An evaluation of the SMP manufacturing chain at peak capacity; where minimum chlorine is employed for CIP from chemical residue & microbiological quality perspectives

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Introduction

- There is increasing demand for dairy products with low levels of chlorate from international customers and these demands must not only be met, but exceeded if Ireland is to remain competitive in the international dairy market.

- In response to market demands regarding chlorate levels in dairy products chlorine use in CIP across the dairy industry was prohibited from January 1st 2021.

- This decision is coupled with the initiative taken by many dairy processors to utilise chlorine gas for on-site water treatment as an alternative to chlorinated water.

- The success of these strategies depends on;
  - **The chlorate and perchlorate residue levels of dairy products produced in these "minimum chlorine scenarios".**
  - **The bacterial quality of the dairy products produced in these "minimum chlorine scenarios".**
  - **Whether levels are within product specification?**
  - **Whether these levels compare favourably to those found when chlorine was still employed (Pauldetti et al., 2019a; 2019b; Li et al., 2019).**

Study of the SMP Manufacturing Chain

Objectives

1. Evaluate the quality of SMP across its manufacturing chain in a minimum chlorine environment (CF detergents/ sanitizers & chlorine gas treated water).
2. Establish baseline data on chlorate/ perchlorate occurrence in Irish SMP which can be utilised by DAFM when contributing to EU MRL updates.

How?

Track a single SMP run from WMS to SMP and test samples for chemical residue levels & microbiological counts (also study their microbiomes).

What is a "single SMP run"?

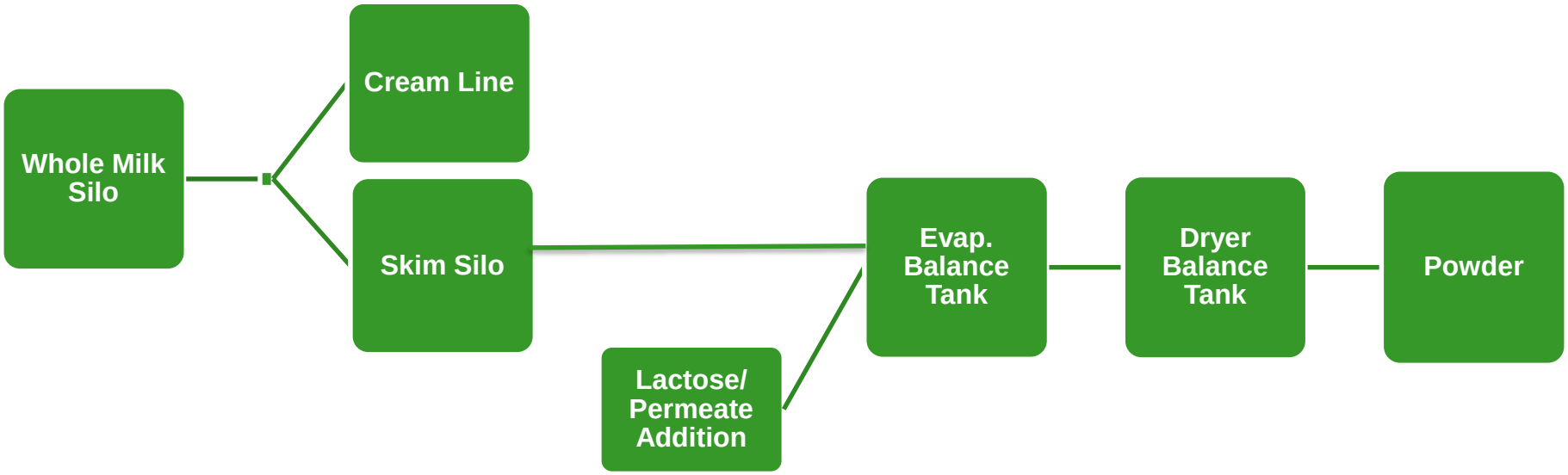
The amount of time that it takes to manufacture the contents of one batch of whole milk (e.g. 1 x WMS) into SMP.

