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# **Chemically Enhanced Biodegradation of High Organic Matter in Wastewater – Confectionary Plant Effluents**

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**Abstract:** The study explored the merit of chemically enhanced biodegradation of high organic matter as a sustainable treatment scheme for industrial wastewaters. For this purpose, an integrated, chemically enhanced activated sludge configuration was tested for confectionary effluents with a COD level of around 10.000 mg L<sup>-1</sup>. In this configuration, chemical settling acted as a polishing step, which removed 50% of the total COD load, including 10% of colloidal COD in the soluble COD range. The sequential batch reactor, selected as the final biological treatment step, was able to remove the remaining biodegradable COD completely. The study primarily demonstrated the merit of in-plant pollution footprint assessment and wastewater characterization with significant COD fractions as necessary prerequisites for the management and final biodegradation of industrial wastewaters with high organic matter content.

# **Keywords:** biodegradation; chemical settling; COD fractionation; confectionery industry; pollution footprint; sequencing batch reactor; strong wastewater.

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# 1. Introduction

The activated sludge process was discovered and implemented for sewage, wastewater with moderate organic matter content in the range of 400 to 600 mg COD L<sup>-1</sup> [1]. The satisfactory performance of treatment plants severely deteriorated in the face of industrial effluents with much higher organic loads and micro-chemicals; observed problems triggered efforts to develop process modifications restoring the stability of the microbial culture that ensured good performance [2,3]. Nevertheless, accumulated information and experience on the nature of substrate and biomass indicated that complete biodegradation of high COD levels could not be achieved in *single-stage* biological processes [4,5]: anaerobic treatment is inherently incapable to reduce the effluent down to meet effluent limitations and requires the following polishing stage of aerobic treatment [6-9]. Activated sludge configurations alone would also confront a number of severe problems such as oxygen shortage; excessive generation of residual metabolic products; sludge bulking and foaming, which would impede biodegradation and process efficiency; depending on the nature of the wastewater to be treated, they need the support of either an anaerobic step or a wide array of chemical processes as pre-treatment [10,11] Strong industrial effluents with excessive COD loads are well explored in

studies, which generally confirmed the need of two-stage treatment [12-17]. The majority of experimental assessments implemented and successfully tested chemical settling, as a simple and reliable pre-treatment before the final activated sludge step [18-24].

This study selected confectionary plant effluents for a sustainable treatment strategy. The confectionery industry is an important branch of the food industry with a high potential for organic pollution. The industry produces various products such as candies, bars, etc., and it generates an effluent with a COD level of around 10.000 mg L<sup>-1</sup>. Although the organic matter is mainly readily biodegradable [25-27], this COD level is too high to meet the discharge standards into the receiving media. Therefore, studies suggested two-stage biological systems, generally a sequence of anaerobic/aerobic processes, as the appropriate treatment scheme. Berardino et al. [28] investigated anaerobic digestion of confectionery industry wastewater with COD of 0.53 - 2.62 g L<sup>-1</sup> and obtained COD removals higher than 80%. Methane content of the biogas produced varied between 84.3 - 89.9% conducted on a waste survey for a confectionery plant and operated an anaerobic expanded granular sludge bed (EGSB) reactor as a pretreatment before conventional aerobic treatment. The average COD removal and biogas production for the EGSB reactor were reported as 88% and1730 Nm<sup>3</sup> day<sup>-1</sup>, respectively. Balcioğlu et al. [29] operated an anaerobic membrane bioreactor (AnMBR) system to treat wastewater originating from a confectionery plant. 99% COD removals were obtained, and methane yield was 0.31 L CH<sub>4</sub> (g COD)<sup>-1</sup> removed at organic loading of 4.4 kg COD ( $m^3$ .d)<sup>-1</sup>. A relatively early study tested a two-stage anaerobic treatment approach [30], where the COD removal from the confectionery wastewater was investigated by means of a bench-scale sequential anaerobic treatment system; in this study, an up-flow anaerobic sludge blanket reactor (UASB;) followed by a down-flow anaerobic filter (DFAF) was operated at 35 °C and 25 °C, respectively. COD concentration was reduced from 30 to 0.3 g L<sup>-1</sup> at a total organic loading of 5 kg/m<sup>3</sup>/d and a total hydraulic retention time of 2.4 d. Review of the available information in the literature indicated no study testing the merit of chemical settling as a pretreatment step for this wastewater.

