after piling but the air available under the heap accelerated the composting process.

Calcium oxide was added in formula 2 for two reasons: its direct effect on the mixture temperature and its effect on the pH of the piles. Figure 2 C shows that the maximum pile temperatures were reached faster in those with CaO. Even more clear was the indirect effect on the change in temperature in the latter mesophilic phase (i.e. weeks 10 to 20).

The maximum temperatures measured in the individual piles are shown in Table 2. Aerating by turning also promoted higher peak temperatures than the piped air. This is analogous with Figure 2.

The lowest maximum temperature (65 °C) among all the 24 piles was recorded in a pipe-aerated winter pile ($A_2B_2C_1$, II), and the highest (81 °C, excluding CaO piles), surprisingly, in a mechanically turned winter pile ($A_2B_1C_1$, II). The difference between the means of the three seasons was only a few degrees, with the lowest temperature in the winter system. An example of the microchanges in the temperature profile

Table 2. Maximum temperatures of the trial members.

| Pile group | Mean air temp. on the piling date, °C | Mean of the maximum pile temperatures, °C (n) | Highest maximum temp. of the piles, °C (pile code) | Lowest maximum temp. of the piles, °C (pile code) |
|--|---------------------------------------|---|--|--|
| Winter, mechanically turned (C ₁ B ₁) ... | -10 | 73,5±5,5 (4) | 81 (A ₂ B ₁ C ₁ II) | 69 (A ₁ B ₁ C ₁ II) |
| Winter, pipe aerated (C ₁ B ₂) ... | -8 | 67,8±2,6 (4) | 70 (A ₂ B ₂ C ₁ I and A ₂ B ₂ C ₁ I) | 65 (A ₂ B ₂ C ₁ II) |
| Spring, mechanically turned (C ₂ B ₁) ... | +13 | 77,0±1,8 (4) | 79 (A ₁ B ₁ C ₂ I) | 75 (A ₂ B ₁ C ₂ II) |
| Spring, pipe aerated (C ₂ B ₂) ... | +10 | 69,5±2,9 (4) | 73 (A ₂ B ₂ C ₂ II) | 66 (A ₁ B ₂ C ₂ II) |
| Autumn, mechanically turned (C ₃ B ₁), with CaO ... | +13 | 83,5 (2) | 86 (A ₁ B ₁ C ₃ recipe 2, II) | 81 (A ₁ B ₁ C ₃ recipe 2, I) |
| Autumn, mechanically turned (C ₃ B ₁) ... | +12 | 75,5±1,9 (6) | 79 (A ₁ B ₁ C ₃ recipe 3, I) | 74 (A ₁ B ₁ C ₃ recipe 3, III and recipe 4, II) |

of the piles is shown in Figure 3. The inlayed temperature recorder had to be reloaded once a week (denoted by 'a' on the curve). The operation caused local cooling but also aeration and mixing of material around the thermometer. This slightly increased the temperature in the thermophilic range of the windrow (e.g. September 10) but decreased it later on (e.g. September 26). Aeration by turning (denoted by 'b' in Fig. 3) naturally had a greater effect on the whole system (September 17 and 30). These 'disturbances' show the very sensitive and easily broken biological equilibrium in each stage of

the process. The recorder for Figure 3 was not in the same place in the windrow as when the measurements for Figure 2 and Table 2 were made. In all test piles the thermophilic period lasted from about one to two months; this is important with respect to the hygienization of the product.

Dry matter content, pH, and conductivity

The dry matter content of the mixtures was very low after preparing the compost (30 to 40 %), mainly because of the wet sludge and feathers. Acceleration of warming was, however, very effective because of the quality of the raw materials used and the large volume of air space in the piles. High moisture *per se* is not a limiting factor for thermophilic combustion (ERIKSSON 1972). A very typical and logical difference between the piles in each season was the change in their dry matter content (Fig. 5). In the winter the cool surface functioned as a return cooler and very effectively prevented the evaporation of moisture. The slight precipitation (Fig. 4) also explains the very small standard deviations in dry matter in the winter piles (Fig. 5 A), although half of them were covered. In June to July the

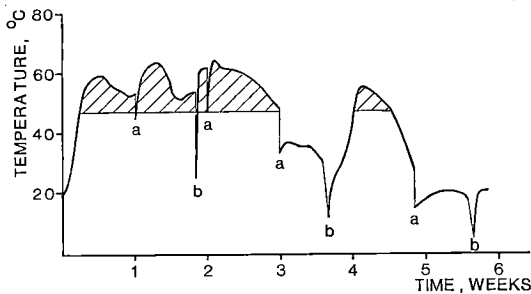


Figure 3. Temperature profile of an uncovered autumn pile (A₁B₁C₃, formula 3, II), recorded with a continuously recording thermometer. Shaded area shows the thermophilic period of the windrow.

a = loading of the thermometer recorder
 b = turning of the pile

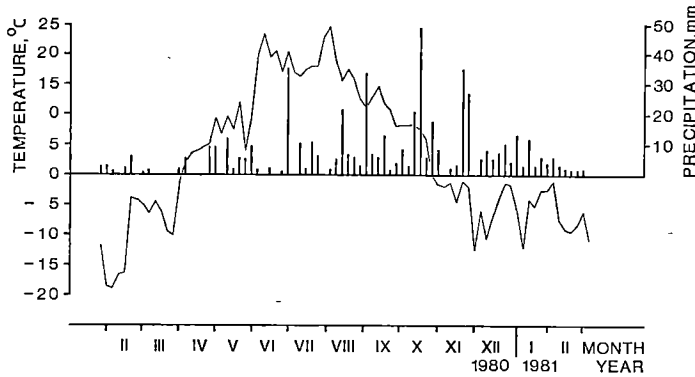


Figure 4. Temperature and precipitation (five-day periods) at Saariöinen during the experimental period (January 21, 1980, to March 3, 1981).

