

Wastepaper as a Source for Xenoestrogenic Compounds

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1. ABSTRACT

Endocrine disruption has become one of the most important environmental issues causing scientific, political, legislative, and administrative action. Bisphenol A (BPA), octylphenol, and nonylphenol are three prominent representatives of xenobiotic estrogenic endocrine disrupting compounds (EDCs). Since both BPA and alkylphenol polyethoxylates are used in paper production the contamination of recycled paper products with these compounds can be hypothesised. Concentrations of BPA and four alkylphenolic compounds in toilet paper, wastepaper, and cellulose have been investigated at the Department of Waste Management, Dresden University of Technology, Germany. Preliminary results and calculations are presented.

With one exception, all xenoestrogens studied were determined in all toilet paper samples at very high concentrations of 2 – 430 mg/kg dry mass (DM). The BPA concentrations in seven classes of wastepaper amounted to 0.093 to 5.1 mg/kg DM. In three types of cellulose the EDC concentrations were below or hardly above the respective limit of quantification. Toilet paper, thus, was shown being an important source of xenoestrogen emissions to wastewater. Thermal paper again is estimated being a major source for the contamination of recycled paper with BPA. Because of distinct contamination with xenoestrogens, both paper waste and recycled paper products should not be mixed with biological waste e.g. for co-composting or co-fermentation in order to derive organic fertilisers.

KEYWORDS: estrogenic endocrine disrupters, wastepaper, toilet paper, cellulose, alkylphenol ethoxylates, bisphenol A

2. INTRODUCTION

Municipal wastewater is polluted with a huge number of organic substances of both natural and xenobiotic origin. An increasing number of these compounds is identified to interact with the hormonal (endocrine) system of exposed organisms, e.g. through direct binding to and activation of steroid hormone receptors (Keith *et al.*, 2000; Metzler, 2001; NRC, 1999). Substances mimicking endogenous female sexual hormones, i.e. 17 β -estradiol (E2), via this mode of interaction are called estrogenic endocrine disrupting compounds (estrogenic EDCs). If these substances are not satisfactory removed during wastewater and sewage sludge treatment, sensitive aquatic and terrestrial species can be affected by wastewater treatment plant effluents or surface run-off from agricultural land fertilised with sewage sludge (e.g., SRU, 2002). The most sustainable way to prevent adverse effects of EDCs on health and ecosystems is to prevent the emission of such compounds to the environment.

2,2-bis-(4-hydroxyphenyl)propane (bisphenol A, BPA), 4-*tert*-octylphenol (OP), and 4-nonylphenol (NP) are three prominent representatives of xenobiotic estrogenic EDCs, also called environmental estrogens. BPA is mainly processed to polycarbonate plastics, epoxy resins, and the flame retardant tetrabromobisphenol A, and, in its monomeric form, used as an antioxidant in plasticisers and in the colour developing layer of thermal paper (ThP). OP and NP are degradation products of their respective polyethoxylates (OPnEOs, NPnEOs) that are non-ionic surfactants and used for instance in cleaning agents, as wetting agents, and as emulsifiers, e.g. for de-inking of recovered paper. BPA, OP, NP, and alkylphenol polyethoxylates (APnEOs) are frequently determined in wastewater and sewage sludge (e.g., Kolpin *et al.*, 2002; Tennhardt *et al.*, in press). Only under anaerobic conditions, i.e. during anaerobic digestion of sewage sludge, APnEOs are terminally degraded to the respective alkylphenols (APs). The APs themselves are degraded solely under aerobic conditions. Therefore, the anaerobic treatment of APnEO polluted sewage sludge results in formation and accumulation of APs (Giger *et al.*, 1986; Maguire, 1999; Tennhardt *et al.*, in press).

Since both BPA and APnEOs are used in paper production the contamination of recycled paper products with these compounds can be hypothesised. In Germany, approximately 0.5 Mio t of toilet paper are emitted to wastewater per year (Bilitewski, 2002). The rate of recovered paper input to production of hygiene papers (HyP) amounts to approximately 75 %. Therefore, toilet paper not only represents an important mass flux of solid material into municipal wastewater treatment plants (WWTPs). It also can be suspected being an important source for BPA and APnEOs/APs in wastewater and sewage sludge. In order to verify this hypothesis, the concentrations of BPA and four alkylphenolic compounds (APX) in several sorts of toilet papers, wastepaper, and cellulose are investigated at the Department of Waste Management, Dresden University of Technology, Germany.

