Organochlorine in paper products

Douglas W. Reeve

Given the relatively low concentration of chlorinated organic matter in paper products which will be mobile under conditions of use, and the very small fraction which might be transferred to a user, no significant health risk is anticipated.

When kraft pulp is bleached with chlorine or chlorine-containing compounds, most of the chlorine atoms (Cl) applied in some form of bleaching chemical will end up as chloride ion, while some chlorine atoms become covalently bound to carbon atoms in the pulp. A portion of the covalently bound Cl, "organochlorine," will appear in the bleach plant effluent, where it is measured as AOX (adsorbable organic halide) (*1–5*). Some covalently bound Cl will remain in the pulp, as recognized by a number of studies dating back to the Sixties (*6–8*). A recent survey of commercial, chlorine-bleached kraft pulps showed total covalently bound Cl in pulp to be in the range 200-2300 μg/g o.d. pulp (*9*). Only a small fraction was extractable by water, 4-6%, while 50–70% was extractable by organic solvents. The presence of such "organochlorine" in chlorine-bleached kraft pulp inevitably leads to "organochlorine" in paper made from this pulp.

There has been considerable concern about the environmental impact of AOX discharged in effluents, which has led to regulations limiting AOX discharge in several countries. There have been many investigations concerning organochlorine in effluents, and there is still substantial controversy about the role and importance of organochlorine in environmental impact. There has also been great concern about chlorinated dioxins and furans in effluents, and in pulp and in paper products. Market forces have been pressing for diminished use of chlorine and chlorine-containing compounds in bleaching and advocating use of unbleached paper products. There has been a call for "chlorine-free" paper products by environmental groups (*10–12*) and the labeling of paper products as "chlorine-free," "non-chlorine bleached," "bleached without chlorine," etc., by consumer groups, producers, retailers, and government agencies.

There has been very little published about organochlorine in pulp or in paper products. Some work has been published on chlorinated wood extractives, fatty acids and resin acids, in pulp and their influence on pulp processing and pulp properties (*6–8*). The chlorinated extractives in bleached pulp have been characterized by several techniques (*13*) and a number of compounds identified (*14*). Small concentrations of chlorophenols have been found in bleached kraft and sulfite pulps (*15, 16*). There have been many publications on chlorinated dioxins in pulp and paper products; particularly on their occurrence and on means of minimizing their formation. As a result of newly implemented bleaching technology, dioxins are not detectable in many present day pulps. Several studies have concluded that dioxins in paper products are not a health risk to users (*17–18*). There have been reports of traces of polychlorobiphenyls (PCBs), pentachlorophenol (PCP) and hexachlorocyclohexane (BHC) in paper products. The origin of these compounds is not related to pulp bleaching but rather, respectively, to the use of carbonless copy paper, to wood preserving with PCP and to the use of BHC as a pesticide (*19–23*).

Our concern in this paper is the measurement and functional characterization of organochlorine in paper products. How much covalently bound chlorine is present? How much is water extractable? How much is solvent extractable? Is all the organochlorine from the bleaching process? We must learn more about the character of this organochlorine in order to consider its effects and fate through the use and disposal phases of paper product life cycles. With this understanding, appropriate responses may be made to minimize or eliminate any adverse effects.

Procedures

The 60 paper-product samples tested in this study were all purchased from retail stores. The procedures for measurement of various forms of Cl have been described in detail previously, as applied to pulp samples (*9*). All Cl measurements in solid samples were made by neutron activation analysis. Total Cl concentration was measured in samples straight out of the package. Samples were then extracted with water; at 0.5% consistency and room temperature for 2 h, with continuous agitation. The pulp was filtered and water-inextractable Cl was measured. Water-inextractable Cl is assumed to be covalently bound Cl. AOX was determined in the filtrate using a Mitsubishi TOX analyzer. Total covalently bound Cl is calculated from the sum of water-inextractable Cl plus water-extractable AOX. Water-extracted samples were then extracted with ethanol:toluene 1:1 in a Soxhlet extractor. Solvent-inextractable Cl in the sample was measured by

Reeve is director of the Pulp and Paper Centre, Department of Chemical Engineering and Applied Chemistry, University of Toronto, 200 College St., Toronto, ON M5S 1A4, Canada.

