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Emission of Bisphenol A and Four New Analogs from Industrial Wastewater Treatment Plants in the Production Processes of Polypropylene and Polyethylene Terephthalate in South America

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Abstract: The study of the presence of bisphenol analogs in the environment has been very relevant in recent years because their toxic potential has been discovered, and since they are not regulated like bisphenol A, their use and presence in industry has been excessive. This study identifies and quantifies for the first time the presence of bisphenol A and four uncommon bisphenol analogs in waste from polypropylene (PP) and polyethylene terephthalate (PET) production processes that may originate from the degradation of some compounds used during resin synthesis in Colombia to determine the effectiveness of removal of these components. The data obtained show that the treatments used in these waters are insufficient to eliminate 40% of the bisphenols present in them, and when evaluating the profiles of compounds, it is clear that the compound with the highest removal during the PP process was D-BPA-1, while the compound with the highest removal during the PET process was D-BPA-4, indicating that identification and elimination systems for bisphenols are rudimentary.

Keywords: bisphenol A; bisphenol analogs; wastewater treatment plants; pollutions; polypropylene manufacturing

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1. Introduction

Bisphenols are molecules widely used at the industrial level, and within bisphenols, bisphenol A stands out [1], of which the harmful effect it has on the health of human beings has been evidenced, since it acts as an endocrine disruptor and causes affectations at the neuronal, immunological, and cardiovascular levels [2,3]. For this reason, regulations and restrictions have been established for its use in various products in countries such as the United States and in the European Union [4–6]. However, these regulations only apply to bisphenol A, so molecules analogous to it that are structurally or physicochemically similar can be freely used, which has increased its implementation to replace bisphenol A in many applications at the industrial level [7,8]. Bisphenol analogs (BPs) are chemical compounds with two hydroxyphenyl functional groups (e.g., bisphenol B, bisphenol F, and bisphenol C) [9–12] and are applied in a variety of consumer products [9,13]. The concern of the scientific community and government agencies responsible for public health to investigate the effects that these substances have on the reproductive systems of living organisms has also increased [14].

The presence of BPs in the environment has been documented by several authors, who detected these molecules in drinking water, domestic wastewater, sediments, surface water from rivers, etc. [8,13,15–19]. Wastewater goes through different processes that

allow it to have the necessary requirements for its discharge into bodies of water. The implementation of evaluation systems for the removal effectiveness of BPs in domestic wastewater treatment plants (WWTPs) and in industrial wastewater treatment plants (iWWTPs) is of great importance to ensure that there are no dumping substances that are potentially dangerous to people's health. Reported efficiencies of up to 100% for these plants are biased, leaving out a wide range of emerging contaminants that are not removed.

Recent studies have found BPs in different WWTPs throughout the world [18,20,21]. In Latin America, the presence of bisphenols has been reported, with average values of 1.2 ng/L in drinking water, 183.26 ng/L in wastewater, 4.11 ng/L in drinking water treatment plants, and 64,200 ng/L in surface water [22,23]. Such values of bisphenol serve as warnings to the scientific community and public health control agencies that stricter controls and regulations on the use, disposal, emission, and dumping of BP are required to ensure the protection of bodies of water and to aim for more sustainable processes. In this sense, the quantification of the emissions generated in the industrial sector is of interest, especially in polymer production plants that, year after year, increase their production volumes and focus their research on monitoring the removal of BPA, but even so, this has been ineffective because tens of thousands of nanograms per liter of this compound have been found in industrial wastewater (iWW), including wastewater from paper mills [24,25]. Currently, information related to the identification and quantification of BPs other than BPA in industrial waste remains scarce, and reports on BPs present in waste from petrochemical complexes producing polymers such as polypropylene (PP) and polyethylene terephthalate (PET) are limited, making it difficult to understand the profiles of these molecules from different industrial sources.