In this context, the study's objective was to explore the merit of an integrated, chemically enhanced activated sludge configuration as a sustainable treatment scheme for confectionary effluents. In this configuration, chemical settling acted as a polishing step for the sequencing batch reactor (SBR), adopted as the second stage of activated sludge modification for biological treatment.

# 2. Materials and Methods

# 2.1. Production scheme.

The confectionary plant selected for the study produces a wide spectrum of products. It is kept in operation on three shifts a day and six days a week basis. The number of total personnel varies between 150-300 depending on production requirements. Production processes in the plant can be classified into three groups. The first group covers the production of the main materials used to prepare all products. These main products are chocolate, creme, and chocolate creme. The preparation of these materials involves similar procedures of mixing and grinding raw materials such as sugar, milk powder, cocoa, emulsifier, vegetable oil, and natural flavor. Process materials are conveyed into storage tanks, from which they are fed to different production schemes. The second group of production involves the manufacturing of semi-products such as fondants and marshmallows. These production groups are carried out in reactors that may be heated with non-contact steam and mixing, followed by beating and cooling operations. Manufacture of the final products such as chocolate-coated bars, candies, chocolate-coated wafers, etc., constitutes the third group. In this group, the main operations are mixing, shaping, cooling, cutting, and packaging. A representative example of the footprint of chocolate-coated wafer production is schematically shown in Figure 1.



Figure 1. Process footprint of the chocolate-coated wafer (Product II).

The total production capacity of the plant is given in Table 1. The capacity of the plant in terms of final products is given in Table 1, in terms of five major products, namely is a marshmallow sandwiched between two biscuits and coated with chocolate (Product I); chocolate creme covered bars of caramel (Product II); chocolate-coated wafer (Product III); chocolate-coated bars of fruit paste (Product IV) and crème of chocolate (Product V).

	Total production (ton year <sup>-1</sup> )	Production time (hour year <sup>-1</sup> )
Marshmallow sandwiched biscuits	3800	11120
Caramel bar	300	800
Chocolate-coated wafer	980	5300
Chocolate-coated bars of fruit paste	235	900
Crème of chocolate	750	5500

**Table 1.** The production capacity of the selected confectionary.

### 2.2. Wastewater footprint.

The traditional end-of-pipe inspection of wastewater is perhaps the worst applicable approach for industrial effluents, which generally exhibit significant flow and pollutant loads variations depending on the production scheme of confectionery plants. Instead, a footprint analysis based on a detailed in-plant survey emerged as an effective way to assess the relationships between production steps and pollutant generation and to derive essential data for wastewater management. A typical example of the footprint approach was reported for a textile dye house processing different fabrics [31]. A similar method was used to evaluate energy recovery based on sludge footprints in municipal treatment plants [32]. The same methodology was adopted in this study, which indicated that the wastewater sources could be classified into

three different groups: The first group involved equipment washing and cleaning. The second group was washing the machine parts and/or the rejected products in the dishwashing center. The third group was floor washing and cleaning. Washing of equipment and some material tanks was performed on an intermittent basis. Wastewater sources and volumes determined on the basis of the in-plant survey are tabulated in Table 2. It should be noted that some of the equipment such as silos, storage tanks are cleaned using oil being partly recovered or disposed of as hazardous waste.

