

OVERVIEW OF WASTEWATER CHARACTERISTICS OF CARDBOARD INDUSTRY

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Abstract: The purpose of this study is to characterize wastewater from the corrugated cardboard industry and to highlight the nature and sources of pollution. Wastewaters from the corrugator and printing processes as well as homogenization tank, which collects all effluents from industrial processes, were analysed using standard methods. The results indicate that these effluents had a significant pollution load. The wastewater from the homogenization tank had high concentration of COD ($24243 \pm 2374.6 \text{ mg}\cdot\text{L}^{-1}$), BOD₅ ($413.33 \pm 17.14 \text{ mg}\cdot\text{L}^{-1}$) and total solids ($36.84 \pm 10.62 \text{ g}\cdot\text{L}^{-1}$). In addition, the biodegradability indices were less than 0.4, indicating that the effluents from the cardboard industry are not readily biodegradable. The printing process is the main source of liquid pollution in the cardboard industry facilities. The pollution load resulting from this process was much greater than that of the corrugator process wastewater. In accordance with current standards, these industrial effluents require treatment before discharge or re-use.

Keywords: *cardboard industry, wastewater characteristics*

INTRODUCTION

The corrugated cardboard industry is a highly important economic activity in the manufacturing chain, which links the raw product to the final consumer. This industry is currently experiencing many changes in terms of the increase in demand, the administrative nature that oversees it and the new commitments forced on it in the field of safeguarding of the environment.

The paper and cardboard industry is highly concentrated in North America. Fifty percent of global production was achieved in America, 26 % in Asia Pacific, 21 % in Europe and only 3 % in the Middle East and Africa. To satisfy the increase in demand, world paper and board production increased by 0.8 % in 2017 and 0.5 % in 2018. In Morocco, the paper and cardboard industry area accounts for about 62 companies in 2011 and produces a total of 230000 tons of paper and cardboard [1].

The corrugated cardboard industry frequently uses starch, glue, pigments and inks for the conditioning of cardboard and the printing of advertisements. The process generates 75 to 275 m³ of wastewater per ton of product containing the high Chemical Oxygen Demand (COD), high concentration of Suspended Solids, color and organic substances [2 – 4]. The discharge of such wastewater into the environment impedes light penetration, damages the quality of the receiving streams and may be toxic to treatment process, to food chain organics and to aquatic life [5 – 7].

This study aims to characterize the wastewater generated in a cardboard mill. The main pollution parameters are assessed and compared to results reported in previous works.

EXPERIMENTAL METHODS

Wastewater sources

The wastewater used in this study was collected from a corrugated cardboard industry located in Agadir, (South of Morocco) with an annual cardboard production capacity of 66596 t. The farm is equipped with physicochemical treatment plant with a homogenization tank and coagulation flocculation unit.

The wastewater generated by the manufacturing of corrugated cardboard can be classified into the following categories: Blackwater; rainwater; wastewater from clean-up of combining adhesive (corrugator and glue making machine) and wastewater from clean-up of printing ink. The company uses tap water or ground water as water resources.

The most significant sources of wastewater are ink and glue. The ink contains pigments dispersed in the varnish; the pigments are organic or inorganic. Organic pigments contain diverse metal oxides, which give certain colors and inorganic pigments are produced from synthesis and mineral oils. Other compounds which are found in the ink are water, acrylic resins, anti-foams additives, and other additives. The compounds found in the glue other than water are starch, boric acid, anti-foams additives, caustic soda and other additives. This mixture of many compounds may complicate the wastewater treatment.

Samples

Wastewater samples were obtained from three wastewater tanks: the first one receiving wastewater of printing process (PW), the second one collecting wastewater from clean-up of combining adhesive (corrugator and glue making machine) (CW) and the last tank serving as an homogenization tank (HW) which collect the whole industrial process wastewaters.

From each tank, twelve 2 L composite samples were collected using an automatic sampler programmed to collect 1 L of wastewater every 60 min for 24 h. Each composite sample was obtained by mixing thoroughly two samples of 1 L each.