In Colombia, there are PP and PET production plants that have a production capacity of thousands of tons per year, and in some of these, contaminants of interest have been found, mainly during the production of polypropylene, which has allowed the observation of the establishment of analytical techniques to recognize and measure different types of contaminants [26–35]. For bisphenol, it is becoming increasingly important to be able to know the levels that are emitted for this contaminant for the implementation of controls and regulations that guarantee greater safety and sustainability of the processes. Unlike PP, no BP values have been reported in residues evaluated from PET plants. Due to the risks to health and the environment that BP generates, it is imperative that the contribution of PP and PET industrial processes in increasing the concentration of this molecule in the environment be evaluated.

In this research, industrial wastewater from two plants located in the industrial sector of Cartagena, Colombia which are producers of PP and PET were selected for the identification of various levels of bisphenol A and different analogous bisphenols that have not been reported in previous research. These plants have WWTPs to treat the water from polymer production, and they have reported that they do not use bisphenol analogs but do use additives that contain similar molecules which can generate bisphenol analogs during resin synthesis. The objectives of this research are to investigate the profiles of BPs in different sources of wastewater and evaluate the removal efficiency of BPs in WWTPs. This research enables reporting for the first time the presence of BPs other than BPA in industrial wastewater producing PP and PET in the industrial sector of Colombia.

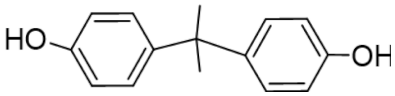
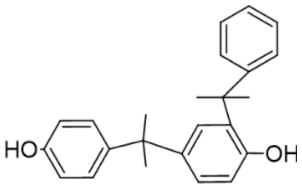
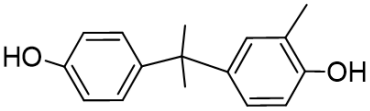
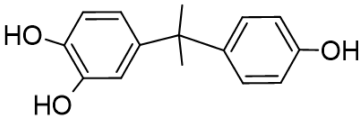
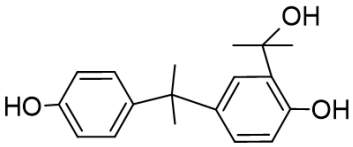
2. Materials and Methods

2.1. Materials

Table 1 shows the five bisphenols to be identified. BPA (STD1) and its internal standards were purchased with isotopic labeling BPA-d16 (>99%) from Superlco (Bellefonte, PA, USA). The four bisphenol A analogs (STD2, STD3, STD4, and STD5) were obtained from AccuStandard (New Haven, CT, USA). For this research, we worked with an internal standard of bis (4-hydroxyphenyl) sulfone-d8 (BPS-d8) and acquired it from TRC (Winnipeg, Canada). Merck (Darmstadt, Germany) purchased HPLC-grade methanol and ammonia water. The water used was ultrapure and was produced using a Milli-Q purification ap-

paratus (Veolia, UK). Each target compound's stock standard solutions were prepared in methanol and then refrigerated at −20 °C.

Table 1. Bisphenols of interest.

ID	Formula	Name	Structure
BPA	$C_{15}H_{16}O_2$	4,4′-(propane-2,2-diyl) diphenol	
D-BPA-1	$C_{24}H_{26}O_2$	4-[2-[4-[2-(4-hydroxyphenyl)propan-2-yl]phenyl]propan-2-yl]phenol	
D-BPA-2	$C_{16}H_{18}O_2$	4-[2-(4-hydroxyphenyl)propan-2-yl]-2-methylphenol	
D-BPA-3	$C_{15}H_{16}O_3$	4-[2-(4-hydroxyphenyl)propan-2-yl]benzene-1,2-diol	
D-BPA-4	$C_{18}H_{22}0_3$	4-[2-(4-hydroxyphenyl)propan-2-yl]-2-(2-hydroxypropan-2-yl)phenol	