3. MATERIALS AND METHODS

Major properties of the toilet paper and cellulose samples studied are briefly summarised in table 1. Three sorts of toilet paper made from 100 % recycled paper have been purchased from local supermarkets in Dresden, Germany, in autumn 2001. Seven classes of wastepaper (WP; table 2) were collected, separated from impurities and sorted out within the scope of an extensive German study completed in 1999. Sampling, preparation, and storage of wastepaper samples have been given in detail elsewhere (Bilitewski, 2001; Schulkies, 2001). Three sorts of import cellulose were kindly supplied by Mr. Rainer Spörl, PTS Paper, Heidenau, Germany.

Table 1. Sample description toilet paper and cellulose

toilet paper	TP1	TP2	TP3
dry matter content [%]	95.2	94.2	94.0
loss on ignition [%]	82.7	98.9	97.9
mass per area [g/m ²]	62.5	68.6	46.8
no. of layers	1, crepe	3	2
cellulose	C1	C2	C3
dry matter content [%]	96.6	96.1	97.3
loss on ignition [%]	99.9	99.7	99.6
wood	spruce, pine	eucalyptus	spruce
disintegration	sulphate	sulphate	sulphite
bleaching	ECF ^a	ECF ^a	TCF ^b

^a elemental chlorine free bleaching

^b total chlorine free bleaching

The methods of sample preparation and EDC determination are described in detail elsewhere (Weltin *et al.*, in press; see also Tennhardt *et al.*, 2003). In the case of paper and cellulose samples, a simplified procedure was found to be optimal: Samples are freeze-dried and extracted by Soxhlet extraction. Clean-up of the extracts by means of silica gel, and derivatisation (silylation) of the dissolved compounds follow. The silyl-methyl derivatives finally are exchanged into 1 ml of toluene containing mirex as internal standard. EDC determination is carried out by means of a gas chromatographic system coupled to a mass selective detector (GC/MS). BPA-d₁₆ and 4-*n*-nonylphenol are used as surrogate standards. Toilet paper samples were prepared using the complete procedure described by Weltin *et al.* (in press) including size exclusion chromatography (SEC). Three 1 – 2 g aliquots of a paper sample were simultaneously extracted and prepared for determination of EDCs. BPA, OP, NP, and, in part, 4-nonylphenol monoethoxylate (NP1EO) and 4-nonylphenol diethoxylate (NP2EO) have been determined in this study. Since attention initially was only turned to BPA and NP, no specific surrogate standard for OP, NP1EO, and NP2EO was added before sample preparation. Therefore, these compounds were also quantified in relation to 4-*n*-nonylphenol. Therefore, whilst the exact values calculated after GC/MS determination are presented in tables and figures, approximate values are discussed in the text. Recovery of surrogate standards (table 3) was calculated as proportion of the mean peak area in all sample aliquots in relation to the mean peak area in all calibration standards.

Table 2. Sample description wastepaper

	dry matter content [%]	loss on ignition [%]	description ^a
WP1	94.6	92.8	corrugated board
WP2	95.6	78.0	advertising supplements
WP3	95.9	68.7	magazines
WP4	96.2	70.7	catalogues
WP5	94.6	98.7	newspapers
WP6	94.7	88.9	free advertising papers
WP7	94.9	86.6	chromo board ^b

^a sample from Dresden, Germany

^b from Neuss-Norf. Germany

Table 3. Recovery of surrogate standards [%]; paper samples, in relation to calibration standards, peak areas

sample	4- <i>n</i> -NP			BPA-d ₁₆		
	recovery [%]	N	p (t-test)	recovery [%]	N	p (t-test)
TP1	3.1	3	0.004	1.1	3	0.006
TP2	1.2	2	0.014	1.1	2	0.02
TP3	5.9	3	0.004	6.2	3	0.007
C1,2,3	115.5	9	0.61	116.6	9	0.40
WP1	122.0	3	0.12	116.8	3	0.19
WP2,3,5,6	94.0	15	0.48	86.8	12	0.22
WP4	94.0	15	0.48	13.7	3	0.00005
WP7	86.7	3	0.24	76.4	3	0.23