	Equipment Washing		Dish-Washing	
	Frequent	Infrequent	Center	Floor washing
Chocolate creme	-	17 m <sup>3</sup> (2y) <sup>-1</sup>	-	-
Chocolate	-	6 m <sup>3</sup> y <sup>-1</sup>	-	-
Creme	-	$16 \text{ m}^3 (2\text{y})^{-1}$	-	-
Marshmallow	$0.80 \text{ m}^3 \text{ w}^{-1}$	-	-	-
Fondant	$0.15 \text{ m}^3 \text{ d}^{-1}$	-	-	0.5 m <sup>3</sup> d <sup>-1</sup>
Product I	-	-	-	0.5 m <sup>3</sup> d <sup>-1</sup>
Product II	-	0.20 m <sup>3</sup> d <sup>-1</sup>	-	-
Product III	0.25 m <sup>3</sup> d <sup>-1</sup> +0.20 m <sup>3</sup> w <sup>-1</sup>	-	0.30 m <sup>3</sup> d <sup>-1</sup>	0.06 m <sup>3</sup> d <sup>-1</sup>
Product IV	$0.20 \text{ m}^3 \text{ d}^{-1}$	-	-	1.5 m <sup>3</sup> d <sup>-1</sup>
Product V		0.20 m <sup>3</sup> d <sup>-1</sup>	$3.0 \text{ m}^3 \text{ d}^{-1}$	-
Total			3.85 m <sup>3</sup> d <sup>-1</sup>	$2.56 \text{ m}^3 \text{ d}^{-1}$

 Table 2. Wastewater sources and volumes derived from footprint analysis.

Table 2 shows wastewater sources and wastewater amounts generated from the investigated plant. Although all productions are fed through central raw material production, the equipment washing effluents are rather infrequent and have a limited contribution to the total wastewater volume. Since the dishwashing center wastewaters are the most important sources in terms of both quality and quantity, while products IV and V play a key role in wastewater generation, the continuous effluent from product V can be considered as the decisive step in affluent generation in terms of the overall wastewater quality and quantity. In general, the central dishwashing and those products necessitating the use of this center are important in evaluating the pollution characteristics in confectionery industries.

# 2.3. Wastewater characterization.

As mentioned above, the major process wastewater source was the dishwashing center; two types of samples were collected from this center as displayed in Table 3: Concentrated samples represented the pre-wash containing a heavy load of contaminants. Grab samples were collected since the flow of pre-wash takes a very short time. The second type sample taken from the dishwashing center was a composite sample of final washing and rinse, which flows for several hours. The volume of the concentrated wastewater was approximately 20% of the total effluent flow.

	COD (mg L <sup>-1</sup> )	BOD5 (mg L <sup>-1</sup> )	Oil&Grease (mg L <sup>-1</sup> )	pH
Concentrated I	59500	39000	1420	6.0
Concentrated II	22550	-	320	7.4
Composite I	10250	15750	-	5.8
Composite II	19220	-	532	4.6
Composite III	14400	-	-	6.5

 Table 3. Dishwashing center and cafeteria wastewater characterization.

# 2.4. Experimental set-up.

Bench-scale experiments were conducted using an effluent sample of the plant prepared by mixing the overall composite samples VI and V at an equal 1:1 ratio on a volume basis and passing the mixture through a 3 mm-opening screen.

Chemical treatment experiments were run using a 1000 mL-capacity Jar-Test apparatus. 5 minutes flash-mixing at 100 rpm, 15 minutes flocculation at 30 rpm, and 2 hours settling sequence were followed. FeCl<sub>3</sub>.6H<sub>2</sub>O, FeSO<sub>4</sub>.7H<sub>2</sub>O, and Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>.18H<sub>2</sub>O, were used as coagulants. pH adjustment was made with lime. Analyses were carried out on the supernatant of the chemically treated samples.

Biological treatment experiments were performed using a 2-L batch reactor equipped with porous stone diffusers to supply air. Activated sludge seed provided from a domestic wastewater treatment plant was acclimated for a month at an organic loading of 1 g COD (g MLVSS.day)<sup>-1</sup>. A mixture of urea, KH<sub>2</sub>PO<sub>4</sub>, and K<sub>2</sub>HPO<sub>4</sub> was added to supply the nutrients and buffer pH. The soluble COD measurements monitored activated sludge process performance. Filtration was made using Millipore 0.45  $\mu$ m membrane filters.