2.2. Sampling

Industrial wastewater was collected in January 2022. Two sampling points were established in the PP production plant, and one sampling point was established in the PET plant. In the polypropylene plant, the different sources of wastewater from the process that could contain bisphenols were evaluated, determining two sampling points for the water coming from the pellet cutting system and the condensate from the pellet deodorization column. In the PET production plant, the industrial waters entering the iWWTP from PET synthesis were evaluated. Wastewater samples were collected three times from each processing unit at 9:00 a.m., 12:00 p.m., and 4:00 p.m. The water samples were then combined and separated into three parallel samples. The amount of wastewater discharged to each iWWPT unit was estimated to be approximately 1000 L per day. Industrial wastewater is not pretreated with a disinfectant such as NaClO or ClO_2. These effluents are transferred to iWWPTs through a pipe network. All of our iWW samples that were collected within this investigation were stored in glass bottles. After sampling, the iWWs were treated with 4.0 M H_2SO_4 to adjust the pH to 3.0 and then treated them with a 5%, v/v methanol solution to prevent microbial development. All samples were brought to the lab in ice boxes at a temperature of around 4 °C [36].

2.3. Sample Preparation

A previously established method was used for the extraction and analysis of water samples [37]. A 0.7-µm glass fiber filter (Whatman GF/F) was used to filter the wastewater. The obtained membranes were transferred to 30-mL glass tubes in pieces, and ultrasonic extraction was applied for 10 min. As a solvent, methanol was used followed by methanol:water (5:5 v/v) with 0.1% formic acid. The extracted residue was mixed with the water samples and spiked with BP standards (100 µL at 1 mg/L each) [36].

2.4. Treatment of Samples by SPE

2.4.1. Pretreatment

As a method to reduce microbial activity and facilitate sample preparation, they were then mixed at a temperature of 25 °C and subsequently passed through a 0.22-m Teflon PTFE filter.

2.4.2. Cleaning and Pre-Concentration

This procedure involved conditioning Strata X-33 cartridges (6 mL, 500 mg) with methanol and distilled water, with 5 mL of each one. Then, at a rate of 1 mL min^{-1}, 15 mL of material was uploaded. After percolating the entire sample, 3 mL of MeOH:H_2O was used to rinse the cartridges (80:20). With 10 mL of ACN, the substances retained in the solid phase were eluted. Using nitrogen, the eluate was evaporated until dry. The final volume was 1 mL after being reconstituted with ACN, and the final extract produced a 10:1 pre-concentration [31].

2.5. Instrumental Evaluation

To determine the concentrations of the BPs, HPLC (Agilent, 1100 series, Agilent Technologies Inc., Santa Clara, CA, USA) and a mass spectrophotometer (API 2000, Applied Biosystems, Foster City, CA, USA) were used, followed by an analytical column connected to a Javelin precolumn to perform the CL separation. An injection volume of 10 µL and methanol and water (0.3 mL min^{-1}) as mobile phases were used [13].

2.6. QA and QC (Quality Assurance and Quality Control)

The BP recoveries in the spiked blanks (n = 4) are shown in Table 2.

Table 2. BP recoveries in spiked blanks.

Compounds	Mean
n = 4	
D-BPA-1	75.4 ± 1.44
D-BPA-2	256 ± 1.32
D-BPA-3	45.3 ± 1.7
D-BPA-4	325 ± 5.14

The calculated LOQs for BPA, D-BPA-1, and D-BPA-2 were 0.40 ng L^{-1}, and for D-BPA-3 and D-BPA-4, they were 0.9 ng L^{-1} and 1.5 ng L^{-1}, respectively. To check the sensor sensitivity drift, a halfway calibration standard was injected after every 20 samples. In order to confirm the BP carryover between the samples under examination, pure methanol was added at regular intervals. The calibration of the instruments was checked using daily injections of 10 calibration standards at concentrations ranging from 0.01 to 250 ng mL^{-1}, and the linearity of the calibration curve (r) was more than 0.99 for each component objective. The amounts of BP in the sludge were given as dry weight, and the blank and matrix-enriched samples were consistently examined to validate the analytical approach [13].

