Method for the determination of product losses in dairy processes

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Abstract

The chemical oxygen demand (COD) in the wastewater of the dairy industry is high. A high COD demand has two problems: First, it could lead to problems with the sewage treatment plant; second, it means a loss of product. It is well-known that approximately 90 % of the COD load is milk components, but it is difficult to specify how much effluents occur from the different processes. Thus, it is necessary to develop a method to determine product losses. This paper shows a possibility of determining product losses and provides information on where, why and how many COD-loaded wastewaters occur. Here, the first rinse water of the cleaning process in a dairy is collected and the COD is analysed. In relation to the COD of the pure product, one can determine the product loss. The presented method was reproducible with a variation coefficient of < 3.6 %, except for cream, which had a variation coefficient of approximately 6.5 %. This method allows for the optimization of processes and the reduction of the COD load of wastewater.

Keywords:Product loss, rinse milk, rinse water, wastewater, COD, chemical oxygen demand, dairy

Introduction

Different losses of milk in the dairy industry result in wastewater. The effluents are generated through the starting, interrupting and cleaning processes. The losses of milk in these processes are approximately 1 – 3 % of the total milk input [1].

The first rinse water from the cleaning process is one of the main parts for the product loss [3]. One method to record product loss is based on the difference between the quantities of raw milk and the product (exclusion of additives). However, this method does reflect the total product loss in the dairy process. Determination of specific product losses dependent on the process step have been not developed. It has been discussed that the COD of wastewater is an adequate parameter for a simple determination of the overall product loss [3]. The output after the cleaning process is a highly COD-loaded wastewater, which varies greatly from 0.5 – 10 g COD / L [1,2]. Therefore, it is recommended to take the total COD mass of wastewater and the equivalent COD of a milk product to calculate product loss.

The aim of this study is to establish a method for the determination of product losses dependent on the process step.

Materials and Methods

Sampling: The samples for COD-Analysis were of the following origin: Samples directly from the process in a dairy were taken over a period of several days:

- Skim milk$_1$ high-heated skimmed milk
- Skim milk$_2$ high-heated skimmed milk
- Skim milk$_3$ pasteurized skimmed milk
- Skim milk$_4$ pasteurized skimmed milk.

Samples from the retail market was bought over a period of several

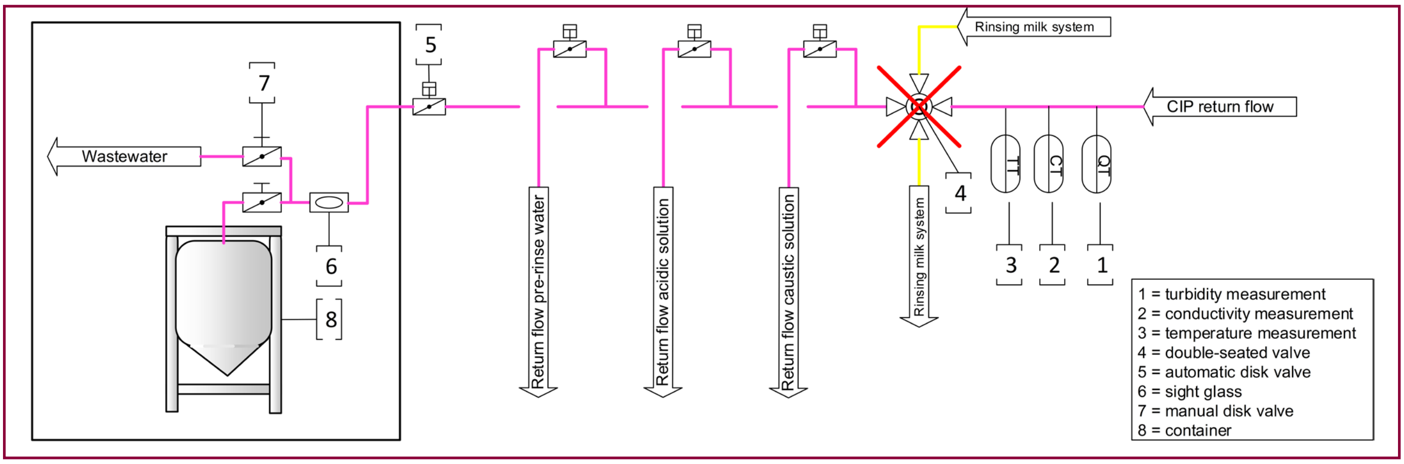

Figure 1: Schematic illustration of the experimental setup

Table 1: Results of COD-analysis for evaluating the reproducibility (each line is obtained from four repeated measurements)