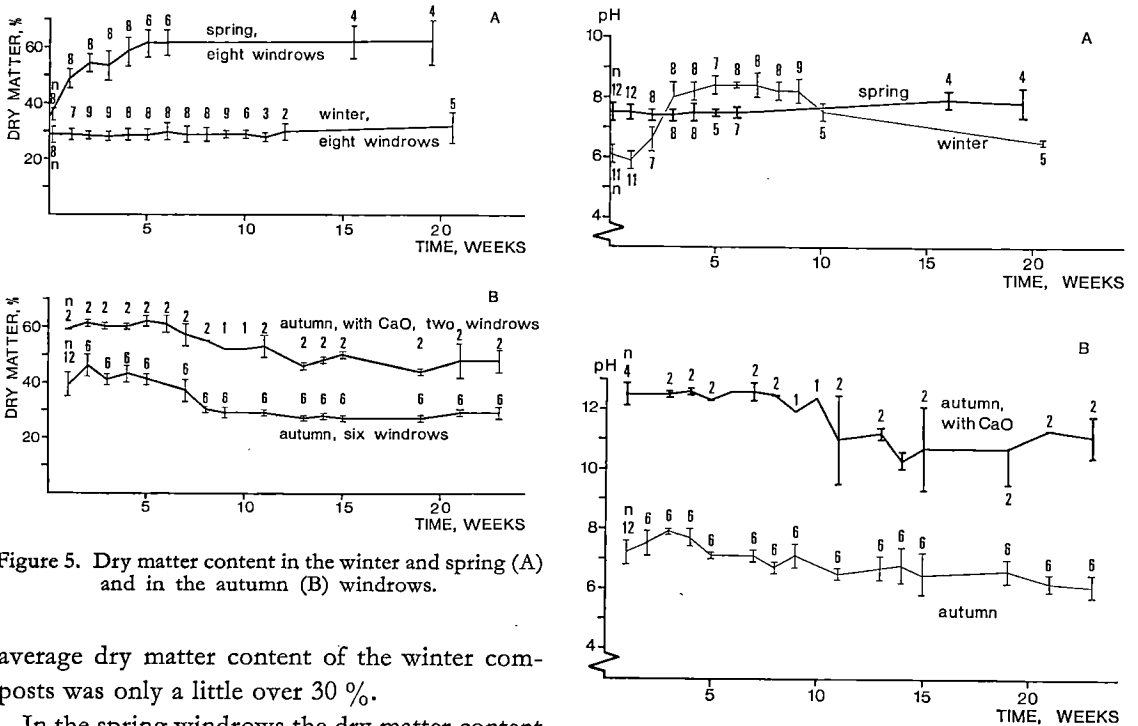


Figure 5. Dry matter content in the winter and spring (A) and in the autumn (B) windrows.

average dry matter content of the winter composts was only a little over 30 %.

In the spring windrows the dry matter content rapidly exceeded 60 % because of low precipitation, warm weather, and effective acceleration of warming in all the trial units. The extremely high precipitation in the summer and autumn months did not moisten the composts once they had become dry. Dry matter in the autumn windrows rapidly exceeded 40 %, but abundant and almost permanent rainfall in the autumn moistened the

Figure 6. pH of winter and spring (A) and autumn (B) windrows.

piles again (Fig. 5 B). Adding CaO to the mixture stabilized the dry matter to about 20 percentage units more than was the case with no added CaO. Thus the varying dry matter content of the final product was one of the clearest differences caused by the weather conditions.

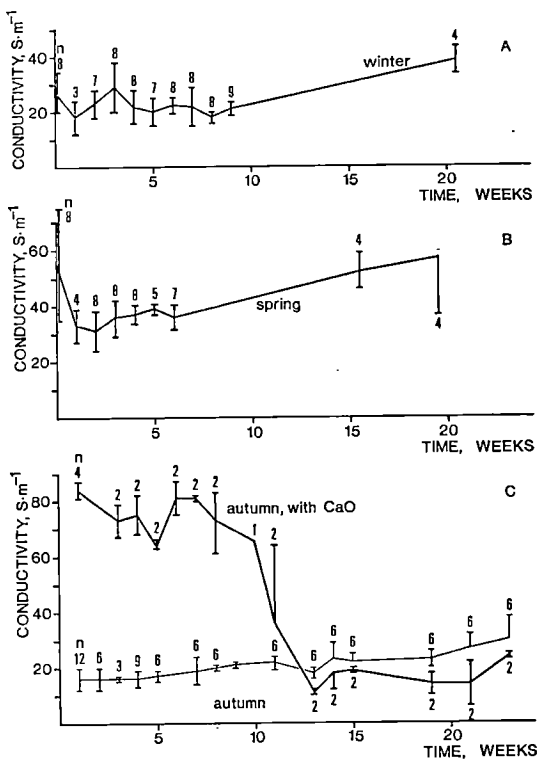


Figure 7. Conductivity of winter (A), spring (B), and autumn (C) windrows.

The pH of piles (Fig. 6) is indirectly dependent on the ambient temperature. In the winter, ammonia was 'raining back' with the condensing water (Fig. 6 A) and a plateau maximum was recorded from weeks three to nine; it was 2,5 pH units higher than after the first week. In the spring and autumn no pH maxima were observed. After five months of composting the pH was usually around neutral (pH 6 to 8). CaO stabilized the pH for the first weeks at pH 12 to 13 (Fig. 6 C), after which a slight decrease was observed. Standard deviations of conductivity were mostly very large (Fig. 7). Usually the conductivity diminished during the first days and rose slowly to a higher level than at the beginning of the experiment (to about 30 to 50 $S \cdot m^{-1}$). In the winter, after three weeks of composting, a low peak was observed; this may indicate accumulation of ammonia. In the autumn the

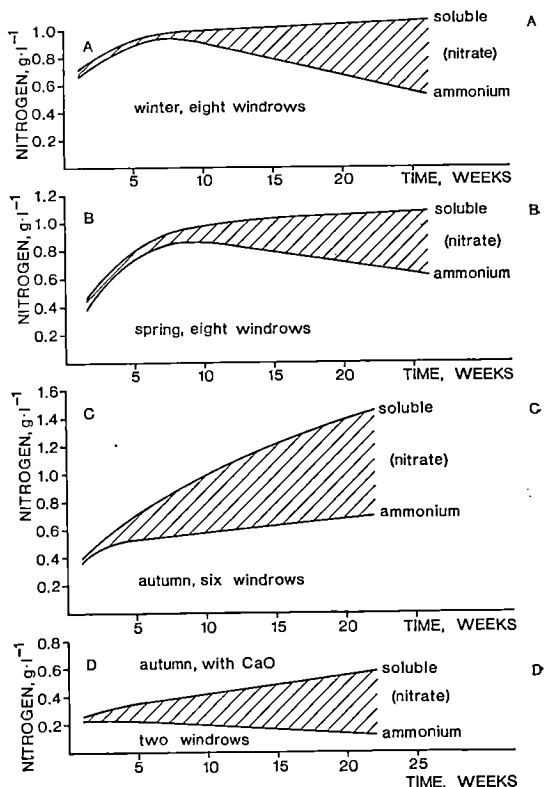


Figure 8. Nitrogen balance in winter (A), spring (B), and autumn (C) and (D) windrows.

conductivity of piles with CaO diminished drastically after the thermophilic phase, in the same way as the pH (Fig. 6 C), which showed binding of the lime. The slower nitrification (Fig. 8) and lower level of soluble nitrogen in the autumn piles with CaO also explain the curve in Figure 6 C.

Nitrogen and carbon content

The total amount of nitrogen in the compost systems (without CaO) ranged from 3,1 to 4,1% (dry weight) during the six-month experimental period. On average, the content decreased somewhat, but the reduction was not statistically significant. In the CaO piles the corresponding proportion was only 1,2%. The change in

soluble nitrogen and ammonium nitrogen content in the three seasons is shown in Figure 8. At first the content of soluble nitrogen was highest in the winter piles (about $700 \text{ mg} \cdot \text{l}^{-1}$). During the experimental period all piles showed effective solubilization process of nitrogen. The most drastic rise was found in the autumn windrows (from 400 to $1\,350 \text{ mg} \cdot \text{l}^{-1}$). The highest content of ammonium nitrogen was observed in the winter piles after a six to seven week reaction time ($973 \text{ mg} \cdot \text{l}^{-1}$). This was due to the cool windrow surface inhibiting evaporation. After six months in each season the level of ammonium nitrogen was rather uniform (500 to $700 \text{ mg} \cdot \text{l}^{-1}$). The CaO piles showed an analogous pattern, but at a much lower level. The content of ammonium nitrogen, in particular, was very low and decreased to about $100 \text{ mg} \cdot \text{l}^{-1}$. The shaded areas in Figure 8 indirectly show the efficiency of the net nitrification process. The ratio of nitrate nitrogen to the total soluble nitrogen increased and reached $49 \pm 14 \%$, $42 \pm 13 \%$, and $54 \pm 23 \%$ in the winter, spring,

and autumn windrows (without CaO), respectively (Fig. 8).