Statistical analysis was performed with the data obtained from the concentrations, calculating the mean and the median while assuming as zero the value of the concentrations that were below the LOQ, and for the statistical analysis, a value equivalent to half of each was assigned.

2.7. Removal Extension Calculation

To calculate the average of the removal efficiencies of the BP in the tributaries of the wastewater plant, the equation shown below was used, which uses the average concentration of the BPs in the influents and tributaries of the plants studied [21,38]:

$$\text{Extent of removal (\%)} = [(C_{influent} - C_{effluent})(ng/L)]/[C_{influent}(ng/L)] \times 100 \quad (1)$$

3. Results

3.1. BP Concentrations in WWTPs

The concentrations of the BPs in the different water flows of the different sampling points in Colombia are shown in Table 2. For the tributaries of the different influents, the contents of the bisphenols of interest were verified in each sample, obtaining average values of the sum of the concentration of the five bisphenols studied (Table 3). After going through the preparation of the sample and the established procedure, the concentrations of the bisphenols in the predetermined affluents were analyzed and compared to see if the process used was sufficient to remove these compounds. In the polypropylene production plant, data were obtained at two sampling points: the extruder and the desorber. In the first, the difference between the concentrations of the influents and the effluents was analyzed, obtaining the lowest value for D-BPA-4, which indicates that this was the one that was removed in the greatest proportion compared with the others at this sampling point. The average difference value for bisphenol A was 274.29 ng L^{-1}; for D-BPA-1, it was 117.88 ng L^{-1}; for D-BPA-2, it was 59.76 ng L^{-1}; for D-BPA-3, a difference of 67.06 ng L^{-1} was obtained; and for D-BPA-4, it was 53.82 ng L^{-1}. Unlike the above, in the desorber, the most removed molecule was D-BPA-2, with an average difference between the concentrations of the influents and effluents of 49.69 ng L^{-1}, followed by D-BPA-4 with a difference of 72.88 ng L^{-1}. Meanwhile, for D-BPA-1, the difference was 74.71 ng L^{-1}, and the difference in the highest concentrations were those of BPA and D-BPA-3, with values of 307.94 and 167.69 ng L^{-1}, respectively. In the case of PET, the values were only obtained at one sampling point where, as in the PP extruder, the highest substance removed was D-BPA-4 with a difference of 31.51 ng L^{-1}, followed by D-BPA-3 with 133.71 ng L^{-1} and already having values for BPA, D-BPA-1, and D-BPA-2 of 1623.94 ng L^{-1}, 279.88 ng L^{-1}, and 209.82 ng L^{-1}, respectively. These data suggest that a large percentage of the bisphenols used at some stage of the polypropylene and PET processes is not completely removed. Although these concentrations can be considered low due to the large volumes of water discharged into bodies of water on a constant basis, they can generate a fairly large environmental impact, in addition to affecting the health of the workers and the inhabitants surrounding the plants and bodies of water.

When analyzing the profiles of the concentrations of the different BPs in the different points studied in the polypropylene and PET processes presented in Figure 1, it was observed that the difference in the total concentration of phenols in the influents and effluents was high, with values of 572.82 ng L^{-1} for the PP extruder, 672.91 ng L^{-1} for the PP desorber, and 2278.86 ng L^{-1} in the PET process, indicating that in the three points studied, only a very small amount of bisphenols was removed, and the amount of BPA in the effluents was very high, revealing the few controls that existed for this substance despite being regulated by different control and protection entities. Although no concentration limit has been established for bisphenol analogs, their harmful effects on human health have been studied. The presence of these compounds in bodies of water can affect the surrounding population and the ecosystem where it is being discharged.

Table 3. BP concentrations in influents and effluents.