Analytical procedures

After collection, the samples were stored at 4 °C and preserved by acidification to $pH \leq 2$ using sulphuric acid or nitric acid when necessary. Tests were carried out for pH , temperature, conductivity, turbidity, solids, sulfate, volatile fatty acids, biochemical oxygen demand (BOD_5), chemical oxygen demand (COD), total phosphorus (Pt), color and total Kjeldahl nitrogen (TKN). All samples were preserved and analyzed according to the standard methods recommended by the American Public Health Association [8]. Oxydable matters (OM) are calculated according to Boeglin [9]. ($OM = COD + 2(BOD_5)/3$)

RESULTS AND DISCUSSION

Corrugated cardboard manufacturing process

Corrugated packaging manufacturing can be roughly classified into two processes: the containerboard combining process, which glues one or more sheets of fluted corrugating medium to one or more flat facings of linerboard; and the box manufacturing process, which is used to assemble the corrugated sheets into boxes and printing. Figure 1 depicts the manufacturing flow and ancillary facilities used in a corrugated packaging plant.

Corrugator process

The corrugated board manufacturing involves several processes. First, a paper was steam heated to form the linerboards (outer layers of cardboard) and other paper was steam heated and corrugated to form the ‘medium’ of the cardboard. Then, top and bottom linerboards are glued and attached with ‘medium’ to form cardboard. To remove excess moisture and to allow the glue to set, the cardboard is pressed with hot plates. At the end, the corrugated boards are dried, scored, cut and loaded (Figure 2).