The total carbon content decreased in each series of windrows during the experimental period. The values in the beginning were $46,5 \pm 1,5 \%$, $47,7 \pm 4,0 \%$, and $42,4 \pm 1,5 \%$ of dry weight in the winter, spring and autumn piles, respectively. After six months of composting the corresponding proportions had decreased to $36,8 \pm 0,8 \%$, $43,3 \pm 1,6 \%$, and $38,3 \pm 0,4 \%$. The low ratio of carbon to nitrogen (about 10) showed the addition of urea to be insignificant. In no trial unit was the nitrogen content a limiting factor of the composting process (ALEXANDER 1964).

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SELOSTUS

Suomalaisen elintarviketeollisuuslaitoksen jätevesilietteen ja muiden jätteiden hyötykompostointi

HEIKKI KALLIO ja ELISA TIKANMÄKI

Saarioinen Oy ja Turun Yliopisto

Työssä toteutettiin ja optimoitiin Saarioinen Oy:n Saha-
lahden elintarviketuotantolaitosten jätehuoltoon liittyvä
teollista mittakaavaa oleva kompostointiprosessi. Lähtö-
kohtana oli tärkeimpien biologisesti hajotettavien jätteiden
ja sivutuotteiden kompostointi ohjattuna biotekni-
senä prosessina vakiolaatuiseksi humustuotteeksi ympäri-
vuotisesti ulkona. Teolliset raaka-aineet olivat jäteveden-
puhdistamon aktiiviliete, teurasbroilereiden höyhenet
ja broileremokanojen turvelanta, jotka käytettiin proses-
sissa tuotetussa suhteessa. Kuivikkeena käytettiin murs-
kattua männynkuorta.

Lämpötilan, pH:n, kuiva-aineen, johtoluvun, typpi-
tasapainon ja hiilipitoisuuden kehitystä seurattiin kom-
postoitumisprosessin kuluessa. Kompostiaumat perus-
tettiin talvella, keväällä ja syksyllä. Kokeen muuttajat
olivat ilmastus- ja sekoitustapa, aumojen suojaus sateelta,
urealisäys ja stabilointi sammuttamattomalla kalkilla.

Prosessi on mahdollista toteuttaa kontrolloituna teol-
lisuusmittakaavaisena kompostisysteeminä ulkoilmassa.
Alle -10°C :een lämpötilassa ei ole syytä suorittaa uuden
kompostin sekoitusta ja aumausta konerikkojen riskin
kasvaessa suureksi. Yksittäisten aumojen (24 koejäsentä)
maksimilämpötilat ilman CaO-lisäystä vaihtelivat välillä
 $65-81^{\circ}\text{C}$ (CaO-lisäyksellä $81-86^{\circ}\text{C}$). Auman kuivumi-
seen vaikuttivat erityisesti prosessin ensimmäisten viik-
kojen sateet ja ulkoilman lämpötila. Termofiilinen vaihe
kesti 1-2 kk. Lopputuotteen pH oli neutraalin vaiheilla
(6-8), kun kalkkia ei lisätty, ja johtoluku oli $30-50$
 $\text{S} \cdot \text{m}^{-1}$ noin puolen vuoden kompostoinnin jälkeen.
Nitraattityppi lisääntyi koko koejakson ajan. Sammutta-
mattoman kalkin lisäys aiheutti huomattavia typpitap-
pioita.

VOLATILIZATION AND LEACHING OF SELENIUM ADDED TO SOILS

TOIVO YLÄRANTA

YLÄRANTA, T. 1982. **Volatilization and leaching of selenium added to soils.** Ann. Agric. Fenn. 21: 103—114. (Agric. Res. Centre, Inst. Soil Sci., SF-31600 Jokioinen, Finland.)

No volatilization of selenium was observed during a 96-day laboratory experiment from either clay soil or fine sandy soil to which Se 1 mg/kg had been added as sodium selenite or sodium selenate. Between 2,1 % and 4,6 % of the selenium added to Carex peat was lost by volatilization. Liming and the addition of organic matter to the soil increased the volatilization of selenium to some extent.

Less than 0,2 % of the selenium added in the form of sodium selenite or sodium selenate (Se 100 µg/320 ml soil) was leached through a 20 cm column of either clay soil or fine sandy soil. During a two-month laboratory experiment water corresponding to 500 mm of rainfall was added to the leaching tubes.

More than 7 % of the selenite was leached through the Carex peat. Selenate was leached quite readily from Carex peat, as 84 % of the added selenium was found in the drain water.

Liming had no significant effect on the amounts of selenite or selenate leached through either fine sandy or clay soil, though in the latter soil type it promoted the movement of selenate to lower layers of the soil. In the case of Carex peat, liming drastically reduced the leaching of both selenite and selenate.

Index words: selenium volatilization, selenium leaching, ⁷⁵Se, sodium selenite, sodium selenate, liming, clay soil, fine sandy soil, Carex peat.

INTRODUCTION

The volatilization and leaching of selenium from soil are events in which some of the selenium added to the soil and available to plants is lost. Knowledge of the leaching of selenium is also important in assessing the environmental effects of adding selenium compounds such as selenite or selenate to the soil.

Volatilization of selenium from soil

Both inorganic and organic selenium introduced into the soil undergo various oxidation and reduction reactions, depending on the conditions. Soil micro-organisms play an important role in these reactions. As a result, some of the selenium

added to the soil, e.g. as selenite, is reduced to an oxidation number of zero, while some undergoes methylation, as does mercury, leading to the formation of volatile dimethyl selenide $(\text{CH}_3)_2\text{Se}$, dimethyl diselenide $(\text{CH}_3)_2\text{Se}_2$ and dimethyl selenone, or methyl methylselenite, $(\text{CH}_3)_2\text{SeO}_2$ (REAMER and ZOLLER 1980). The methylation process may be brought about by isolates of *Aspergillus*, *Candida*, *Cephalosporium*, *Corynebacterium*, *Fusarium*, *Penicillium* and undoubtedly other fungi and bacteria (ALEXANDER 1977).

The addition of organic matter to the soil increases the volatilization of selenium (DORAN and ALEXANDER 1976). ABU-ERREISH et al. (1968) found that the amount of volatile selenium increased as the water-soluble selenium fraction of the soil increased.

HAMDY and GISSEL-NIELSEN (1976 a) studied the effect of soil type, addition of organic matter, liming and soil moisture content on the volatilization of selenite added to the soil (Se 40 $\mu\text{g}/\text{kg}$ soil) in a laboratory experiment. The greatest loss of selenium due to volatilization during the four-month experimental period, about 8 %, was from limed sandy soil. The addition of organic matter to the soil can increase selenium volatilization by promoting microbial activity in the soil. DORAN and ALEXANDER (1976) found that subjecting soil to autoclave treatment considerably reduces the formation of dimethyl selenide, while adding organic carbon to the soil also increases the volatilization of selenium as dimethyl selenide. They also stress that the formation of dimethyl selenide, which under aerobic conditions is the main volatile selenium compound, is predominantly a microbiological process. Liming the soil steps up the release of volatile selenium by raising the soil pH and thus the amount of soluble selenium it contains.