Product	COD Arithmetic mean [g / L]	Standard Deviation [g / L]	COD [g / L]	
			Min	Max
Skim milk$_1$	105.45	± 0.40	105.00	105.90
Skim milk$_2$	104.50	± 0.56	103.80	105.10
Skim milk$_3$	105.38	± 0.33	105.00	105.80
Skim milk$_4$	104.45	± 0.61	103.70	105.10
Arithmetic mean	**104.94**	**± 0.68**	**103.70**	**105.90**

Table 2: Results of the density-analysis for evaluating the reproducibility (each line is obtained from four repeated measurements)

Product	Density$_{20°C}$ Arithmetic mean [kg / L]	Standard Deviation [kg / L]	Density$_{20°C}$ [kg / L]	
			Min	Max
Skim milk$_1$	1.034127	± 0.000187	1.033851	1.034321
Skim milk$_2$	1.030710	± 0.000730	1.029837	1.031532
Skim milk$_3$	1.028041	± 0.000081	1.027917	1.028143
Skim milk$_4$	1.029600	± 0.000221	1.027917	1.028143
Arithmetic mean	**1.030959**	**± 0.002529**	**1.027917**	**1.034321**

weeks:

- H-Milk 0,3 % from Müller Milch in Leppersdorf
- H-Milk 3,5 % from Müller Milch in Leppersdorf
- Cream 33 % from Deutsches Milchkontor in Zeven
- Low fat curd cheese from Molkerei Omira in Neuburg / Donau
- Soft cream pudding from Molkerei Heideblume in Elsdorf.

Experimental procedure: The experimental setup is shown in Figure 1. The main part is in the frame on the left side. On the right side, there is the normal return flow line of a Cleaning-in-Place line (CIP). The turbidity (1), conductivity (2) and temperature (3) are measured. In some dairy plants, there is also a rinse milk system that is shown here with the double-seated valve (4). For the determination of the total product loss, this valve has to be closed. Beyond the automatic disk valve (5), there is normally no further installation except for the wastewater system.

In this setup, the method to determine product loss is a manual process. By visually examining the turbidity through the sight glass (6), the manual disk valve can be switched toward the wastewater system or the container (8) for the rinse milk.

COD analysis: Standard DIN ISO 15705:2003-01 recommends the use of cuvette tests for the determination of the COD [4]. In this work, the cuvette test LCK 514 from HACH LANGE GmbH is used. It is used for the determination of the HT-COD, which represents a modification with a higher digestion temperature (170 °C instead of 148 °C) and a shorter digestion time (15 minutes instead of 120 minutes). This test is for COD values ranging from 0.1 to 2 g / L with a chlorinity content of 1,500 mg/L. Because of this, the samples need to be diluted with distilled water.

Density analysis: For the determination of the density, a DSA 5000 from Anton Paar GmbH is used. The main component of this device is an oscillating U-Tube. The measurement temperature here is 20 °C, which is same as the temperature for the COD measurement. The volume of the measuring cell is filled to approximately 1 ml. To obtain a correct measurement, the U-Tube is flushed with ca. 5 ml of the sample.

Results and Discussion

Reproducibility of COD analysis with skimmed milk: For the evaluation of the COD analysis, it is necessary to check the results of different analyses of the same product. The results of different skim milks are shown in Table 1.

Skim milk samples were taken from the production line. For example, non-homogenized skim milk from the pasteurizer was taken after a heat treatment of 74 °C with a holding time of 20 s at a temperature of approximately 6 °C. The samples were directly chilled and stored at 6 – 8 °C. The COD analysis was performed within the same day after obtaining sample.

With a variation coefficient of < 1 %, there is a low standard deviation among the single analysis and comparison of the different analyses. Thus, it can be concluded that the COD analysis is a good practical and reproducible method for skim milk.