All chemicals used were of analytical grade. All analyses were made in accordance with Standard Methods [33].

# 3. Results and Discussion

In the plant, process wastewaters cannot be separately collected from domestic wastewaters. The main sewer of the plant carried all process effluents as well as domestic wastewaters. Two daily composite samples, which covered all production activities in the plant, were prepared from this sewer. Table 4 shows the analytical results of these daily composite samples, which accurately reflect the characteristics of the overall plant effluent.

Parameter	Unit	Composite IV	Composite V
Total COD	mg L <sup>-1</sup>	9950	9170
Settled COD	mg L <sup>-1</sup>	6470	6925
Filtered COD	mg L <sup>-1</sup>	3520	6650
Total BOD <sub>5</sub>	mg L <sup>-1</sup>	2970	6640
Settled BOD <sub>5</sub>	mg L <sup>-1</sup>	2660	6100
Filtered BOD <sub>5</sub>	mg L <sup>-1</sup>	2360	5500
Oil and grease	mg L <sup>-1</sup>	320	570
TKN	mg L <sup>-1</sup>	56	73
ТР	mg P L <sup>-1</sup>	2.3	3.1
pH	-	6.0	5.9

**Table 4.** Overall characteristics of the plant effluent based on daily composite samples.

The total COD values in Table 4 also include the input of coarse particles, which were broken down by blending before analysis. These coarse particles can be screened before treatment, as mentioned in the following section. Characterization of composite wastewater samples also included a preliminary fractionation of COD in terms of total COD, supernatant of plain settling (settled COD), and filtrate through 450 nm filters (soluble COD) components; the same characterization was also carried out for BOD<sub>5</sub>, which seems to be correlated with the soluble COD fraction. Currently, this type of preliminary COD fractionation has been improved into an elaborate particle size distribution (PSD) analysis in a much larger size spectrum down to 2.0 nm, and it is widely applied to both sewage and industrial wastewaters [34,35]. It is interesting to note the oil-grease as high as 570 mg L<sup>-1</sup>, which qualifies to be a hard-to-treat parameter for anaerobic processes.

# 3.1. Treatment rationale.

Inspection of the wastewater characteristics reveals two significant clues: (i) an influent COD level of around 9500 mg  $L^{-1}$  is too high to comply with the effluent treatment by means of a single-stage treatment, whether it be an anaerobic or an aerobic process. (ii) it is particularly suitable for the envisaged chemically enhanced activated sludge process.

In fact, the analyses indicated a COD removal potential of around 30%, even with plain settling. Thus, a chemically induced settling would be expected to remove all particulate organic matter as well as a portion of what was assessed as soluble COD. This way, it would also homogenize the wastewater character before being fed into the aerobic treatment stage. A sequencing batch reactor (SBR) was selected as an activated sludge configuration for the aerobic biological treatment stage, mainly because of the well-tested operation flexibility of the SBR process for variations of wastewater volume and strength as well as for settling character.

# 3.2. Chemical settling.

The results of chemical settling experiments through coagulation-flocculation experiments conducted using three different coagulants, alum, FeSO<sub>4</sub>, and FeCl<sub>3</sub>, at dosages varying between 250 and 750 mg L<sup>-1</sup>, are given in Table 4. The data provided a clear indication that all coagulants, when applied at dosages of 250-300 mg L<sup>-1</sup>, yielded a COD removal that varied in the narrow range of 51-55%, including the preliminary screening. Increasing the FeSO<sub>4</sub> dosage to 750 mg L<sup>-1</sup> did not prove feasible as it resulted in a slight increase in the COD removal to 58%. Interestingly, all effluent COD concentrations obtained after chemical settling averaged 4500 mg L<sup>-1</sup>, appreciably lower than the soluble level of 5000 mg L<sup>-1</sup> in the tested mixture. This observation showed that chemical settling provided full removal of particulate COD and 10% of the colloidal COD below the size threshold of 450 nm. This treatment step also secured the complete removal of suspended solids as well as oil and grease.