Leaching of selenium from soil

Of the various chemical forms of selenium pre-

sent in the soil, selenite and selenate are the important sources of selenium for plants. Selenite binds strongly to the active iron oxides and organic matter present in the soil (HAMDY and GISSEL-NIELSEN 1976 b, 1977, JOHN et al. 1976). The most important sparingly soluble selenites in the soil are $\text{Fe}_2(\text{SeO}_3)_3$ and $\text{Fe}_2(\text{OH})_4\text{SeO}_3$. Their occurrence is determined largely by the oxidation-reduction conditions in the soil, the pH and the concentrations of iron and selenite ions (ALLAWAY et al. 1967, GEERING et al. 1968). In most cases the leaching of selenite from cultivated soils is of no significance. The leaching of selenite is increased by liming but reduced by the addition of organic matter to the soil (GISSEL-NIELSEN and HAMDY 1977).

Selenate and selenite added to soils is converted to elemental selenium. Elemental selenium can be slowly oxidised by microbiological and chemical processes, particularly at high pH. The rate of transformation of SeO_3^{2-} to SeO_4^{2-} is relatively slow (GEERING et al. 1968). In soils selenate can be 10—20 times more soluble than selenite (CARY and GISSEL-NIELSEN 1973).

Selenates are leached from the soil far more easily than selenites (REUTER 1975). BROWN and CARTER (1969) introduced barium selenate (Se 0,6—1,5 mg/kg soil) into the top of 20 cm high soil columns. Water corresponding to 500 mm of rainfall was allowed to flow through the column in five batches over a period of 20 weeks. In the case of loamy sand about 6 % of the added selenium was leached out, while the figure for silt loam was only about half of this.

The results obtained in studies of the volatilization and leaching of selenium added to soil vary greatly, presumably due to factors such as differences in experimental methods. In view of the fact that these studies have usually been restricted to mineral soils, there was a need to study the behaviour of selenite and selenate added to peat soils.

MATERIAL AND METHODS

Clay soil and fine sandy soil were used in the study of the volatilization and leaching of selenium in the laboratory. The particle size distributions of the soils were determined using the pipette method of ELONEN (1971):

| Particle size (ϕ) | Clay soil | Fine sandy soil |
|--------------------------|-----------|-----------------|
| $\leq 0,002$ mm | 62 % | 17 % |
| 0,002—0,02 | 24 | 13 |
| 0,02—0,06 | 9 | 16 |
| 0,06—0,2 | 2 | 37 |
| 0,2—2 | 3 | 17 |

After ignition at 500 °C for 16 hours the Carex peat used in the experiments was found to contain 8,6 % of inorganic matter. The soils used in the experiments were obtained from the plough layer (0—20 cm) of cultivated soil, the clay from Hiekkaharju, Vantaa, the fine sand from Viikki, Helsinki, and the Carex peat from Pelsonsuo, Vaala. The degree of humification of Carex peat was according to VON POST's scale H_5 . Using the modification by TARES and SIPPOLA (1978) of ALTEN's wet combustion method, the clay soil was found to contain 4,0 % organic carbon and the fine sandy soil 2,2 %. The $\text{pH}(\text{CaCl}_2)$ values of the soils were 4,7 for clay, 4,9 for fine sand and 3,9 for Carex peat. The $\text{pH}(\text{CaCl}_2)$ values were determined from soil suspensions (25 ml soil + 62,5 ml 0,01 M CaCl_2 solution).

The soluble selenium was extracted by boiling 25 ml of soil in 100 ml of H_2O for 30 minutes in a 250 ml boiling flask using a test tube with water circulation inserted in the neck of the flask as a condenser. The solution was filtered immediately through a filter paper (Schleicher & Schüll, Selecta 602 h1/2). A 20 ml aliquot of the filtrate was used to determine the selenium content by dry ashing with $\text{Mg}(\text{NO}_3)_2$ followed by the hydride method (SIEMER and HAGEMANN 1975). The extraction removed 6,2 $\mu\text{g}/\text{kg}$ of the selenium from the clay soil, 15,0 $\mu\text{g}/\text{kg}$ from the fine sandy soil and 34,5 $\mu\text{g}/\text{kg}$ from the Carex peat soil.

Volatilization of selenium from soil

Experiments were carried out to study the effect of liming and the addition of organic matter on the volatilization of selenium added to clay soil, fine sandy soil and Carex peat soil in the form of sodium selenite and sodium selenate. The samples were incubated in 10 ml glass measuring tubes of a Wallac GTL 500 Automatic Gamma Sample Counter. The experiment included five replicates. The experimental plan for both selenite and selenate tubes was as follows:

- I Untreated
- II Liming
- III Addition of organic matter
- IV Addition of organic matter and liming

Liming was carried out using analytical grade $\text{Ca}(\text{OH})_2$ powder. The aim of liming was to increase the $\text{pH}(\text{CaCl}_2)$ of the soil by about 1,5 pH units. The amount of lime required was determined by the $\text{Ca}(\text{OH})_2$ titration method (MÄNTYLÄHTI and YLÄRANTA 1980). However, the amount of $\text{Ca}(\text{OH})_2$ powder actually added was 40 % higher than this figure in the light of experience gained in the laboratory with incubation experiments.

Air-dried soil ($\phi \leq 2$ mm) was weighed into tubes to give a volume of about 2,5 ml. The weight of clay and fine sand used was 2,0 g. To the clay was added 600 mg of $\text{Ca}(\text{OH})_2$ powder per 100 g of soil and to the fine sand 240 mg. The weight of Carex peat used per tube was 0,4 g and liming amounted to 1 625 mg of $\text{Ca}(\text{OH})_2$ per 100 g of soil.

Shoots of rye grass (*Lolium multiflorum* Lam.) were used as the organic matter. The rye grass was cut at the silage stage and then ground so that 2/3 of it passed through the 0,2 mm square mesh of bolting cloth, the remainder passing through a 0,6 mm mesh. Kjeldahl determination carried out with the Tecator Digestion System

20L/CU4 according to SILLANPÄÄ (1982) showed the air-dried rye grass to contain 2,4 % nitrogen. Five gram of rye grass was used per 100 g of soil.

One milliliter of either Na_2SeO_3 or Na_2SeO_4 solution was added to the tubes. The solutions contained 740 Bq (20 nCi) of ^{75}Se as either sodium selenite or selenate (The Radiochemical Centre, Amersham, England) and 2 μg of the corresponding inactive selenium compounds. The tubes were measured immediately on a Wallac GTL 500 Automatic Gamma Sample Counter (Scaler/Timer SC—23, Pulse High Analyser AS—12, Programmer PP—67). The detector was a 2×2 " NaI(Tl) well crystal (SCDA—4).

The activity of the tubes was measured again one week after the addition of selenite and selenate, and the tubes were then weighed and the moisture content adjusted to field capacity (pF 2) as determined on a pF device (Soil Moisture Equipment Co., California, USA). De-ionized water was added to the tubes to give a total of 1 000 mg of water in the clay tubes, 700 mg in the fine sand tubes and 1 200 mg in the Carex peat tubes. The tubes were weighed and watered at intervals of just over one week throughout the three-month experimental period. Between measurements the tubes were stored open under room lighting and at room temperature (20—22 °C).