I. Chlorine in paper towel — μg/g o.d. towel

Brand	Type	Cl total, covalently bound	Water-extracted AOX	Cl extracted by EtOH/toluene	Total weight extracted by EtOH/toluene	Cl in extract, %
A	White	540	17	230	3500	7
B	White	570	26	190	3200	6
C	White	510	9	150	2000	7
D	White	410	13	190	2500	8
E	White	700	25	250	3600	7
F	White	880	18	580	...	...
G	Recycled paper (Sweden)	210	7	60	1900	3
H	Recycled paper (Sweden)	220	7	90	3500	3
I	Chlorine-free (Germany)	38	8	N.D.(40)	260	

II. Chlorine in facial tissue paper — μg/g o.d. tissue

Brand	Type	Cl total, covalently bound	Water-extracted AOX	Cl extracted by EtOH/toluene	Total weight extracted by EtOH/toluene	Cl in extract, %
A	White	510	12	220	1600	14
B	Pale yellow	640	17	360	1600	23
C	White	420	19	130	1600	8
D	White	660	31	330	2200	15
E	White	630	25	...	2100	...
F	White	510	16	170	2400	7
G	White	740	21	430	3400	13

III. Chlorine in toilet tissue paper—μg/g o.d. paper

Brand	Type	Cl total, covalently bound	Water-extracted AOX	Cl extracted by EtOH/toluene	Total weight extracted by EtOH/toluene	Cl in extract, %
A	White	340	15	100	4300	2
B	White	450	29	240	1500	16
C	White	470	21	150	2100	7
D	100% Recycled paper (Canada)	410	13	140	2500	6
E	100% Recycled paper (Germany)	370	7	180	2800	6
F	100% Recycled paper (Germany)	160	5	83	3800	2

neutron activation analysis. Extractable covalently bound Cl was calculated by difference. The weight of extract was determined by evaporating the solvent and the Cl content of the extract was calculated.

The detection limit by neutron activation analysis was 30 μg/g Cl/g and, for AOX, 4 μg/g (95% confidence limit). It should be noted that Cl in pulp or paper samples can also be determined by combustion either by the classical oxygen flask technique or by combustion in an AOX analyzer (*9*, *24*). The detection limit for the latter is 3 μg/g.

Chlorine in personal-use products

The results of Cl determinations in several types of personal-use products—paper towels, facial tissue, toilet paper, feminine napkins, diapers and serviettes—are given in **Tables I-VI**.

White paper toweling contained 400-900 μg/g total covalently bound Cl of which 10-30 μg/g was water-extractable and carbon-adsorbable, i.e., AOX, and 150-600 μg/g was solvent extractable. This compares closely with early measurements of Cl on commercial pulp samples; for instance, fully bleached softwood pulps were found to contain, on the average, 500 μg/g total covalently bound Cl/g of pulp, 25 μg/g AOX/g and 225 μg/g solvent-extractable Cl/g. Toweling made from recycled papers contained somewhat less covalently bound Cl in each category. Presumably this paper was made from recovered fiber which had previously been chlorine bleached and, perhaps, the recovered fiber was bleached with hypochlorite during recycling. The "100% chlorine-free" toweling from Germany contained measurable covalently bound Cl although the values were close to the detection limit.

Tables II and III present similar findings for facial tissue and toilet tissue papers. For white paper from virgin fiber, the Cl data are comparable to the data for toweling. Paper from recycled fiber contained comparable concentrations.

Table IV contains Cl data for various feminine napkins. The standard products contained somewhat less total covalently bound Cl than toweling, 200-400 μg/g Cl/g. Water-extractable AOX was about the same while solvent-extractable Cl concentration was significantly lower. Products "bleached-without-chlorine" were nondetectable in chlorine in most instances. Note that for these products there was still a significant amount of solvent-extractable material, 1400-1800 μg/g of product. By using the combustion and microcoulometric technique in an AOX analyzer, the detection limit for Cl in paper samples decreases to 3 μg/g. Unbleached kraft and CTMP pulps have been found to contain 4-13 μg/g covalently bound Cl/g pulp. It is expected that the "bleached-without-chlorine" products, most likely made from hydrogen peroxide bleached CTMP, will contain such concentrations. Appropriate tests are being conducted.

Analysis of disposable diapers in a similar way proved impossible. The diapers that were tested contained super-absorbent material which led to gel formation on contact with water. This prevented further testing. As a result, only the total Cl in the original sample was measured and is reported in Table V. This value will include chloride ion and covalently bound Cl.

There were several anomalies found in the data on serviettes, as reported in Table VI. Serviettes made from recycled paper had significantly higher Cl concentration than was expected. This may be due to wet-strength additive, epichlorohydrin. It is interesting that the serviette labeled "made from unbleached pulp" contained the same concentration of covalently bound Cl as would be expected from a chlorine-bleached product. This may also be due to wet strength additives.