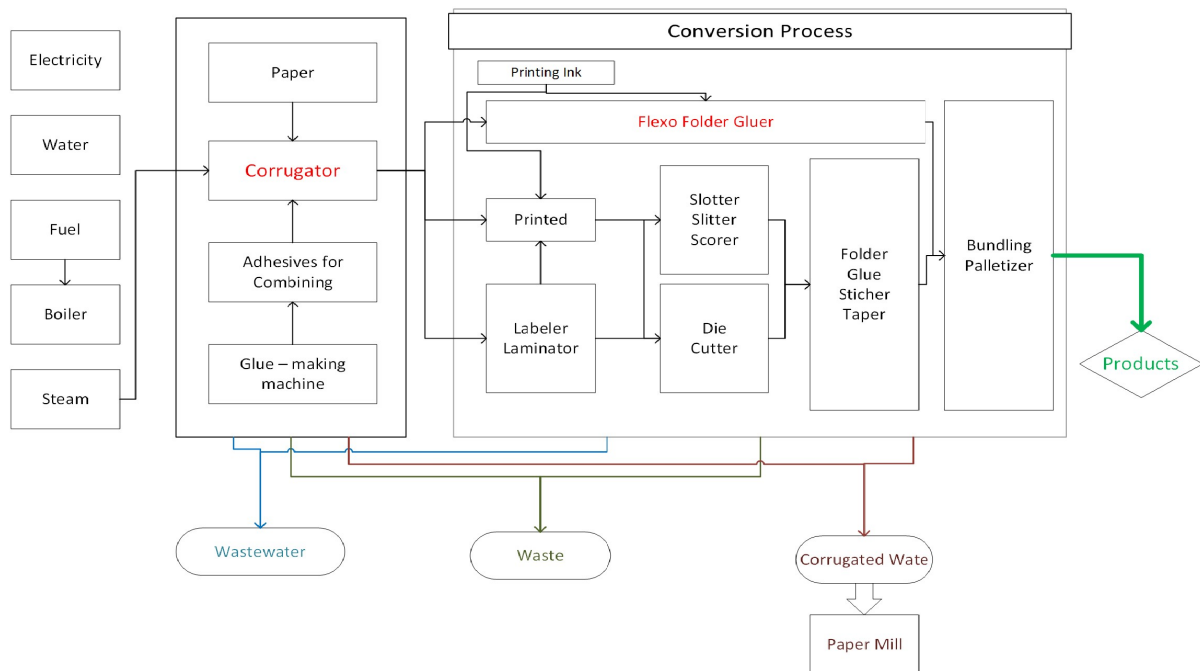


Figure 1. Overview of manufacturing process flow for corrugated packaging

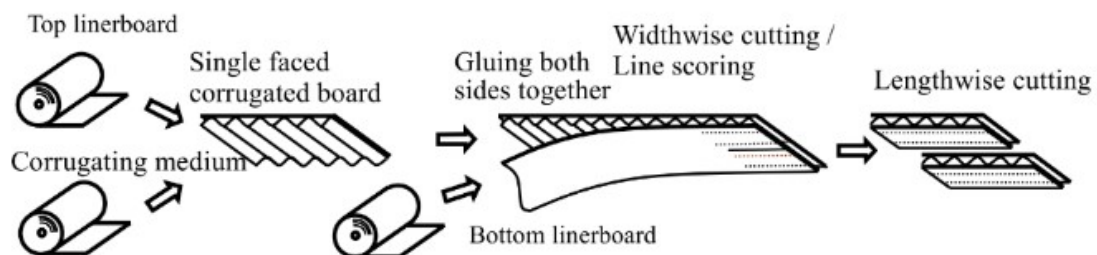


Figure 2. Containerboard combining process

Printing process

Among many printing processes, the corrugated cardboard manufacturing usually uses a flexography, which was initially developed for packaging applications (corrugated cardboard) [10]. This printing process is direct and uses a relief flexible plate that applies a fluid ink in contact with a metering roller called "anilox" and closed by two doctor blades. The ink comes from an ink chamber. A Flexo ink was formulated by the following components (Table 1):

Table 1. Components of Flexo ink

Components	Percentage [%]
Water	50 – 75
Acrylic polymers	15 – 30
Pigments organic and inorganic	8 – 20
Additives (wax, anti-foams...)	3 – 5

Corrugated cardboard wastewater

Corrugator process wastewater (CW)

Wastewater results from cleaning of the corrugator, specifically from the glue circuit and the glue preparation tanks. These effluents are very rich in glue and have a yellowish coloring. They are collected and then poured into a wastewater tank (CW). The results obtained from the experimental analysis of the wastewater samples are given in Table 2.

The corrugator process wastewater was characterized by substantial organic matter, high salinity, and high solid content (Table 2). COD and BOD values were 2305 and 260 $\text{mg}\cdot\text{L}^{-1}$ respectively. The high organic load of the effluent is essentially due to the presence of the starch during washing water process. BOD_5/COD index value was of 0.11 and indicates that the effluent is not easily biodegradable. Wastewater is considered readily biodegradable if it has a ratio value between 0.4 and 0.8 [11]. Total solid in the effluent was 9.66 $\text{g}\cdot\text{L}^{-1}$, which can be attributed to the starch glue in the water discharged. The pH value was 9.6, is attributed to the use of caustic acid in the process.

Table 2. *Physicochemical characteristics of corrugator wastewater*

Parameters	Value
pH	9.6
Temperature [$^{\circ}\text{C}$]	23
Conductivity [$\text{mS}\cdot\text{cm}^{-1}$]	11.63
COD [$\text{mg}\cdot\text{L}^{-1}$]	2305.00
BOD_5 [$\text{mg}\cdot\text{L}^{-1}$]	260.00
Settleable Solids [$\text{mL}\cdot\text{L}^{-1}$]	41.00
TSS [$\text{g}\cdot\text{L}^{-1}$]	1.53
TS [$\text{g}\cdot\text{L}^{-1}$]	9.66
TKN [$\text{mg}\cdot\text{L}^{-1}$]	22.41
Pt [$\text{mg}\cdot\text{L}^{-1}$]	0.38
BOD_5/COD	0.11
Oxydable matters (OM) [$\text{mg}\cdot\text{L}^{-1}$]	941.67

Printing process wastewater (PW)

During the process of printing the corrugated cardboard, particularly the washing of the flexographic printing machines, the water is used to remove the rest of the printing ink, then this water is evacuated and sent to printing process wastewater tank (PW). Water used for the washing was directly evacuated and the flow varies according to the variation of the number of orders by machine. This effluent has a black color mainly caused by the solid ink residue.

Wastewater quality varies with different kinds of ink, which has different connected makings, pigments, and additives. The quality and quantity of wastewaters also varies with different process sections of the ink production. Characteristics of this wastewater are presented in Table 3.

The pH tracking during 24 h shows that it fluctuates between 8.36 and 9.24 indicating the alkaline character of this rejection. Ink was present in colloidal form in the wastewater; as a result, the contents of TS, TSS and TVS are higher with main values of

5.9, 3.16 and 4.3 $\text{g}\cdot\text{L}^{-1}$ respectively. The turbidity indicates a very high value >17500 NTU and the conductivity varies between 20.3 and 39 $\text{mS}\cdot\text{cm}^{-1}$.

The ink wastewater is rich in organic materials. Indeed, the contents of COD, BOD_5 and TKN are of the order 9496.67 ± 3103.8 , 650 ± 182.6 and 252.18 ± 70.89 $\text{mg}\cdot\text{L}^{-1}$ respectively (Table 3). The large oxydable matters (3815.56 $\text{mg}\cdot\text{L}^{-1}$) was mainly due to the nature of the water-based raw material, which contains organic pigments, polymers and chemicals.