Tuble 2. Results of Chemical Treatment.				
Coagulant	Dosage	pН	Effluent COD	COD Removal
	(mg L)		(mg L)	(%)
$Al_2(SO_4)_3$	250	6.7	4685	30
$Al_2(SO_4)_3$	300	6.7	4660	30
FeCl <sub>3</sub>	250	8.0	4530	32
FeCl <sub>3</sub>	250	9.0	4320	36
FeSO <sub>4</sub>	250	8.3	4440	34
FeSO <sub>4</sub>	750	8.5	4030	40

Table 5. Results of Chemical Treatment.

### 3.3. Biological treatment.

The successful application of SBR, mainly due to its flexibility of operation for different types of wastewaters, is widely reported in the literature [36-40]. In this study, the operation of the lab-scale SBR unit was adjusted to a cycle time of 1.0 days, with a processing time,  $T_P$  of 22 h, and an additional 2.0 h for settle; decant; idle period. The feeding time,  $T_F$ , covered the first 5.0 h of the cycle, as in *fill and draw*-type of an operation. The effluent of chemical treatment (FeSO<sub>4</sub> experiment at 250 mg L<sup>-1</sup>) was selected as the wastewater feed, which sustained a biomass concentration of 4000 – 4500 mg MLSS L<sup>-1</sup> at a steady state. The performance of the SBR system under different organic loadings is outlined in Table 6.

Organic load		Effluent COD Removal	
	(gCOD (g MLVSS.d) <sup>-1</sup> )	(mg L <sup>-1</sup> )	%
	1.69	115	97
	1.51	110	97
	1.13	100	98
	0.93	90	98
	0.76	90	98

 Table 6. Performance of the SBR system.

As seen from Table 5, the SBR system yielded very high COD removals even for the highest organic loadings. Decreasing the organic loading did not further increase the COD removal efficiency. Lower organic loadings resulted in the same efficiencies indicating that all biodegradable COD was fully utilized at almost all organic loadings rates, and the effluent COD corresponded to the soluble residual metabolic products generated in the course of biological reactions in the reactor, as confirmed by the glucose test performed in accordance with the methodology recommended by Germirli *et al.* [41]. Flocculent settling of the biomass was quite good with an average SVI value of 75 mL g<sup>-1</sup>, and the effluent suspended solid concentration always remained lower than 40 mg L<sup>-1</sup>.

# 3.4. Recommended treatment scheme.

The pollutant footprint analyses and differentiation of organic matter in terms of settled and filtered COD fractions provided significant clues for a sustainable treatment scheme: (i) substantial load and flow variations in different effluent streams; (ii) the low magnitude of total daily effluent flow; (iii) a total COD level of approximately 10.000 mg L<sup>-1</sup>, which necessitates a two-stage treatment. Based on these indications, treatability studies justified the application of a sequence of chemical settling and SBR that would be operated batch-wise, involving the following configuration: a separate collection and pre-treatment of individual waste streams; an aerated equalization tank with sufficient volume to allow batch feeding of the main treatment sequence; a chemical treatment tank that can function both as a reactor and a settler and an SBR unit. A schematic display of the proposed treatment scheme is displayed in Figure 2.

# 4. Conclusions

The study primarily demonstrated the merit of in-plant pollution footprint assessment and COD characterization with differentiation of particulate, settled and soluble fractions in the effluent as necessary prerequisites for the management and final biodegradation of industrial wastewaters with high organic matter content. It also underlined the need for particle size distribution analysis as a useful complement of biodegradation experiments. This approach constituted the basis for assessing the treatability of the confectionary effluents, which identified a two-step chemically enhanced SBR system as the sustainable treatment scheme; wastewater characteristics were particularly suitable for chemical settling, which provided 50% removal of the total COD load, including 10% of colloidal COD in the soluble COD range. The sequential batch reactor, selected as one of the most convenient types of biological treatment for batch operation, achieved full removal of biodegradable COD, leaving only soluble residual metabolic products of around 100 mg COD L<sup>-1</sup> in the effluent.



Figure 2. Recommended chemically enhanced SBR treatment.

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