Table 4. Limits of detection (LOD) and limits of quantification (LOQ) [µg/l]; paper samples, in GC/MS vial, respectively; for explanation of acronyms see text

[µg/l] in vial		OP	NP	NP1EO	NP2EO	BPA
TP (N = 1)		0.15	0.15	40.83	63.2	99.16
WP (N = 7)	MEAN	0.19	0.65			0.21
	SD	0.03	0.26			0.10
	LOD	0.29	1.44			0.50
	LOQ	0.40	2.22			0.78
C (N = 3)	MEAN	1.59	5.67	4.41	5.83	3.86
	SD	0.35	0.55	0.87	2.22	0.70
	LOD	2.65	7.33	7.01	12.49	5.97
	LOQ	3.71	9.00	9.61	19.15	8.08

4. RESULTS AND DISCUSSION

4.1 Toilet paper

Due to the very poor recovery of the surrogate standards after toilet paper sample preparation (see table 3) most probably caused through the SEC clean-up step, the data reported in this paper have to be regarded as preliminary results. In addition, since concentrations of EDCs were unexpectedly high, i.e. in the case of BPA, chromatographic peaks in part were not clearly quantifiable. Thus, interpretations are subject to extensive restrictions.

With one exception, all xenoestrogens studied were determined in all toilet paper samples at very high concentrations of 2 – 430 mg/kg DM (see table 5). These values exceeded the concentrations in wastepaper by 1 – 2 orders of magnitude (see chapter 4.2). The OP, NP, and BPA concentrations in TP1 and TP2 exceeded those in TP3 by a factor of up to 15, respectively. The concentration of BPA in the toilet paper studied amounted to up to 46.1 mg BPA/kg DM (table 5). Only a minor portion of the BPA of 0.9 – 4.9 % (m/m) was leached from these sorts of toilet paper during 24 h by means of deionised water (data not shown, Gehring *et al.*, 2002).

4.2 Wastepaper

The BPA concentrations in brown and grey corrugated board (WP1), advertising supplements (WP2), magazines (WP3), catalogues (WP4), newspapers (WP5), free advertising papers (WP6), and chromo board (WP7) amounted to 0.093 to 5.1 mg/kg DM (table 5). The highest concentrations of BPA were determined in corrugated board and chromo board (WP1, WP7), whereas the lowest concentrations were detected in magazines and catalogues (WP3, WP4). The OP and NP concentrations in wastepaper amounted to 0.03 – 0.09 mg/kg DM and 0.40 –

Table 5. Concentrations of xenoestrogens in paper samples [mg/kg DM]; for explanation of acronyms see text

[mg/kg DM]	OP	NP	NP1EO	NP2EO	BPA
TP1	2.2	68.9	11.6	74.1	45.5
TP2	5.1	67.5	31.1	428	46.1
TP3	n.d.	14.3	10.0	57.3	3.2
C1	n.d.	n.d.	n.q.	n.d.	(n.q.)
C2	n.d.	n.d.	n.d.	n.d.	(n.q.)
C3	n.d.	0.04	0.06	0.16	(n.q.)
WP1	0.09	0.70			4.23
WP2	0.03	0.48			1.32
WP3	0.06	0.40			0.21
WP4	0.09	1.01			0.09
WP5	0.03	0.96			3.24
WP6	0.03	0.92			2.56
WP7	0.04	0.74			5.10