The mean value of BOD_5/COD ratio was 0.068 and varied between 0.046 and 0.074, showing that the effluent is not biodegradable. The measurement of the absorbance at different wavelengths displays important values, which shows the high concentrations of colored matters.

It can be concluded that printing process wastewaters are more substantially loaded on organic matters than corrugator wastewater. In fact, TSS, OM and TKN of PW were 2, 4 and 11 times more important than those of corrugator process effluent respectively. The printing process effluent seems to be the main pollution source in cardboard industry. In fact, pollution load in terms of TSS, OM and TKN are 1125.75, 48.24 and 22.46 $\text{kg}\cdot\text{d}^{-1}$ respectively.

Table 3. Physicochemical characteristics of printing process wastewater

Parameters	Printing Wastewater		
		Min	Max
Flow rate [$\text{m}^3\cdot\text{day}^{-1}$]	191.28 ± 0.57	128.16	253.68
pH	8.7 ± 0.225	8.36	9.24
Temperature [$^{\circ}\text{C}$]	22 ± 0.916	19.8	22,7
Cond. [$\text{ms}\cdot\text{cm}^{-1}$]	25.67 ± 4.81	20.3	39
Turbid. [NTU]	>17500	-	-
COD [$\text{mg}\cdot\text{L}^{-1}$]	9496.67 ± 3103.8	4805	20205
BOD_5 [$\text{mg}\cdot\text{L}^{-1}$]	650 ± 182.6	360	940
TSS [$\text{g}\cdot\text{L}^{-1}$]	3.16 ± 0.86	1.8	4.15
TS [$\text{g}\cdot\text{L}^{-1}$]	5.9 ± 0.68	4.864	7.34
TVS [$\text{g}\cdot\text{L}^{-1}$]	4.3 ± 0.862	3.166	5.91
TKN [$\text{mg}\cdot\text{L}^{-1}$]	252.18 ± 70.89	112.08	364.26
P [$\text{mg}\cdot\text{L}^{-1}$]	0.284 ± 0.05	0.204	0.366
Volatile fatty acids [$\text{mg}\cdot\text{L}^{-1}$]	63 ± 15.1	42	90
BOD_5/COD	0.068	0.046	0.074
Oxydable matters [$\text{mg}\cdot\text{L}^{-1}$]	3815.56	1961.67	7675
Color	Abs. 436 [nm]	1.13 ± 0.7	0.34
	Abs. 525 [nm]	0.74 ± 0.46	0.23
	Abs. 620 [nm]	0.82 ± 0.52	0.21

Homogenization tank wastewater (HW)

Parameters of physical-chemical characteristics of the corrugated cardboard industry wastewater samples analyzed in this work are summarized in Table 4. They are discussed in comparison with data of Cardboard Industry effluents available in the literature (Table 5), where the COD, solids, pH, BOD_5 and other more specific parameters are analyzed by the authors.

Table 4. Physicochemical characteristics of homogenization tank wastewater

Parameters	Homogenization tank Wastewater		
		Min	Max
Flow rate [$\text{m}^3 \cdot \text{day}^{-1}$]	6000	4000	8000
pH	7.24 ± 0.21	6.32	7.8
Temperature [$^{\circ} \text{C}$]	24	-	-
Cond. [$\text{mS} \cdot \text{cm}^{-1}$]	31.62 ± 1.94	27.5	40.2
Turbid. [NTU]	>17500	-	-
COD [$\text{mg} \cdot \text{L}^{-1}$]	24243 ± 2374.6	19110	34010
BOD ₅ [$\text{mg} \cdot \text{L}^{-1}$]	413.33 ± 17.14	200	660
TS [$\text{g} \cdot \text{L}^{-1}$]	36.84 ± 10.62	7.688	76.94
Sulfate [$\text{mg} \cdot \text{L}^{-1}$]	546.79 ± 185.95	171.875	1296.875
TKN [$\text{mg} \cdot \text{L}^{-1}$]	366.6 ± 41.08	252.18	532.38
P [$\text{mg} \cdot \text{L}^{-1}$]	0.318 ± 0.08	0.069	0.685
Volatile fatty acids [$\text{mg} \cdot \text{L}^{-1}$]	834 ± 8.43	348	1572
BOD/COD	0.017	0.01	0.019
Oxydable matters [$\text{mg} \cdot \text{L}^{-1}$]	8359.67	6503.33	11776.67
Color	Abs. 436 [nm]	0.643 ± 0.44	0.032
	Abs. 525 [nm]	0.562 ± 0.40	0.028
	Abs. 620 [nm]	0.684 ± 0.67	0.014

Table 4 illustrates the analysis of homogenization tank wastewater which served as a receptacle of the whole industrial process effluents. Except for turbidity, the values of other parameters are in the same order to those cited in other studies. In addition, the values of all parameters analyzed exceed the wastewater discharge limits.