Sampling Conducted

Sampling and analyses was conducted across the peak milk production months of April, May & June;

- A total of 11 "SMP runs" were sampled and analysed.
- 3 x milk processing sites situated in different geographical locations participated.

Sampling Points along the Manufacturing Chain



Sampling at each processing site

Each point was sampled twice; approximately 30 – 60 minutes apart when the process was at equilibrium.

At each of these times separate samples were taken for both residue and microbiology.

The sampling methodology remained the same at each SMP run.

Therefore, at each run there was a minimum of 2 x samples taken from each point; where more than one silo/ evaporator/ dryer etc. was in operation samples were taken from all.

Any additional materials added i.e. lactose or permeate were also sampled and analysed.

Chlorine gas treated water was sampled at all 3 sites at a selection of the manufacturing runs.

- **3 x processing sites;**
  - **A = 2 x SMP manufacturing runs yielding 31 samples in total**
  - **B = 6 x SMP manufacturing runs yielding 80 samples in total**
  - **C = 3 x SMP manufacturing runs yielding 36 samples total**

Analyses

At each SMP run samples were taken for the following analyses;

Residues

- Chlorate/ Perchlorate analysis of all samples (Teagasc Ashtown).
  - LOD for milks & cream was 0.0020 mg/kg
  - LOD for concentrated skim & powder was 0.010 mg/kg
  - LOD for water was 0.00020 mg/L

- TCM analysis of whole milks & creams (Teagasc Moorepark).
  - LOD for milk was 0.00050 mg/kg
  - LOD for cream was 0.0050 mg/kg

Microbiological Plating

Whole milk, skim milk, evaporated milk & powder were tested for;

- TBC (30), TBC (55), Thermoduric bacteria, Mesophilic Spores, Thermophilic Spores, Mesophilic Thermo-Resistant Spores & Thermophilic Thermo-Resistant Spores.
- Additional analyses were conducted on SMP – *Enterobactericeae*, *Bacillus cereus* & Sulphite Reducing Clostridia counts.
- Colonies from a representative number of spore plates were isolated and will be identified via 16S sequencing.

Microbiome

At each available sampling point sub samples were taken with a view to performing “a cutting-edge DNA sequencing-based microbiome analysis of the milk composition”. This is currently being conducted by a member of Professor Paul Cotter’s team.

Detection of Chlorate Across the SMP Manufacturing Chain – Entire Study

Sampling Point	Samples Taken (n=)	Detected Chlorate (n=)	Mean (mg/kg)	Range (mg/kg)	LOD (mg/kg)
Whole Milk Silo	23	1	0.0027	N/A	0.002
Skim Silo	22	4	0.35	0.013 - 1.1	0.002
Evap. Balance Tank	10	0	-	-	0.002
Dryer Balance Tank	24	9	0.21	0.033 - 0.72	0.01*
Skim Milk Powder	22	9	0.018 **(18 ppb)**	0.01 - 0.031	0.01* **(10 ppb)**
Cream Silo/ Line	19	3	0.12	0.0045 - 0.35	0.002*
Lactose Silo	5	1	0.0028	-	0.002*
Permeate Silo	12	6	0.09	0.0028 - 0.19	0.002*
Water Supply	11	7	0.016 **(16 ppb)**	0.00027 - 0.059	0.0002 **(0.2 ppb)**

LOD- Lowest level of detection

Chlorate analysis was conducted on all dairy samples in accordance with FADM 530 which is only accredited for milk. All unaccredited analyses are annotated with an asterix on their LOD figure.

Chlorate analysis was conducted on water in accordance with FADM 530 which is accredited.

All WMS and cream samples were also analysed for TCM but no sample exceeded their respective industry limits of 0.00124 & 0.0124 mg/kg.

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How do Residue Levels Compare to Previous Research?

Paludetti et al. (2019a) conducted a similar study of the SMP chain in 2016 at a similar time period (mid-lactation).