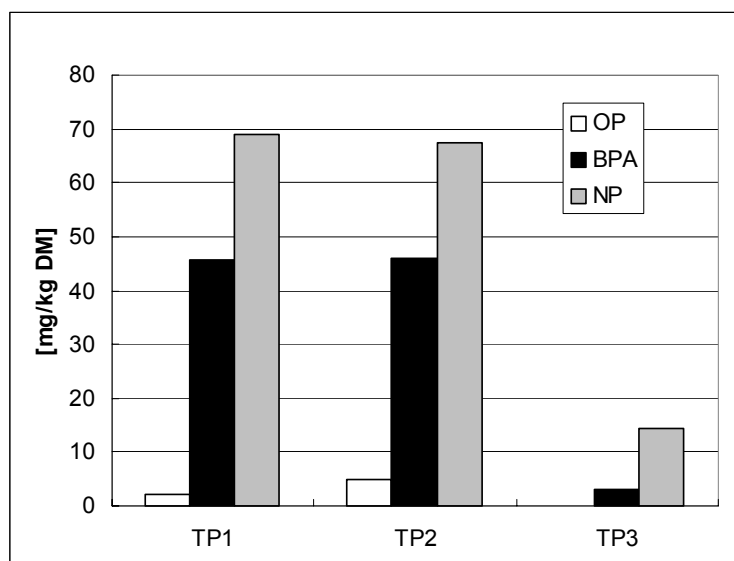


Figure 1. Concentrations of xenoestrogens in toilet paper; for explanation of acronyms see text

1.01 mg/kg DM, respectively. These values were 1 – 2 orders of magnitude lower than the concentrations determined in toilet paper, respectively (see above). No correlation occurred between these three variables. Only a minor portion of the BPA of 0.7 – 8.1 % (m/m) was leached from wastepaper by means of deionised water (data not shown; Gehring *et al.*, 2002).

4.3 Cellulose

In order to prove the hypothesis that the BPA does not derive from natural material, three different types of cellulose subsequently were investigated. Indeed, no or very little amounts of BPA were detectable in the respective triplicates. OP was not detectable in cellulose at all whilst NP, NP1EO, and NP2EO were determined in one of the three samples at 35.7 – 164.3 µg/kg DM (table 5).

4.4 Discussion

The occurrence of OP, NP, NP1EO, and NP2EO as well as the order of magnitude of the relation between the concentrations of OP and NP in toilet paper and wastepaper, respectively, are in accordance with the characteristics of the production and the use of these four alkylphenolic compounds and with literature data (e.g., Bennie, 1999; Krogh *et al.*, 2003). APnEO surfactants, which are industrially produced at very high production volumes, are mostly mixtures of NP and OP polyethoxylates, and contain reasonable impurities of ethoxylates of other alkylphenols (Fiege and Voges, 1991). This mixture is the source material for the production of NPnEO surfactants which again in most cases consist of ethoxylates with a varying length of the ethoxy side chain (Fiege and Voges, 1991). NPnEO surfactants are utilised in wastepaper pulping and de-inking in order to clean the fibres from colours, glues, etc. (Baumann and Herberg-Liedtke, 1993). Paper and board industry discharges in many cases are major point sources for AP and APnEO emissions to surface waters (Bennie, 1999; Maguire, 1999). Since water solubility of APnEOs decreases with the length of the ethoxylate side chain, short-chained APnEOs are expected to adsorb to paper fibres at considerable amounts. Taking the comparable high concentrations of OP, NP, NP1EO, and BPA in TP1 and TP2 into account (see table 5), the concentration of NP2EO in TP2 has to be estimated being exceptional high. The cause for this phenomenon as well as for the high AP concentrations in toilet paper at all is, so far, unexplained.

Since BPA is a constituent of the colour developing layer of the most thermal papers (ThP), it is introduced into the paper cycle through recovering of used thermal paper and, thus, can be expected being determined in recycled paper products. The BPA concentrations in the 7 classes of WP obviously correspond to the respective proportion of wastepaper reused for