		Influents						Effluents					
Sampling Point	**Parameters**	**BPA**	**D-BPA-1**	**D-BPA-2**	**D-BPA-3**	**D-BPA-4**	**ΣBPs**	**BPA**	**D-BPA-1**	**D-BPA-2**	**D-BPA-3**	**D-BPA-4**	**ΣBPs**
PP Extruder							(n = 16)						
	Mean	389.88	159.00	85.94	99.47	67.82	802.12	115.59	41.12	26.18	32.41	14.00	229.29
	Median	386	163	86	93	43	797	112	39	25	34	14	227
	Range	312–485	109–198	68–100	57–165	4–465	689–1202	104–148	32–57	16–40	18–43	430–40	202–249
	DR	100	100	100	100	100	100	100	100	100	100	100	100
Desorber PP							(n = 16)						
	Mean	448.35	153.71	86.00	213.35	109.71	1011.12	140.41	79.00	36.31	45.66	36.82	338.21
	Median	444	153	89	206	105	1002	142	81	35	42.1	33.9	354.5
	Range	402–499	112–197	49–124	189–263	76–142	923–1121	99–176	34–114	11.2–63.4	31.2–74.3	10.5–81.3	236.4–418.3
	DR	100	100	100	100	100	100	100	100	100	100	100	100
PET Production							(n = 16)						
	Mean	1949.00	534.82	262.82	180.47	73.79	3000.91	325.06	254.94	53.00	46.76	42.29	722.05
	Median	1254	536	265	169	75	2227	344	256	52	47	31	725.4
	Range	1025–12,478	485–575	203–296	108–478	45–96	2014–13,514	210–391	198–301	45–59	40–52	20–142.5	592–845.5
	DR	100	100	100	100	100	100	100	100	100	100	100	100

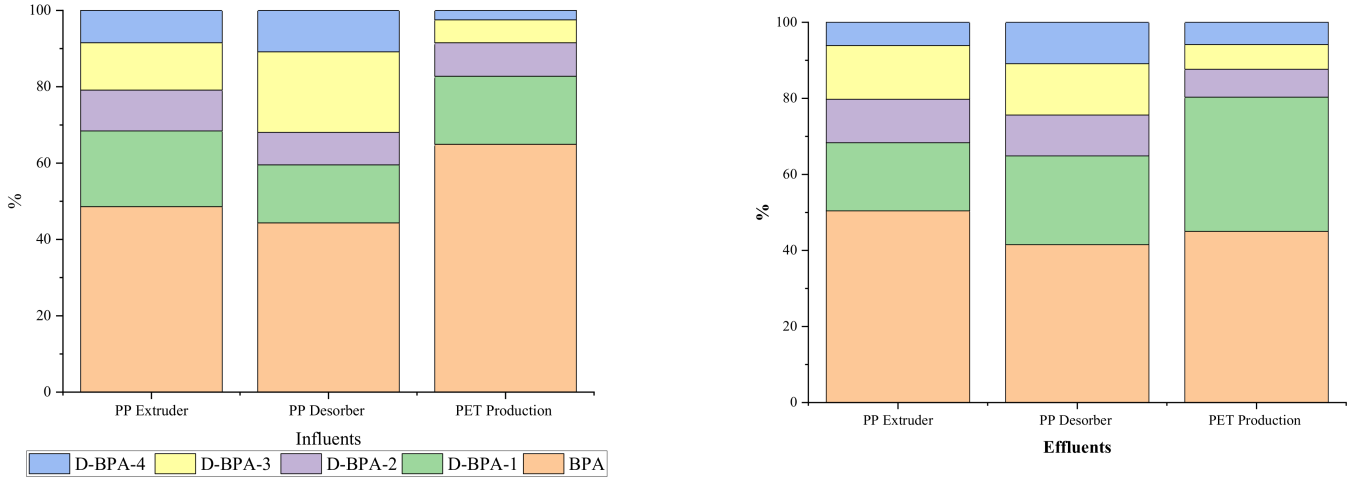


Figure 1. BP concentration profiles: (**a**) influents and (**b**) effluents.