Chlorine based CIP was in place at this time and chlorinated mains water was used in conjunction with this.

2022 Study

Chlorate **WAS NOT DETECTED** in 13 of the 22 **SMP** samples analysed;

- Mean chlorate level of the 13 non-detected samples was <0.010 mg/kg
- Mean chlorate level of the 9 SMP samples with detected chlorate was 0.018 mg/kg; ranging from 0.01 – 0.031 mg/kg

Paludetti et al. (2019)

Chlorate **WAS DETECTED** in all 9 of the SMP samples analysed;

- Mean chlorate level of these 9 samples was 0.057 mg/kg

2016: 57 ppb **2022:** 18 ppb

The low level of chlorate occurrence in SMP sampled in the 2022 study indicates a wide scale compliance with stringent chlorate limits; such as those employed for IMF (0.01 mg/kg); making this commodity grade SMP suitable for more specialised applications such as SNP; from a chlorate perspective.

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Residues in the SMP Chain - Summary

Chlorate

- Only 1 of the 23 WMS samples analysed displayed detectable levels of chlorate (0.0027 mg/kg).
- Only 4 of the 22 skim silo samples analysed displayed detectable levels of chlorate (mean level = 0.35 mg/kg).
- Only 3 of the 19 creams sampled displayed detectable levels of chlorate than in creams (mean level = 0.12 mg/kg).
- Chlorate levels in SMP (where detected) have decreased 3 fold relative to levels detected by Paludetti et al. (2019); 57 ppb (‘16) versus 18 ppb (‘22).
- Chlorate in added ingredients i.e. lactose/ permeate can serve to contaminate the skim milk being dried.

TCM

- Both whole milk (n=23) and cream (n=23) samples were analysed for TCM but none of the samples exceeded the industry limits of 0.00124 mg/kg or 0.0124 mg/kg.

Bacterial Counts Across the Manufacturing Chain – Entire Study

Sampling Point	TBC 30	TBC 55	Thermoduric	MES	HHR-MES	THERM	HHR-THERM
Whole Milk Silo	5.94	3.54	2.12	1.61	1.5	1.5	2
Skim Silo	2.69	3.31	1.59	1.51	1.4	1.65	1.27
Evap. Balance Tank	3.28	2.67	2.25	1.32	1	1.29	1
Dryer Balance Tank	2.03	2.7	1.9	2.03	1.51	2.05	1.23
Lactose Silo	3	1	<1	<1	<1	<1	1
Permeate Silo	2.89	2.46	2.15	1.12	1.08	1	<1

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Bacterial Counts in Skim Milk Powder – Entire Study

Bacteria	No. Plates	No. Plates Detected	Mean (log cfu/g)	Range (log cfu/g)	LOD (log cfu/g)
MES	44	44	2.34	1.85 – 2.81	1
HHR - MES	38	20	1.30	<1 – 1.78	1
THERM	44	44	2.40	1.70 – 3.18	1
HHR - THERM	38	32	1.59	1 – 2.34	1
Presumptive B. cereus	44	8	2.25	2 – 2.78	2
SRC	44	5	1.22	1 – 1.48	1

- Mean TBC of 2.60 log cfu/g in SMP ranging from 1.70 log cfu/g – 3.43 log cfu/g
- Mean thermophilic bacteria count of 3.38 log cfu/g in SMP ranging from 3 log cfu/g – 4.04 log cfu/g
- Mean thermoduric count of 2.20 log cfu/g in SMP ranging from 1.48 log cfu/g – 2.64 log cfu/g

How do Bacterial Counts in WMS Compare to Previous Research?

Paludetti et al. (2019b) is a relevant comparison;

- 2 x WMS samples produced in mid-lactation in 2016 were tested for both mesophilic bacteria (TBC 30) & thermoduric bacteria.
- In 2016 chlorine would likely have been a staple ingredient in both farm and factory CIP.