The range 70—190 keV (photopeak 136 keV) was selected for measurement of the radioactivity of the samples. Measurements were also carried out on five standard selenite and five standard selenate tubes containing 740 Bq (20 nCi) of ^{75}Se and 100 mg/l of Se. In order to reduce statistical error, a minimum of 100 000 impulses were measured for each tube. The duration of the measurement varied from four to ten minutes. The background radiation was about 70 counts per minute (cpm). Determinations of radioactivity were performed 7,28 and 96 days the start of the experiment.

In parallel with this experiment, another experiment was carried out to determine the

volatilization of selenium from measuring tubes containing samples of the same soils as in the replicate experiment but only 2 ng Se as sodium selenite or sodium selenate. The aim of using such a small amount of selenium was to reduce any inhibitory action on micro-organisms.

Many of the studies published on the volatilization of selenium from soil have used an air, argon or nitrogen flow into the incubation chamber. The selenium released from the soil and carried away with the gas was then analysed (e.g. ABU-ERREISH et al. 1968, FRANCIS et al. 1974, DORAN and ALEXANDER 1976, and HAMDY and GISSEL-NIELSEN 1976 a). However, in this work it was thought desirable to avoid bringing the soil into contact with an artificial atmosphere.

Leaching of selenium from soil

Laboratory experiments were carried out to determine the leaching of selenite and selenate from clay, fine sand and Carex peat soils both with and without liming.

A total of forty-eight 30 cm high leaching vessels were made for the experiment out of grey PVC tubing, internal diameter 45 mm. Ten saw cuts were made in the tube at 2,5 cm intervals to half the tube diameter. Grey PVC sheet with five 2,5 mm diameter holes drilled in it was glued to the bottom of the tube. The saw cuts were covered with plastic tape and 1 g of white vaseline (Ph. Nord.) was spread on the inside wall of the tube to prevent water leaking out. A disc of polythene bolting (0,2 mm square mesh) was placed at the bottom of the tube. On top of this was placed 40 ml of quartz sand (\varnothing 0,2—0,5 mm) washed in 6 M HCl to form a filter layer 2,5 cm thick. The purpose of the bolting disc was to prevent the sand from blocking the holes in the bottom of the tube.

The tubes were charged with 390 g of fine sand ($\varnothing \leq 2$ mm), 300 g of clay ($\varnothing \leq 2$ mm) or 60 g of Carex peat ($\varnothing \leq 2$ mm). The amounts of analytical grade $\text{Ca}(\text{OH})_2$ powder used as liming

agent were 936 mg for the fine sand tubes, 1 800 mg for the clay tubes and 975 mg for Carex peat tubes. The $\text{Ca}(\text{OH})_2$ powder was mixed thoroughly with the soil in question. Four replicates were prepared for each experimental soil.

The soil was watered with de-ionized water both by capillary action and by adding water to the top of the column. The columns were allowed to stand for three weeks to equilibrate. The fine sand tubes contained an average of 36,3 % water (standard deviation 1,3 %), the clay tubes 60,1 % (1,4 %) and the Carex peat tubes 290 % (20 %).

A hole 15 mm in diameter and 10 mm deep was formed in the top of the soil column using an acrylic plastic rod. One milliliter of an aqueous solution containing 100 μg Se as sodium selenite or selenate labelled with 185 kBq (5 μCi) of ^{75}Se was measured into each of the holes. After the solutions had been absorbed the soils were covered with 40 ml of quartz sand. The leaching tubes were allowed to hang from their stands and 250 ml beakers placed beneath them.

Three days later 80 ml of de-ionized water was added to each tube in two lots. This corresponds to a water column of 50 mm. The tubes and the mouths of the beakers were covered with polythene film to minimize loss of water by evaporation. It took 1—2 days for the 80 ml of water to pass through the fine sand, 2—3 days for it to pass through the clay and two hours for it to pass through the Carex peat. When this was complete, the volume of water in the beaker was measured and a 5 ml sample taken for determination of radioactivity on the Wallac GTL 500 Automatic Gamma Sample Counter. Two lots of 40 ml of de-ionized water added to each tube at weekly intervals, giving a total of

800 ml of water and ten ^{75}Se determinations per tube. The leaching experiment was carried out in the laboratory, where the temperature was 16—19 °C.

The energy range 70—190 keV was chosen for the radioactivity determinations. In addition to the actual samples, determinations were also carried out on four standard selenite and four standard selenate tubes containing 5 ml of solution prepared from the same batch of solution that was added to the leaching tubes. This made it possible, at the time of each determination, to establish how much of the radioactive selenium added to the column had passed through.

Each ^{75}Se sample tube was measured for 35 minutes, during which the number of impulses from the leaching waters from the clay and fine sand averaged only a few thousand. Considerably more radioactive selenium leached through the Carex peat soil than through the two mineral soils. For this reason, the number of impulses measured from the water that had drained through the Carex peat ranged from ten thousand to several million. The background was about 70 cpm.

When the measurements were complete, the 20 cm soil column was divided into eight 2,5 cm sections using the saw cuts made earlier. The ^{75}Se activity was measured from 5 ml soil samples after mixing. The radioactivities of the quartz sand filter beds were also measured in the same way.

Finally, the $\text{pH}(\text{CaCl}_2)$ of the soil column was measured. A soil sample representing the whole column was obtained by mixing the 5 ml samples taken from each column section.

RESULTS

Selenium volatilization

No selenium was lost by volatilization from either fine sand or clay during the entire three-

month experimental period, since the amount of selenium measured in the test tubes did not differ from the amounts of added to the tubes at the start of the experiment at the 1 % risk level.

The highest amounts of selenium lost from *Carex* peat by volatilization (2,1—4,6 %) were from those experiments given both lime and organic matter (Table 1). Supplementing the lime with organic matter considerably increased the volatilization of selenium from *Carex* peat: after 7 days of the experiment, far more selenium had volatilized than from the control. Volatilization of selenium continued throughout the 96-day experimental period.

Table 1. Volatilization of selenite and selenate (Se 5 mg/kg) from *Carex* peat. Figures given for each measurement time at points selenite and selenate not marked with a common letter differ at the 1 % risk level (DUNCAN 1955). Tukey's honestly significant difference (hsd) procedure has been tested between the different measurement times.

| | Selenite | | | Selenate | | |
|---|-------------------------|--------------------|-------------------|-------------------------|-------------------|-------------------|
| | 7d | 28d | 96d | 7d | 28d | 96d |
| Carex peat .. | 99,2 ^b | 100,0 ^h | 99,0 ⁿ | 98,5 ^a | 99,3 ^g | 99,2 ⁱ |
| Ct + Ca(OH) ₂ | 99,0 ^b | 99,8 ^h | 96,3 ^l | 99,0 ^a | 99,4 ^g | 99,1 ⁱ |
| Ct + organic matter | 99,4 ^b | 98,8 ^g | 97,9 ^m | 99,0 ^a | 99,9 ^g | 99,3 ⁱ |
| Ct + organic matter + Ca(OH) ₂ | 97,9 ^a | 97,8 ^f | 95,4 ^k | 99,0 ^a | 97,9 ^f | 96,6 ^k |
| | HSD _{0,01} 1,8 | | | HSD _{0,01} 3,1 | | |

The volatilization of selenium from the soils treated with selenate did not show as clear a pattern as in the case of soils treated with selenite (Table 1). However, there was a 3,4 % selenium volatilization from soils treated with both lime and organic matter.