3.2. WWTP Removal Efficiency

Figure 2 shows the removal efficiencies of each bisphenol at the different points studied, which did not reach 60% removal, indicating that a high number of these molecules left the different bodies of water. In the PP extruder, the removal percentages ranged between 26.64 and 34.43%, the lower value being for D-BPA-1 and the higher for D-BPA-3. In the PP desorber, the values ranged between 21.62 and 52.06% for the removal of the compounds, where the compound with the highest removal efficiency was D-BPA-1. The widest range was obtained in the percentages of the removal efficiencies, ranging from 20.34 to 59.16% and with the substance mostly removed being D-BPA-4.

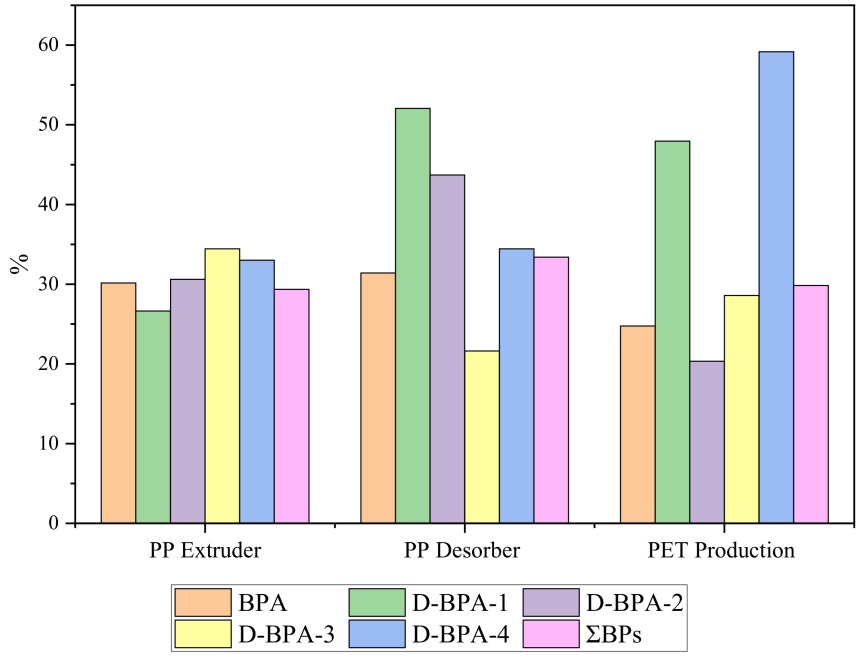


Figure 2. Percentages of BP removal.

In the case of the removal efficiency of the total concentration of bisphenols, they did not reach 40%, demonstrating the inefficiency of these processes for the removal of emerg-

ing contaminants such as bisphenols. These low percentages of efficiency are warning signs that it is necessary to implement processes that allow the identification, quantification, and removal of bisphenols in polymer production processes at an industrial level. The percentages obtained in the removal extension were not far from those obtained by other authors, which varied from 9% to percentages greater than 97% in the different bisphenols [39–41]. The increase or decrease in the extent of removal depends on the treatment used for the wastewater and the ease of separating the molecules during these processes.

4. Conclusions

With this study, we showed for the first time the quantification of bisphenols in industrial wastewater in the production of PP and PET in Colombia, where high concentrations were obtained in the effluents. The presence of these analogous bisphenols in the process wastewater when it was determined that these compounds were not used in the process indicates that they can be formed from other molecules used in the synthesis of the resins, which should be evaluated to implement actions that mitigate the formation of bisphenols. Studies focused on the identification and quantification of bisphenols in industrial waste are of the utmost importance for the implementation of measures to ensure both the process and the health of industrial workers and inhabitants who are affected by these substances, and by their ignorance, they cannot take the necessary measures for their protection.

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