Chlorine in food-related products

A variety of products for containing, storing, and serving food have been analyzed. In **Table VII**, the data for various products contained several anomalies. The total and solvent-extractable Cl in the paper bowl samples was very high. These data are comparable to birch market pulp samples measured previously: 2300 μg/g total covalently bound Cl/g pulp and 1800 μg/g solvent-extractable Cl/g pulp (*9*). Another anomaly is the very high concentration of solvent-extractable material in the cups, bowls, and milk carton. This is most likely due to the wax or other coating materials used to make these products. Note the unbleached bag extractives content is also rather high and there is no detectable Cl in the unbleached bag.

Several types of coffee filters were analyzed. The Cl data are given in **Table VIII**. Coffee filters made from bleached pulp contained concentrations of covalently bound Cl which one might expect from softwood pulps. However, the total extractives and solvent-extractable Cl concentrations were particularly low. This is likely due to the especially good washing which is employed in preparing pulp for this application. An analogous product is condenser paper made from unbleached pulp, which is also washed exceedingly thoroughly, decreasing the extractives content to approximately 300 μg/g or 0.03%. This is much lower than the typical extractives content of unbleached softwood pulp.

Our analysis for the Cl content of tea bags provided a particularly startling result as shown in **Table IX**. The total covalently bound Cl was found to be in a range 25,000 to 90,000 μg/g or 2.5-9% Cl on pulp. This high Cl concentration is attributed to addition of 10 to 15% polyvinyl chloride(PVC) copolymer fiber or vinyl chloroacetate (vinyon) which permits heat sealing the tea bags.

In paper products, there may be chloride ion which would be measured in the original sample but washed out by the water extraction. In most of the samples tested, chloride was nondetectable (at 40 μg/g detection limit) or less than 100 μg/g. Bleach board products—cups, bowls, plates, and milk cartons—contained chloride at an average of 700 μg Cl/g.

Characterization of extractable organochlorine

Some characterization of the solvent-extractable organochlorine has been done. In a study of 10 commercial pulps, the solvent-extracted organochlorine was found to be highly diverse, comprising many individual com-

IV. Chlorine in feminine napkins—μg/g o.d. napkin

Brand	Type	Cl total, covalently bound	Water-extracted AOX	Cl extracted by EtOH/toluene	Total weight extracted by EtOH/toluene	Cl in extract, %
A	...	180	12	60	1300	5
B	...	250	15	80	7500	1
C	...	320	20	40	2700	2
D	...	300	14	40	450	9
E	...	410	41	90	740	12
F	Bleached without chlorine (Sweden)	N.D.[a] (30)	N.D. (4)	N.D. (40)	1400	...
G	Bleached without chlorine (Sweden)	N.D. (30)	N.D. (4)	N.D. (40)	1800	...
H	Bleached without chlorine (Sweden)	N.D. (30)	7	N.D. (40)	1500	...
I	100% Non-chlorine bleached (Canada)	N.D. (30)	10	N.D. (40)	1500	...

[a]Non-detectable.

pounds over a wide range of molecular weights and having an average molecular weight range from 300 to 900. Chlorine content was from 7% to 25%. More than 90% was highly lipophilic. There were some acid compounds and some neutral compounds. The aromaticity of the extracts was low (*13*).

Specific compounds are highly dependent on wood species. Most of the compounds identified to date are the chlorination products of wood extractives (*14*). These compounds are generally formed by addition of Cl_2 or HOCl across double bonds. Some extractive compounds have more than one double bond. Thus for a given wood extractive, there will be multiple congeners, e.g., for linoleic acid, there will be mono-, di-, tri- and tetra-chloro-congeners (*25*). Chlorohydrins, having Cl and OH on adjacent carbons, are common. Chlorinated fatty acids are prominent in a number of extracts tested (*14*). These compounds have also been found in flour treated with chlorine to enhance baking properties (*26*).

The identity of the water-extractable AOX has not yet been determined. It is likely comprised mainly of water-soluble fragments of chlorinated lignin, chlorophenolic structures, and non-aromatic fragments, perhaps of higher molecular weight to account for the failure of this material to dissolve during bleaching. Some of this AOX may also be dislodged, water-insoluble particles, i.e., chlorinated pitch. Our work on identification of mobile organochlorine is continuing.