	Twomey 2022 (unpublished)	Paludetti et al. (2019b)
WMS TBC 30	5.94	5.89
WMS Thermoduric	2.12	1.58

Values presented from this study (Twomey 2022) are mean log -10's of the mean cfu/g counts.
Values presented from Paludetti et al. (2019b) are are mean log -10's of the mean cfu/g counts.

Lowest industry limit for thermoduric in raw milk is 2.70 log-10.

When compared to Paludetti et al. (2019b) TBC and thermoduric levels of WMS samples are;

Different but not considered to be biologically important!

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How do Spore Counts in SMP Compare to Previous Research?

Li et al. (2019) provides a comparison for this work;

- 22 x SMP samples produced in 2016 by multiple Irish processors were tested for a range of spore forming & non-spore forming bacteria.
- In 2016 chlorine would likely have been a staple ingredient of factory CIP.

	MES	HHR-MES	THERM	HHR-THERM	Thermoduric	TBC 30	TBC 55
Twomey 2022 (unpublished)	2.32	1.30	2.38	1.60	2.11	2.26	3.00
Li et al. (2019)	1.33	1.01	1.53	1.22	1.57	1.96	2.15

Values presented from this study (Twomey 2022) and Li et al. (2019) are median log cfu's/g of SMP.

When compared to Li et al. (2019) all SMP bacterial counts from this study are higher than those in SMP produced when chlorine was still widely employed in the dairy industry.

2022 figures are higher but still within specification

Summary

When compared to similar Irish research conducted before chlorine removal from the dairy chain it is evident that;

- Bacterial quality of whole milk supplied to processors is still of excellent quality in 2022 considering 2022 counts are numerically higher than those enumerated in 2016.
- Furthermore, bacterial counts of skim milk and concentrated skim milks were of excellent quality with low TBC, thermoduric and spore counts.
- Total spores counts (MES + THERM + HHR-MES + HHR -THERM) from 2022 average 198 cfu/g (2.04 log cfu/g) when all spore counts are averaged out for all 22 SMP samples.
- This demonstrates excellent SMP quality as spore counts are in compliance with the international standards;
    - US dairy export councils limits for mesophilic spores (<500 cfu/g) and thermophilic spores in SMP (<2,000 cfu/g).
    - EU legislative limit on *B. cereus* of <500 cfu/g in 1/5 samples analysed in accordance with EN/ISO 7932.

Further Discussion – Chlorate in Water

Analysis conducted on chlorate levels in factory water supplies suggests that chlorine gas treatment of water can deliver low levels of chlorate in water.

However, the capacity to achieve these low levels is influenced by the actual source of the water that is treated with chlorine gas;

- **Site A** sourced water from a **local river** and treated this with chlorine gas and had an average chlorate level of 1 ppb (0.29 ppb – 2.9 ppb) in treated water.
    - It is likely that the raw river water contains insignificant levels of chlorate.
- **Site B** sourced water from both an **on site well** and a **municipal water supply** and had an average chlorate level of 30 ppb (0.27 ppb – 59 ppb) in treated water.
    - It is likely that the municipal water is contributing chlorate to this water supply and that chlorine gas treatment did not reduce existing levels; just contributed little chlorate itself.
- In depth investigations of these water supplies pre and post treatment would be needed to definitively understand the contribution of both the original water and the chlorine gas treatment effects.

Conclusions

The adaptation of minimum chlorine strategies by Irish milk processors to minimize chlorate occurrence in dairy ingredients is proving to be effective;

- Chlorate occurrence in SMP is low with only 9 of 22 samples analysed displaying detectable levels of chlorate.

- Spore counts in SMP produced in a minimum chlorine environment are compliant with international standards.

- However, to maintain the utmost standards in terms of spore counts it may be necessary to increase the frequency of CIP in the spray drier to reduce the incidence of spores in SMP.

In conclusion, the outcomes of this research suggests that chlorine free chemical use for CIP coupled with the employment of chlorine gas systems for on-site water treatment can;

- Effectively reduce the occurrence of chlorate in dairy ingredients.
- Maintain satisfactory levels of bacterial quality.

Thank you

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