If an effluent with a high oxygen demand is discharged directly into surface water, the sensitive balance maintained in the water becomes overloaded. Oxygen is stripped from the water, causing oxygen dependent plants, bacteria, fish present in the river or stream to die. This results in increase of non-oxygen dependent (anaerobic) bacteria leading to toxic water conditions. A healthy river can tolerate substances with low levels of oxygen demand. The load created by industrial wastewater, however, is often excessive, and effluents require treatment prior to discharge. The oxygen demand needs to be determined in order to assess a discharged effluent's impact on surface waters or determine the costs of treatment. This can be achieved in two different ways, COD and BOD₅ assessments.

Chemical oxygen demand analysis showed values between 19110 and 34010 $\text{mg} \cdot \text{L}^{-1}$ and an average value of $24243 \pm 2374.6 \text{ mg} \cdot \text{L}^{-1}$ (Table 4). These values are similar to those obtained by Gilboa [12], Fendri *et al.* [13] and Touil [14] (Table 5). Biochemical oxygen demand was of $413.33 \pm 17.14 \text{ mg} \cdot \text{L}^{-1}$ and ranged between 200 and 660 $\text{mg} \cdot \text{L}^{-1}$. BOD concentrations are similar to those obtained by Nassar [15], and Mansour and Kesentini [16]. This important load of oxygen demand matters is mainly due to the nature of the raw material based on chemicals, starch, and ink used in manufacturing processes. Investigations showed that COD is very higher than BOD₅, suggesting that products used in Cardboard processing are not easily biodegradable. In fact, BOD₅/COD ratio, used by many authors as biodegradability index, was of 0.017 and ranged from 0.010 to 0.019, indicating that cardboard industry wastewaters are not readily biodegradable.

Table 5. *Parameters of raw cardboard industry wastewater from the literature*

Ref. Parameter	[19]	[21]	[22]	[20]	[12]	[17]	[16]	[15]	[18]	[14]	This work
Flow rate [m ³ ·day ⁻¹]	-	-	-	6000-8000	-		-	-	-	-	4000-8000
T [°C]	27	-	-	-	-	25.01 ±0.1	-	-	50	16.8	24
pH	6.03	-	6.8	6.9 - 7.1	-	7.79 ±0.79	7.5	-	6.4	8.86	7.24
VFA [mg·L ⁻¹]	-	1100 - 3500	640-1040	-	-	-	-	-	-	-	834
COD [mg·L ⁻¹]	17 606	4500 – 11400	740-1006	2358	6000 – 30000	38595 ±20926	3600	1400	6795.5	20000	24243 ±2374.6
BOD ₅ [mg·L ⁻¹]	4053			-	-	9750 ±3126	500	500	-	1760	413.33 ±17.14
TS [mg·L ⁻¹]	-	-	3150-3760	5000 - 5850	-	27555 ±11488	-	3000	-	-	36 840 ± 10620
TSS [mg·L ⁻¹]	4 232	-	-	3000 - 5000	2000 – 10000	4228 ±2600	266	-	6450	8 200	-
Cond. [mS·cm ⁻¹]	-	5733	-	2.271-2450	-	-	-	-	3450	-	31.62
Turbid. (NTU)	-	230 – 670	-	783	-	-	-	-	1942.5	-	>17500
PO ₄ -P [mg·L ⁻¹]	-	-	-	2.8	-	-	-	-	-	-	-
Phenols [mg·L ⁻¹]	-	83	-	-	-	-	-	-	-	-	-
SO ₄ ²⁻ [mg·L ⁻¹]	-	-	-	241	-	-	-	-	-	-	546.79
Arsenic [mg·L ⁻¹]	0.014	-	-	-	-	-	-	-	-	-	<0.01
Al [mg·L ⁻¹]	-	-	-	-	-	-	-	-	64.5	-	337
Pb [μg·L ⁻¹]	-	-	-	-	-	-	-	-	15.25	-	<0.01
Ni [μg·L ⁻¹]	-	-	-	-	-	-	-	-	16.1	-	0.2905
Zn [mg·L ⁻¹]	2.51	-	-	-	-	-	-	-	53.3 ·10 ⁻³	-	1.8029
Color	-	-	-	-	Black	Black	-	-	Grayish	-	Black