Behavior of chlorine during use of paper products

The mobility of covalently bound chlorine in paper products during use of the product will vary widely; depending, of course, on the design and use of the product and also on the origin and properties of the organochlorine. Some paper products will be aggressively extracted with water during use, e.g., coffee filters. Some will be exposed for long periods to fatty materials, e.g., bacon board. Some will not be exposed to any extracting substances and will contact users only briefly, e.g., book

V. Chlorine in diapers—μg/g o.d. material

Brand	Type	Total in original sample
A	...	400
B	...	330
C	...	560
D	Chlorine-free (Sweden)	66
E	Chlorine-free (Canada)	75

paper. As demonstrated in the laboratory work reported above, some of the covalently bound Cl is mobile, i.e., the water-extractable AOX and the solvent-extractable Cl. Both extraction tests are very severe compared to exposure conditions during virtually all uses of paper products. The aqueous extraction is carried out in a dilute suspension of individual fibers with vigorous agitation for 2 h. The solvent extraction is carried out on comminuted samples using a very effective solvent mixture, ethanol/toluene, with freshly regenerated solvent as provided by the Soxhlet extractor, for 6 h. These techniques were devised to extract exhaustively, and the results will represent the amount of material ultimately extractable. During paper product use, exposure will not involve disintegrated samples that are thoroughly and intimately combined with the extracting substance continuously over long periods. In most instances of paper product use, only the surface or small segments of the paper product will be exposed for brief periods. Further, in many food packaging paper products a liner, coating or other barrier is used to help contain the food and will simultaneously be a barrier to migration of organochlorine into the food. Thus the water-extractable AOX and the solvent-

VI. Chlorine in serviettes—μg/g o.d. material

Brand	Type	Cl total, covalently bound	Water-extracted AOX	Cl extracted by EtOH/toluene	Total weight extracted by EtOH/toluene	Cl in extract, %
A	White	240	8	80	2100	4
B	Made with unbleached pulp (Germany)	290	9	70	720	10
C	Made from recycled paper (Germany)	1200	14	580	3500	17

VII. Chlorine in paper food serving products—μg/g o.d. material

Brand	Type	Cl total, covalently bound	Water-extracted AOX	Cl extracted by EtOH/toluene	Total weight extracted by EtOH/toluene	Cl in extract, %
A	Waxed cup	620	23	190	21,900	0.9
B	Paper bowl	1600	N.D. (4)	1200	7000	17
C	Paper plate	760	27	290	5600	5
D	White lunch bag	740	21	400	3500	11
E	Unbleached brown lunch bag	N.D.[a] (30)	N.D. (4)	N.D. (40)	8500	...
F	Milk carton	580	14	N.D. (40)	5100	...
G	Egg carton	140	N.D. (4)	100	15,700	0.6

[a]Non-detectable.

extractable Cl represent the maximum mobile organochlorine while the actual Cl that migrates will be only a fraction of the above values.

Data on migration of organochlorine from paper products to food or to users is not presently available. The National Council for Air and Stream Improvement (NCASI) of the Paper Industry in the United States has determined rates of migration of chlorodioxins from various food containment systems. Organochlorine migration studies will be undertaken in our laboratory as part of the continuation of our research.

A significant fraction of the covalently bound Cl was not extractable in these laboratory tests and therefore will be immobile during use. This fraction will therefore not be transferred to foods or users. Only by ingestion would the inextractable organochlorine be transferred to users and even then it is unlikely to be bioavailable. The inextractable Cl is thought to be bound to lignin fragments attached to carbohydrate, i.e., lignin-carbohydrate complexes.

The ultimate question concerning the behavior of organochlorine in paper products is: what, if any, is the hazard to users? Assessing the hazard or relative risk is a complex process involving many steps and requiring a great deal of very specific information. First, we must know the identity and concentration of the compounds in question and then the amount transferred in a given application. For estimating exposure to people, we must know the frequency of use of paper products of various types. Once the estimate of material contacting the individual is made, bioavailability and toxic effects must be estimated. This analysis leads to an assessment of relative risk. This has been done by NCASI for dioxin in paper products (*18*). For organochlorine, generally, there is much less information available. Our efforts to obtain more information and establish estimates of risk are continuing.

Given the relatively low concentration of chlorinated organic matter in paper products which will be mobile under conditions of use and the very small fraction which might be transferred to a user, no significant health risk is anticipated. PVC is used in many applications, for instance domestic water supply piping, where the organochlorine is immobile under conditions of use; therefore, there is no contamination and so no health risk.