Reducing the amount of selenium by a factor of 1 000, i.e. from 2 µg/tube to 2 ng/tube, had no noticeable effect on the volatilization of selenium.

Selenium leaching

Leaching of selenite from fine sand and clay was extremely small: less than 0,1 % of the selenium added in the form of selenite was found in the water collected from the columns. In both limed and unlimed soils 96—98 % of the selenium was in the top 2,5 cm of soil, to which

it was originally added. An average of only 0,5 % of the selenium added as selenite penetrated below 10 cm in the soil column.

The amount of selenite leached through the soil columns containing unlimed *Carex* peat averaged 7 %, while that leached through limed *Carex* peat averaged only 1,1 % (Table 2). Most of the selenium was leached through the unlimed columns when 150-250 mm of water had been added. The flow of selenium through the columns was subsequently much slower, amounting to only 0,3 % for the last addition of water, i.e. the tenth 80 ml lot. In the case of the limed soils, each addition of water after the first 100 mm washed only about 0,1 % of the selenium through.

Table 2. Leaching of selenite and selenate added to the soil from a 20 cm soil column expressed as a percentage of the selenium added with the 99 % confidence limits.

| | | pH (CaCl ₂) | Selenite | Selenate |
|-------------------|---------|----------------------------|-------------|-------------|
| Fine sand | Unlimed | 4,9 | 0,07 ± 0,04 | 0,12 ± 0,06 |
| | Limed | 6,0 | 0,08 ± 0,04 | 0,11 ± 0,06 |
| Clay | Unlimed | 4,9 | 0,01 ± 0,03 | 0,06 ± 0,05 |
| | Limed | 6,1 | 0,03 ± 0,02 | 0,17 ± 0,04 |
| <i>Carex</i> peat | Unlimed | 3,9 | 7 ± 4 | 84 ± 12 |
| | Limed | 5,4 | 1,1 ± 0,6 | 3 ± 3 |

The flow of selenate through fine sand and clay was also slow. In columns containing both limed and unlimed soils only 0,1—0,2 % of the selenium originally added flowed through the column (Table 2). However, selenate penetrated much further down the column than did selenite (Fig. 1). In both limed and unlimed fine sand and in unlimed clay 95—99 % of the selenium added in the form of selenate was found in the upper 10 cm of the soil. In unlimed fine sand an average of 65 % of the selenium was found in the top 2,5 cm of the soil layer, while the corresponding figure for limed fine sand was 52 %. In the case of unlimed clay the top 2,5 cm of soil contained an average of 72 % of the added selenium, but the figure for limed clay was only

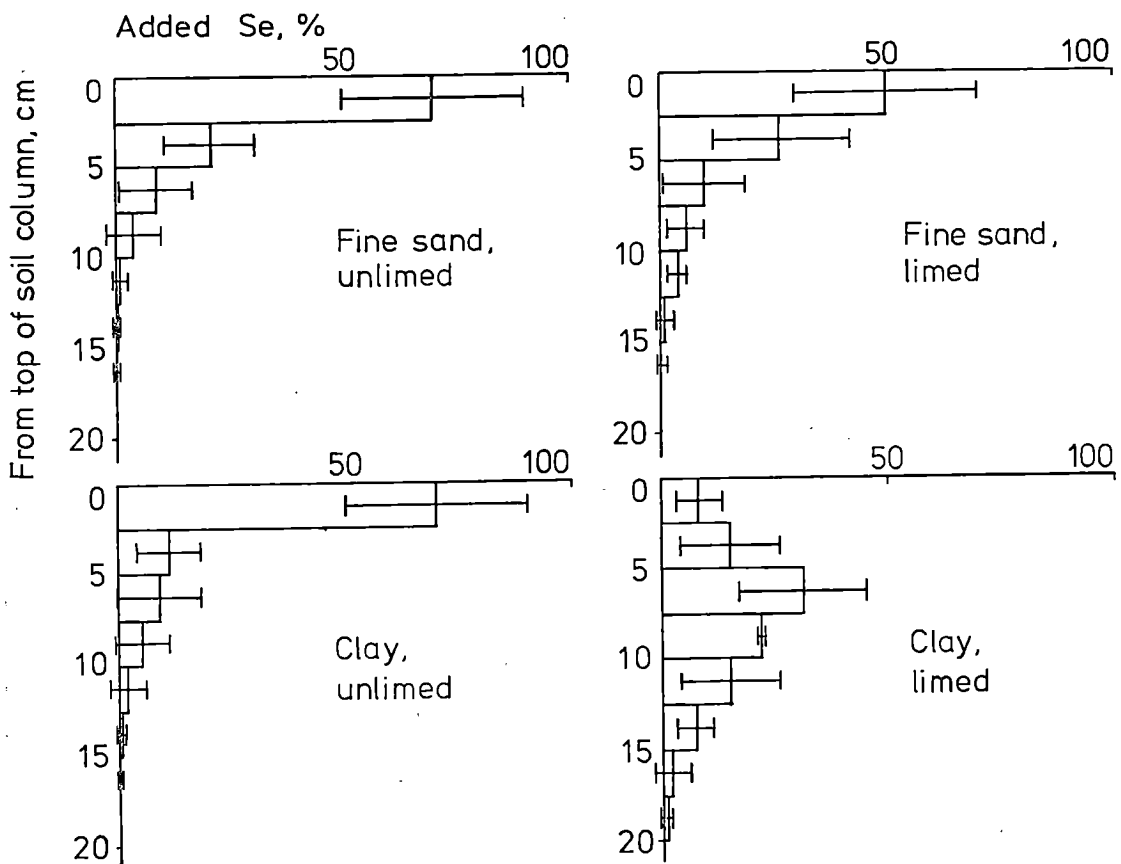


Fig. 1. The distribution of ^{75}Se -labelled selenate in soil columns following leaching with 500 mm H_2O . The amount of radioactive selenium in the various soil layers is presented, together with the 99 % confidence limits, as a percentage of the selenium added to the column at the start of the experiment.

8 %. The selenium added to limed clay in the form of selenate had penetrated much further down the column as shown by the fact that an average of 68 % of the added selenium was found in the 5—12,5 cm soil layer.

An average of 84 % of the selenium added to unlimed Carex peat in the form of selenate passed through the 20 cm soil column, while only 3 % passed through when the Carex peat was treated with lime (Table 2). The third and fourth additions of water, i.e. amounting to the equivalent of 200 mm, washed about 80 % of the added selenium through the unlimed columns, though each subsequent addition of water washed only an average of 0,1 % through. The selenium peak

for limed Carex peat was found to occur at the same water additions, though the highest through-flow values were only 1 % of the added selenium. The average flow of selenium through limed Carex peat was thereafter the same as for unlimed peat, i.e. about 0,1 % for each addition of water.

In the unlimed Carex peat columns 1,1—3,3 % of the selenate added was found in the top 2,5 cm of soil layer. In the limed columns most of the selenium (total 60 %) was found in the 5—10 cm soil layer (Fig. 2).

The quartz sand filter beds contained no notable amounts of ^{75}Se .