Total solids are dissolved solids plus suspended and settleable solids in water. The sources of total solids include industrial discharges, sewage, fertilizers, road runoff, and soil erosion. Total solids affect water clarity. Higher solids decrease the passage of light through water, thereby slowing photosynthesis by aquatic plants. Water will heat up more rapidly and hold more heat; this, in turn, might adversely affect aquatic life that has adapted to a lower temperature regime. Total solids content in the wastewater originating from homogenization tank was higher ($36.84 \pm 10.62 \text{ g·L}^{-1}$) and ranged between 7.688 and 76.94 g·L^{-1} . TS concentration is similar to this found by Khannous *et*

al. [17] and more important than those obtained by other authors. The effluent show a high turbidity, higher than 17500 NTU, related principally to TS load.

In addition to the high organic matter and TS loads, the effluent shows high concentration of sulfate ranging between 171.875 and 1296.875 mg·L⁻¹. The large amounts of sulfate ions may contribute to the formation of H₂S in oxygen-poor environments, which constitute a risk of corrosion. The low phosphorus concentration suggests that P is the limiting nutrient parameter for the biological treatment of the wastewater.

The homogenization tank wastewaters are colored as a result of the presence of ink in the cleaning wastewater that comes from the printing ink. In Table 5, the measurement of the absorbance at different wavelengths shows that the wastewater from this plant absorbs visible light.

Metals

The overall metal contents are given in Table 6. Aluminum content was substantial and showed values of 11.464, 9.7237 and 337.69 mg·L⁻¹ in printing wastewater, corrugator wastewater, and homogenization tank wastewater respectively. Compared to early studies findings (Table 5), the homogenization tank wastewater aluminum content was more important. This can be attributed to the fact that the homogenization tank wastewater is generally made up of wastewater from ink pit, glue pit and the returned water from sludge dewatering tank. Poly-aluminum chloride is used for the treatment of wastewaters in the company using coagulation flocculation process with a sludge dewatering step. With a frame filter press, the sludge is scraped off the belt and collected in a bin while the water content is transferred back into homogenization tank for further treatment. As the optimum physico-chemical condition of the treatment of raw water is not well established, the returned flow water from sludge treatment contains significant amount of residual poly-aluminum chloride. Renault *et al.* [18] and Bellarbi *et al.* [19] reported the same concentration of the heavy metals (Zn, Ni, Pd, As), which could be attributed to the presence of these metals in the raw materials.

Table 6. Mean concentration of the metals [mg·L⁻¹]

Metal	Printing wastewater	Corrugator wastewater	Homogenization tank wastewater
Al	11.464	9.7237	337.69
As	<0.01	<0.01	<0.01
Cd	<0.01	<0.01	<0.01
Cu	<0.01	27.414	51.965
Cr	<0.01	<0.01	0.5766
Ni	<0.01	<0.01	0.2905
Pd	0.0783	0.109	<0.01
Zn	0.8018	2.9703	1.8029

Pollution load

The daily flow discharge from this corrugated cardboard industry ranged from 4000 to 8000 m³·day⁻¹ and contributes to the rejection of an important pollution load. According to the results obtained for this industrial unit, the pollution loads of COD and TS are 145460 kg·d⁻¹, 221014 kg·d⁻¹ respectively. COD pollution loads is 9 times more important than that reported by Gengec [20] (Table 7).

Table 7. Pollution loads [kg·d⁻¹]

	COD	TS	Sulfate
This work	145460	221014	2421.88
Gengec, (2017)	16506	37975	1687

CONCLUSION

This study investigated the corrugated cardboard industry wastewater characteristics. Standard methods were used to analyze organic and inorganic parameters in corrugator process wastewater, printing process wastewater and homogenization tank wastewater. The results indicate that the pollution load in studied wastewaters was high and the mainly wastewater characteristics, total solids, turbidity, COD and color, were present at levels above those allowed by the criteria for release into a receiving body. Furthermore, corrugated cardboard industry wastewater was not readily biodegradable, and can cause considerable damage to the environment.

It can be concluded that, the printing effluent is the main pollution source in cardboard industry and must be studied thoroughly. In order to reduce pollution loads and prevent subsequent impacts, Cardboard industry wastewater requires adequate treatment before its discharge.

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