VIII. Chlorine in coffee filters—μg/g o.d. filter

Brand	Type	Cl total, covalently bound	Water-extracted AOX	Cl extracted by EtOH/toluene
A	White	310	8	70
B	White	400	20	60
C	White	94	5	67
D	Made from unbleached pulp	N.D.[a] (30)	N.D. (4)	N.D. (40)

[a]Non-detectable.

IX. Chlorine in tea bags—μg/g o.d. bag

Brand	Cl total, covalently bound	Water-extracted AOX	Total weight extracted by EtOH/toluene
A	25,500	9	5800
B	70,000	N.D.[a] (4)	9700
C	90,000	N.D. (4)	8200
D	70,000	8	13,800
E	70,000	5	7300
F	300	6	2700

[a]Non-detectable.

The disposal phase

What happens to Cl covalently bound in paper products, after use, in the disposal phase? What is its ultimate fate in the environment?

One path of disposal, leading to ultimate destruction, is incineration. Municipal waste contains up to 6% Cl; organochlorine from plastics, mainly polyvinyl chloride, and from bleached kraft paper and, most likely, some inorganic Cl. Some Cl in the municipal waste fed to an incinerator may end up as chlorodioxin in the flue gas even though there are process strategies and controls for burning waste to minimize chlorodioxins. Any Cl present in combustion has the potential to form chlorodioxins. It should be noted that high ash coals can contain several percent Cl and that even using a conservative 0.1% Cl in coal, annual coal combustion Cl content far exceeds Cl content of municipal waste combustion, as shown in **Table X**. Paper is a significant fraction of municipal waste but chlorine-bleached paper products will contain less than 0.1% Cl (1000 μg/g). PVC on the other hand, contains as much as 57% Cl. As Table X shows, if all the bleached kraft pulp was combusted, the organochlorine in it would contribute less than 1/100 of what coal contributes. We might therefore conclude that organochlorine in paper is not a major contributor to chlorodioxins from combustion.

Landfill is a major disposal pathway for chlorine-bleached paper products. In some landfills, little biological activity takes place and paper products and other waste materials are preserved. Perhaps the bleached paper products and the organochlorine within them will remain unchanged indefinitely. This does not address the issue of ultimate fate very well, but neither does a landfill. We must determine the ultimate fate of organochlorine in discarded paper products. How susceptible is it to biological degradation and to physical degradation—hydrolysis, oxidation, and photolysis? The philosophy of reuse, recycle, and recovery is an increasingly heralded alternative to landfilling. Because kraft pulping and bleaching preserves the strength of fibers, it is ideally suited to produce stronger, therefore more re-usable, fibers. We must then ask the fate of organochlorine through recycling processes.

We must not lose sight of the fact that substantial quantities of organochlorine are formed in the manufacture of PVC, solvents and other products, as shown in **Table XI**. Chlorination of water results in formation of organochlorine. Further, large quantities of organochlorine are formed naturally in the environment and are therefore a natural part of the ecosystem, and so are also naturally destroyed (*34*, *35*). It may be that there is no significant environmental risk from organochlorine in paper products.

Concluding remarks

Our knowledge of the character and behavior of covalently bound Cl in paper products is increasing and bleaching systems are being implemented, such as 100% ClO_2 bleaching, which decrease its formation. More information is required to understand the effects and the fate of this material to ensure protection of users and the environment while attempting to sustain viable paper manufacturing processes.□

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X. Chlorine in combustion processes (global)

	Total production, 10^6 tons/year	Cl concentration	Cl, 10^3 tons/year	Reference
Coal	4200	0.1%	4200	27, 28
Oil	3000	1 μg/g	3	28, 29
Fuel wood[a]	360	50 μg/g[b]	18	28
MSW[c] incinerated	100	0.5%	500	28, 30
For comparison				
All bleached kraft pulp	54	500 μg/g	30	31

[a]Wood burned in forest fires not included.
[b]Range 10-100 μg/g
[c]Municipal solid waste; does not include industrial waste.

XI. Organochlorine-containing products (USA)

	Production, 10^3 tons/year	Cl concentration	Cl, 10^3 tons/year	Reference
Vinyl chloride monomer	4100	56%	2300	32, 33
Solvents and organic intermediates	1600	87%	1400	32, 33
Chlorofluorocarbons	400	50%	200	32, 33
Total			3900	
For comparison				
Bleached kraft pulp	35,000	500 μg/g	20	31

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