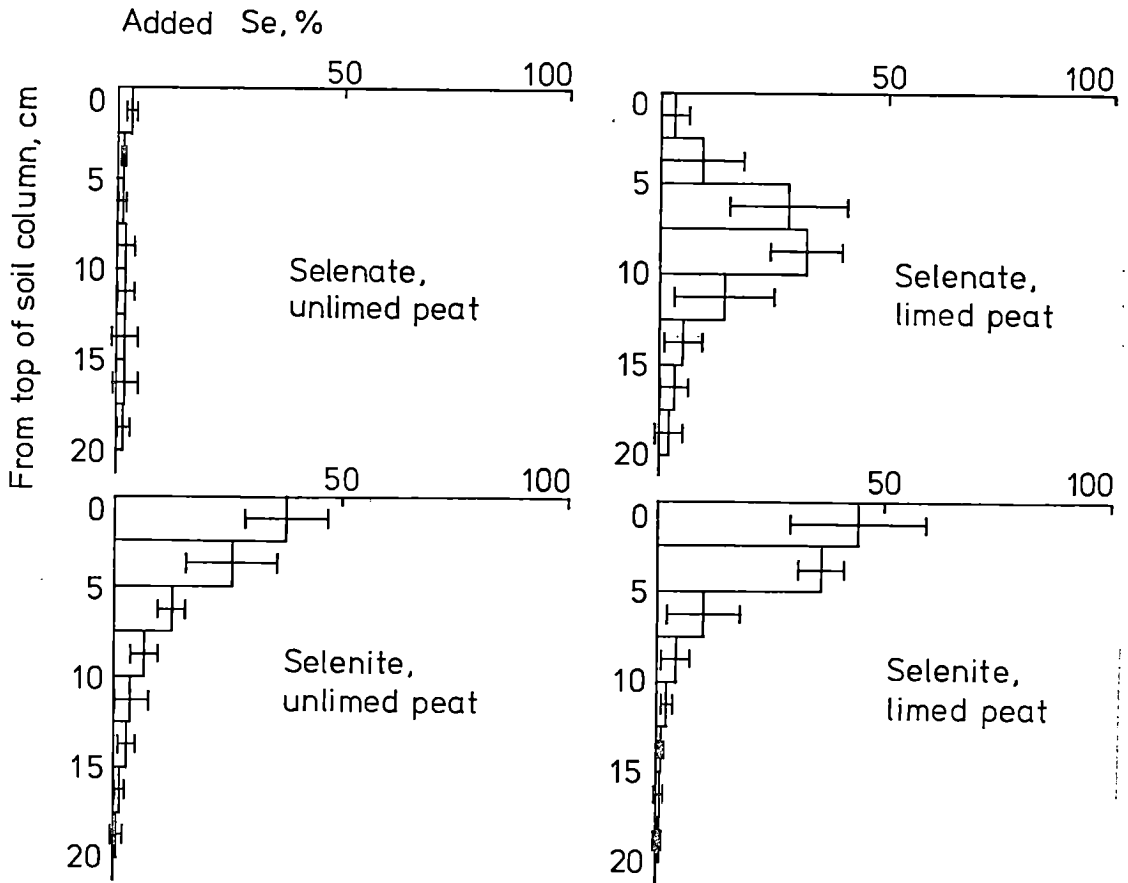


Fig. 2. The distribution of ^{75}Se -labelled selenate and selenite in *Carex* peat columns following leaching with 500 mm H_2O . The amount of radioactive selenium in the various soil layers is presented, together with the 99 % confidence limits, as a percentage of the selenium added to the column at the start of the experiment.

DISCUSSION

Volatilization of selenium from soil

No volatilization of either selenite or selenate from fine sand or clay was found in this experiment. FRANCIS et al. (1974) and DORAN and ALEXANDER (1976) also reported very low volatilization of selenium from mineral soils treated with selenite. FRANCIS et al. (1974) found that 0.3–2 % of selenium added as selenite volatilized in the form of dimethyl selenide during a 48-day laboratory experiment.

DORAN and ALEXANDER (1976) and HAMDY and GISSEL-NIELSEN (1976 a) have both reported that the addition of lime and organic matter increases volatilization of selenium from soil. This was confirmed in the present study, since the addition of $\text{Ca}(\text{OH})_2$ and ground rye grass to *Carex* peat caused an increase in the volatilization of selenium added both as selenite and as selenate.

According to HAMDY and GISSEL-NIELSEN (1976 a) it is clear that fluctuations in the moisture content of the soil also have a profound

effect on the volatilization of selenium. Alternate drying and wetting of the soil causes a greater release of selenium. Thus the volatilization of selenium under field conditions, where changes in the soil's moisture content may be extremely large, is presumably greater than under laboratory conditions.

In this study the 50 mg of water which evaporated from the tubes each day was made up at intervals of slightly more than a week. The moisture content of fine sand varied between 35 % and 8 %, that of clay between 50 % and 23 % and that of Carex peat between 300 % and 160 %.

The volatilization of selenite and selenate from soil is probably small. However, in laboratory experiments conducted by OLSON et al. (1976) as much as 30 % of the selenium added to fine sand in the form of trimethylselenonium chloride (Se 2,5 mg/kg) volatilized from the soil during 42 days (the trimethylselenonium ion is an important urinary metabolite of dietary Se).

Leaching of selenium from soil

Very little selenite was leached through either fine sand or clay. Only 0,1—0,2 % of the selenium added as selenite passed through the soil column. In Denmark the leaching of selenite from mineral soils has been found to be low in both lysimetric studies (GISSEL-NIELSEN 1976) and field studies (GISSEL-NIELSEN and BISBJERG 1970). Using potassium selenite as a top dressing (Se 0,5 mg/kg soil), GISSEL-NIELSEN and BISBJERG (1970) compared the leaching of the added selenite with that of the soil's native selenium. Soil samples taken from a depth of 0—75 cm were analysed for total selenium from plots with and without added selenium. The results showed no significant differences in the selenium content of the soil at a depth of 25—75 cm, even after an experimental period lasting more than one year.

In this study, 800 ml of water, corresponding

to a water column of 500 mm, was introduced into the tops of the soil columns during the three-month experimental period. Over 80 % of the water ran through the column, while the remainder evaporated. Even this large volume of water failed to leach more than 0,1—0,2 % of the added selenite and selenate through the 20 cm soil column. However, the selenate penetrated much further down the column than the selenite.

Selenate leached quite easily through Carex peat, since 84 % of the added selenium was found in the water passed through the column. Most of this selenium was found in the first lots of water collected. Although the amount of selenite leached through the Carex peat soil was only one-tenth that of selenate: the value 7,3 % is high compared with the values for fine sand and clay.

GISSEL-NIELSEN and HAMDY (1977) found that liming with CaCO_3 increased the leaching of selenium in two Danish mineral soils containing 4,1 % and 15,8 % clay, respectively. In the present study, liming had no significant effect on the leaching of either selenite or selenate through the tubes containing either fine sand or clay. In the case of Carex peat, liming reduced the amounts of selenium passing through the soil columns following application of either selenite or selenate (1 % risk level). The pH measurement made at the end of the experiment showed that the addition of lime had caused the pH of Carex peat to rise from its initial value of 3,9 to 5,4 (Table 2).

The marked decrease in the leaching of selenite and selenate from Carex peat as a result of liming with Ca(OH)_2 is probably not due, at least in the case of selenate, to the poor solubility of calcium selenate since it has a solubility of 79 g per litre of cold water (WEAST and ASTLE 1981). The application of lime to Carex peat caused changes which reduce the leaching of selenium and which have not been explained in this study.