Reproducibility of density analysis: The density analysis is conducted on the same skim milk used for the COD analysis. The results are shown in Table 2. The standard deviation among the single analyses is good. The difference of the results between the analyses is much bigger than the standard deviation of the single analyses. Because of this, it can be concluded that the density analysis works in principle, although there are still some doubts.

One possibility is that the variances of the results are caused by storage. The COD analysis is conducted in the dairy shortly after obtaining the sample. The analysis with the DSA 5000 is sometimes performed a few days later at the university. Thus, there is a time shift in the analysis which could be responsible for the variation. Furthermore, during the storage time of the samples, microbiological degradation cannot be excluded.

Table 3: Results of the rinse milk to determine any possible procedural faults

Product	m [kg]	COD in dilution ± 20 [mg / L]	Dilution 1 part of x parts	Density20°C ± 0,001 [kg / L]	Product loss [kg]
Skim milk$_1$	-	1054.50	100	1.03413	9.83 - 10.51
Rinse milk	660	1570.25	1	0.99993	
Skim milk$_3$	-	1053.75	100	1.02804	8.76 - 9.33
Rinse milk	250	1852.75	2	0.99960	

Table 4: Results of the COD analysis by weighing the sample for dilution (each arithmetic mean is obtained from twelve repeated measurements)				
Product	COD Arithmetic mean [g / kg]	Standard Deviation [g / kg]	COD [g / kg]	
			Min	Max
H-Milk 0,3 %	114.06	± 2.93	110.32	119.86
H-Milk 3,5 %	202.60	± 5.49	195.13	211.09
Cream 33 %	901.96	± 58.28	834.44	1006.98
Low fat curd cheese	224.19	± 8.23	212.26	236.76
Soft cream pudding	504.84	± 7.69	494.99	525.13

Product losses in the skim milk production line: The results of two different cleaning objects with very low product losses are shown in Table 3. The calculation for the total product loss is conducted as follows:

$$product\ loss = mass\ of\ rinse\ milk * \frac{\frac{COD\ (rinse\ milk) \pm 20}{density\ (rinse\ milk) \pm 0.001} * dilution}{\frac{COD\ (product) \pm 20}{density\ (product) \pm 0.001} * dilution}$$

Modification of the COD analysis: The COD analysis shows good results, and the procedural method is reliable with a small fault in regards to the density analysis.

Modification represents a customization of the COD analysis. Normally, the sample has to be pipetted in the cuvette. The dilution for the preparation of the sample was also pipetted. However, it is also possible and more exact to weigh the sample for preparing the dilution. If this is done, density measurements would not be necessary because the results could be calculated as g O_2 / kg. Thus, an advantage of this modification is that there is one less source of error.

Reproducibility of the modified COD analysis with different milk products: The results in Table 4 show a good reproducibility, except for the cream with 33 % fat. This replication has a variation coefficient of the total COD content of approximately 6.5 %, which is more than double of the variation coefficients in other products. In the COD analysis of cream, a dilution of up to one to one thousand was used. This dilution level of cream is also found in the rinse water in the dairy process. Analytical methods, such as those based on the dry mass or fat content, have a much higher error than the COD analyses. Thus, the presented method can be considered as practical for this purpose.

Conclusions

The aim of this work was to develop a method for the determination of product losses in the dairy industry dependent on the process step. Therefore, the recommended method seemed adequate. The weakness of this method was the density measurement. With a modification of the COD measurement, it was possible to eliminate this inadequacy. From further investigations, it was shown that this modification made sense. The modification eliminated the error of the density analysis and led to a variation coefficient of < 3.6 %, except for the cream, which exhibited a variation coefficient of approximately 6.5 %. This may have resulted because the dilution of the sample for COD analysis was based on weight instead of volume, as in a normal procedure.

This method may also be used in other industries, such as the beverage industry. The determination of product losses with the COD as a main parameter is a practicable and reproducible way to answer the following, in combination with the determination of the mass flow based on the absolute mass:

- How many product losses result from the process
- How many COD loads result in the wastewater from the process.

These are important information for obtaining an idea of where the process could be optimized in an economical and environmentally friendly way.

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Conflict of interest

The authors declare no potential conflict of interest with respect to the research, the authorship, and/or publication of the article.

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