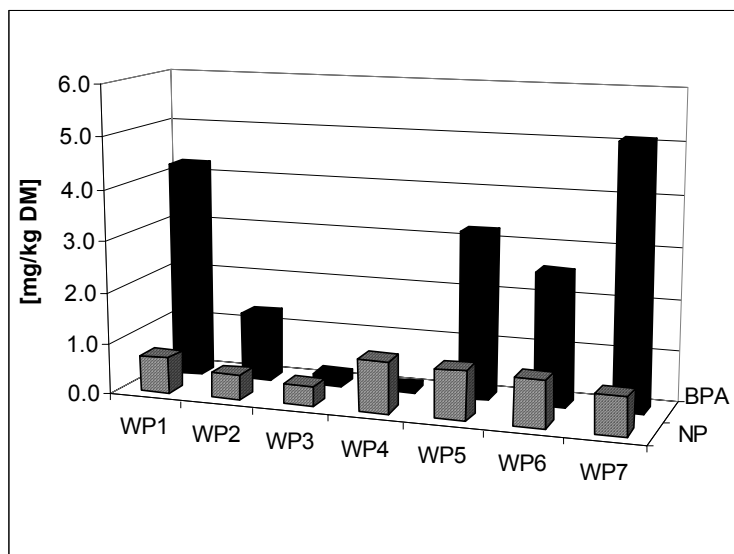


Figure 1. Concentrations of xenoestrogens in wastepaper; for explanation of acronyms see text

production. In the cases of TP1 and TP2, the BPA concentration exceeded the maximum concentration determined in wastepaper by one order of magnitude (see chapter 4.2). Amongst others, accumulation of BPA during the production process, an even higher BPA content in the input material, and a combination of these two possibilities could have caused this observation. Despite of the restrictions to data interpretation described above it can be stated that these BPA concentrations approximately accord with the data published by Vingaard *et al.* (2000) who determined BPA concentrations of 0.6 – 24 mg/kg in 9 recycled paper products for kitchen use (MEAN = 6.21 mg/kg, paper made from 80 – 100 % recycled paper). With regard to the considerable contamination of wastepaper with BPA, the moderate water solubility of BPA (BUA, 1997), and the limited desorption during 24 h leaching experiments we also conclude that long-lasting emissions of BPA from landfills comprising significant masses of wastepaper can occur under anaerobic conditions. BPA is known to be biodegradable under aerobic and anoxic but not under anaerobic conditions (BUA, 1997; Staples *et al.*, 1998; Tennhardt *et al.*, in press).

Approximately 0.5 Mio. t of toilet paper per year are disposed with wastewater in Germany (Bilitewski, 2002). Assuming that the mean BPA concentration in toilet paper in general amounts to 31.6 mg/kg DM as calculated for the three sorts of toilet paper studied, 16.6 t of BPA are annually emitted to wastewater.

In the three types of cellulose studied the BPA and APX concentrations were below or hardly above the respective limit of quantification. These results are in accordance with data published by Vingaard *et al.* (2000) who determined BPA concentrations in virgin paper below 0.1 mg/kg (N = 11). The very low BPA concentrations in cellulose sort 3 as well as the fact that this cellulose was the only one produced by means of total chlorine bleaching and magnesia bisulphite process indicate contamination during production. This contamination could be either inherent in this technology or specific for this certain case.

The terminus graphic wastepaper (grWP) in the following refers to WP2 to WP6 as a whole, the terminus paper and board packaging wastepaper (pbpWP) to WP1 and WP7 as a whole. The four major categories of paper products are hygiene paper (HyP), graphic paper (GrP), paper and board packaging (PBP), and technical paper (TeP). Hygiene paper and technical paper represent minor relevant portions of the entire paper production and, thus, are not discussed in detail in this paper. With recourse to literature data on paper mass fluxes (Bilitewski, 2002; Leisewitz and Schwarz, 1997; VDP, 2002) a preliminary mass balance of the occurrence and distribution of BPA in the paper cycle in Germany, and a theoretical BPA concentration in toilet paper were calculated (for details see Gehring *et al.*, 2003). Table 6 displays a small selection of the data calculated and lists some major assumptions made.

Thermal paper represents a very small portion of the entire paper production but due to its extremely high BPA content it mainly causes BPA input into the paper cycle. Altogether, ~345 t BPA are supplied with wastepaper to the de-inking process, and ~127 t of BPA are inputted with wastepaper and cellulose to paper production. <17 t of BPA are inputted with cellulose. Hygiene paper as well as technical paper is marginally recycled. Including cellulose, 5.4 t of BPA is inputted to toilet paper production. This is only about one third as much as the BPA mass of 16.7 t in toilet paper calculated on the basis of the mean BPA concentration according to table 5 (0.032 kg/t, n = 3) and the entire TP production of 527.000 t (Bilitewski, 2002).