On the other hand, it was rather surprising to find that liming caused a more marked leaching

of selenate into the lower soil layers of clay than in fine sand. It is not clear whether, during the three-week equilibration period used for the soil columns prior to the addition of selenium, structural changes took place in the clay columns that later promoted the leaching of selenate.

This laboratory experiment confirmed the results presented previously in the literature concerning the low leaching of selenite from mineral soils. However, the leaching of selenite from Carex peat cannot be considered insignificant.

Selenate is rapidly leached from Carex peat, even by a small volume of water. This must be taken into account in assessing the environ-

mental effects of selenium even if only a small proportion of the selenium added either to the soil or to plants is taken up each year by the crop. The leaching of selenium from certain mineral soils may also be significant, despite the fact that in this study even the rather high volume of water, corresponding to 500 mm of rainfall, failed to leach more than 0,1–0,2 % of the selenium added to fine sand or clay through the 20 cm soil column. Nevertheless, the selenate had started to penetrate down the soil column.

It was of interest that the fairly large amount of lime used caused a marked decrease in the leaching of selenite and selenate from Carex peat.

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SELOSTUS

Maahan lisätyn seleenin haihtuminen ja huuhtoutuminen

TOIVO YLÄRANTA

Maatalouden tutkimuskeskus

Maahan lisäystä kasveille käyttökelpoisesta seleenistä osa voi haihtua tai huuhtoutua. Seleenin huuhtoutumisen tuntemisella on tärkeä merkitys myös mahdollisten ympäristövaikutusten kannalta.

Laboratoriossa suoritettussa 96 vuorokauden pituisessa muhityskokeessa ei savimaasta eikä hietamaasta havaittu seleenin haihtumista, kun maahan lisättiin 1 mg/kg seleeniä natriumseleniittinä tai natriumselenaattina. Saraturpeesta haihtui seleeniä 2,1—4,6 %. Orgaanisen aineksen lisääminen ja kalkitus tehostivat jossain määrin seleenin haihtumista.

Natriumseleniittinä tai natriumselenaattina maahan lisäystä seleenistä (Se 100 µg/320 ml maata) alle 0,2 %

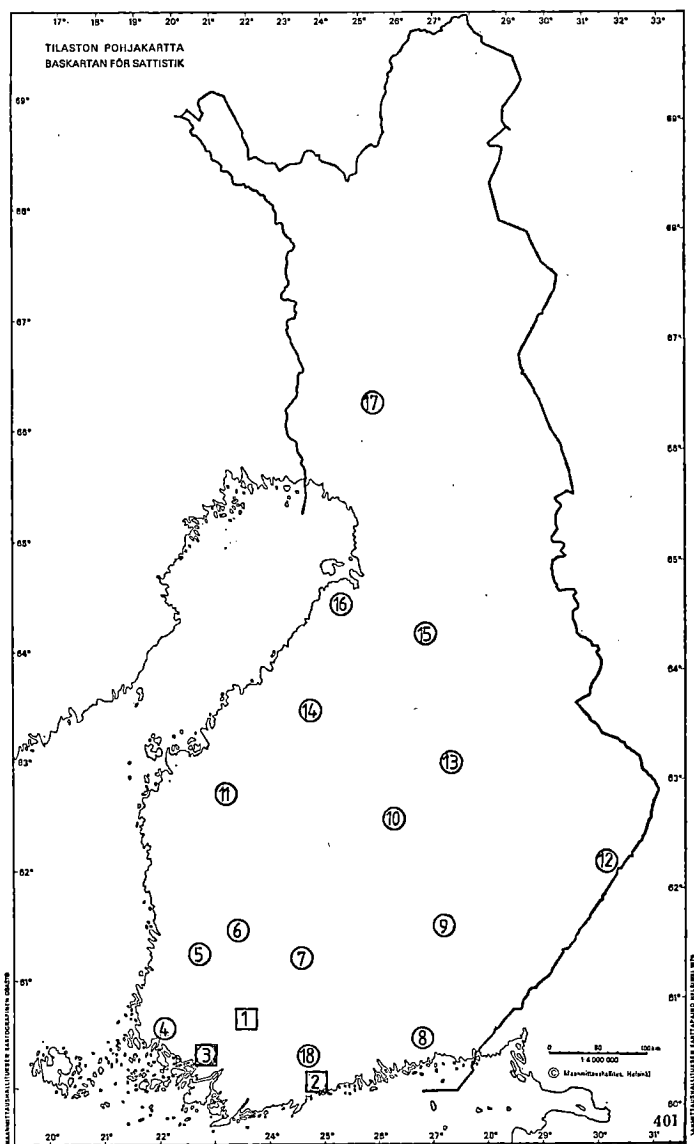
huuhtoutui 20 cm korkuisten savi- ja hietapylväiden lävitse. Laboratoriokokeessa lisättiin kahden kuukauden aikana huuhtoutumisputkiin 500 mm sademäärää vastaava vesimäärä.

Saraturpeen lävitse huuhtoutui seleniittiä runsaat 7 %. Selenaatti huuhtoutui varsin helposti saraturpeessa, sillä 84 % lisäystä seleenistä mitattiin valumavesistä.

Kalkitus ei vaikuttanut merkittävästi hieta- ja savi-putkien lävitse huuhtoutuneen seleniitin tai selenaatin määriin, mutta se lisäsi savimaassa selenaatin kulkeutumista alempiin maakerroksiin. Saraturpeessa vähensi kalkitus voimakkaasti sekä seleniitin että selenaatin huuhtoutumista.

CONTENTS

| | |
|---|-----|
| ETTALA, E., RISSANEN, H., VIRTANEN, E., HUIDA, L. & KIVINIEMI, J. Wilted and unwilted silage in the feeding of dairy cattle | 67 |
| YLÄRANTA, T. Loss of selenium from plant material during drying, storage and dry ashing | 84 |
| KALLIO, H. & TIKANMÄKI, E. Composting of sewage sludge and other wastes of a food processing plant in Finland | 91 |
| YLÄRANTA, T. Volatilization and leaching of selenium added to soils | 103 |



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SISÄLLYS — CONTENTS

| | |
|---|-----|
| ETTALA, E., RISSANEN, H., VIRTANEN, E., HUIDA, L. & KIVINIEMI, J. Wilted and unwilted silage in the feeding of dairy cattle | 67 |
| Selostus: Esikuivatun ja tuoreen säilörehun vertailu lypsykarjan ruokinnassa | 83 |
| YLÄRANTA, T. Loss of selenium from plant material during drying, storage and dry ashing | 84 |
| Selostus: Seleenin haihtuminen kasviaineksesta kuivatuksen, säilytyksen ja kuivapolton aikana | 90 |
| KALLIO, H. & TIKANMÄKI, E. Composting of sewage sludge and other wastes of a food processing plant in Finland | 91 |
| Selostus: Suomalaisen elintarviketeollisuuslaitoksen jätevesilietteen ja muiden jätteiden hyötykompostointi | 102 |
| YLÄRANTA, T. Volatilization and leaching of selenium added to soils | 103 |
| Selostus: Maahan lisätyn seleenin haihtuminen ja huuhtoutuminen | 113 |