Possible explanations of this discrepancy are:

- i) Sampling was not representative.
- ii) Lack of data: The high number of assumptions that had to be made for the calculation of the mass balance results in essential inaccuracy of calculations. The difference between 16.6 t and 5.4 t can easily occur in consideration of 345 t of BPA inputted to de-inking. Furthermore, several assumptions are known to be very strict, e.g. that the entire wastepaper reused for paper production is de-inked before use and that BPA is equally dispersed during de-inking.
- iii) Additional sources of BPA input to paper production: Approximately 48 % of the BPA inputted to de-inking is detracted from the paper cycle via de-inking sludge. This portion of BPA was further ignored though de-inking sludge is, in part, used as an additive in paper production. At the same time, the extent of BPA removal during paper production, potentially visible from the BPA concentration in paper production waste for disposal (2.9 Mio. t/a; Bilitewski, 2002), is unknown as well.
- iv) To a certain extent, concentrations determined are uncertain because of poor surrogate standard recovery.

Table 6. BPA mass fluxes into paper production with graphic wastepaper, paper and board packaging wastepaper, thermal paper, and cellulose as calculated from data in table 4, 5; for explanation of acronyms see text, calculations see Gehring *et al.* (2003)

Year 2000	grWP	pbpWP	ThP	C
Utilised for paper production [Mio. t/a]	7.4	6.1	0.018	4.2
BPA concentration [kg/t]; except ^a [mg/t]	0.0015	0.0047	16	4 ^a
BPA input to de-inking [t/a] ^b	11.1	28.3	289	
BPA with ThP import input to de-inking [t/a]			16.6	
BPA output of de-inking into paper production [t/a] ^c	34.4	61.5	– ^c	17.1 ^d

^a LOQ in the case of 2 g sample aliquot, see also table 4

^b assumption: entire wastepaper used for paper production is de-inked before reuse; import included except ThP import

^c assumption: BPA mass flux with ThP is inputted to production of the four major paper product classes in same mass proportions as WP is reused for (see Gehring *et al.*, 2003)

^d calculation based on LOQ of BPA concentration and cellulose use for paper production

^e assumptions: equal BPA recovery during de-inking independent on sort of input wastepaper; remaining BPA equally dispersed during de-inking through the entire fibre mass

The hypothetical BPA concentrations in graphic wastepaper and paper and board packaging calculated amount to 0.005 kg/t and 0.01 kg/t, respectively, and thus are essentially higher than the concentrations determined (table 5). In addition to numbers i), ii), and iv) listed above, a higher rate of BPA de-inking than assumed (60 %) and/or a different distribution of BPA between process water and de-inking sludge than assumed (1:4) are possible. No studies on these question are known so far. Nevertheless, the calculated mean BPA concentration in the de-inked fibres (0.010 kg/t) is in a realistic order of magnitude as 0.00009 – 0.051 kg/t were determined in the toilet paper and wastepaper studied. 44 % of the entire wastepaper collected separately is highly contaminated with BPA (pbpWP, 6.1 Mio. t/a).

5. CONCLUSIONS

Toilet paper was shown being an important source of xenoestrogen emissions to wastewater. Thermal paper again is estimated being a major source for the contamination of recycled paper with BPA.

Because of the distinct contamination with the xenoestrogens OP, NP, BPA, NP1EO, and NP2EO, paper waste as well as recycled paper products should not be mixed with biological waste in order to derive organic fertilisers, e.g. via co-composting or co-fermentation (see also Tennhardt *et al.* 2003).

The study presently is continued with repetition of EDC determination in the three sorts of toilet paper and in WP4 excluding SEC clean-up of extracts, and with additional leaching experiments. Future research should investigate i.e. the dissipation pathways of the target compounds within in the paper cycle.

6. ACKNOWLEDGEMENTS

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Remarks:

Page P59, Table 6: Please note that the table gives only a selection of the data calculated. Thus, the values discussed within the text of chapter 4.4 (page P60, last paragraph) are not completely calculable from the figures given in table 6.

Additions:

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