SUPPORTING INFORMATION

From Clothing to Laundry Water: Investigating the Fate of Phthalates, Brominated Flame Retardants and Organophosphate Esters.

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Summary

The accumulation of phthalate esters, brominated flame retardants (BFRs) and organophosphate esters (OPEs) by clothing from indoor air and transfer via laundering to outdoors were investigated. Over 30 days cotton and polyester fabrics accumulated 3300 and 1730 ng/dm^2 $\Sigma_5$phthalates, 65 and 77 ng/dm^2 Σ_{10}BFRs, and 830 and 290 ng/dm^2 Σ_8OPEs, respectively. Planar surface area concentrations of OPEs and low molecular weight phthalates were significantly greater in cotton than polyester and similar for BFRs and high molecular weight phthalates. This difference was significantly and inversely correlated with K_{OW}, suggesting greater sorption of polar compounds to polar cotton. Chemical release from cotton and polyester to laundry water was >80% release of aliphatic OPEs (log K_{OW} <4), <50% of OPEs with an aromatic structure, 50–100% of low molecular weight phthalates (log K_{OW} 4-6), and <detection–35% of higher molecular weight phthalates and BFRs (log K_{OW} >6). These results support the hypothesis that clothing acts as an efficient conveyer of soluble semi-volatile organic compounds (SVOCs) from indoors to outdoors through accumulation from air and then release during laundering. Clothes drying could also contribute to the release of chemicals emitted by electric dryers. The results also have implications for dermal exposure.

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(except TCEP and TnBP) and BFRs, respectively, whereas remaining markers are phthalates. Red dotted line indicates zero on vertical axis.

Figure S5: Difference in chemical accumulation, $(C_{cotton} - C_{polyester})/C_{cotton}$, normalized to planar surface area, plotted against (a) polarizability (latter values not available for all chemicals), and (b) K_{OA}. Red dotted line indicates zero on vertical axis.

Figure S6: Difference in chemical accumulation, $(C_{cotton} - C_{polyester})/C_{cotton}$, normalized to BET-SSA plotted against the (a) Henry's law constant (HLC, Pa-m^3/mol), (b) solubility (mg/L) and (c) octanol-water partition coefficient (Log K_{OW}). TCEP, an outlier, was excluded from solubility and Log K_{OW} graphs. In (a), green and purple ellipses indicate OPEs (except TCEP and TnBP) and BFRs, respectively, whereas remaining markers are phthalates. Red dotted line indicates zero on vertical axis.

Figure S7: Percentage of accumulated chemical released to laundry water as a function of water solubility (mg/L). Black, green and purple ellipses indicate phthalates, OPEs and BFRs, respectively.

Figure S8: Percentage of accumulated chemical released to laundry water as a function of Henry's law constant (HLC, Pa-m3/mol).

Figure S9: Percentage released of accumulated chemical to laundry water as a function of percentage in the gas phase measured by Saini et al. (2015, 2016).

Figure S10: The difference of chemical accumulation from air (Ccotton – Cpolyester)/Ccotton, normalized to planar surface area of fabric, plotted against the percentage released to laundry water. The dotted red line indicates zero on horizontal axis.

Figure S11: The difference in chemical accumulation, (Ccotton – Cpolyester)/Ccotton, normalized to BET-SSA plotted against the percentage released to laundry water. The dotted red line indicates zero on horizontal axis.

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Methods

Table S1. Details of chemicals analyzed. Values of water solubility and log K_{OW} were estimated using USEPA EPI Suite's models WSKOWWIN v. 1.42 and KOWWIN v.1.68.

	Full name	CAS number	Molecular weight (g/mol)	Water solubility mg/L at 25°	Log K_{ow}
Organophosphate esters (OPEs)					
TnBP	Tributyl phosphate	126-73-8	266	7.4	3.8
TCEP	Tris(2-chloroethyl) phosphate	115-96-8	285	878	1.6
TCPP-1 (TCiPP)	Tris(2-chloroisopropyl) phosphate	13674-84-5	328	52	2.9
TCPP-2		76025-05-6	328	52	2.9
TCPP-3		76649-15-5	328	52	2.9
TPhP	Triphenyl phosphate	115-86-6	326	1.0	4.7
TDCiPP	Tris(1,3-dichloro-2-propyl) phosphate	13674-87-8	431	1.5	3.7
EHDPP	2-Ethylhexyl diphenyl phosphate	1241-94-7	362	0.07	6.3
New flame-retardants (NFRs)					
ATE (TBP-AE)	Tribromophenyl allyl ether	3278-89-5	370	$7.8x10^{-2}$	5.6
PBBz	Pentabromobenzene	608-90-2	472	$3.4x10^{-3}$	6.4
PBT	Pentabromotoluene	87-83-2	486	$9.3x10^{-4}$	7.0
PBEB	Pentabromoethylbenzene	85-22-3	501	$2.9x10^{-4}$	7.5
HBB	Hexabromobenzene	87-82-1	551	$2.2x10^{-3}$	7.3
TBB (EH-TBB)	Ethylhexyl-tetrabromobenzene	183658-27-7	550	$1.1x10^{-5}$	8.8
TBPH (BEH-TEBP)	Bis(2-ethlyhexyl)tetrabromophthalate	26040-51-7	706	$1.9x10^{-9}$	12.0
s-DP (s-DDC-CO)	Syn-Dechlorane plus	13560-89-9	653	$1.7x10^{-8}$	11.27
a-DP(a-DDC-CO)	Anti-Dechlorane plus				
OBIND (OBTMPI)	Brominated trimethylphenyl indane	893843-07-7 108488-51-9 102595-65-3	867	$1.9x10^{-11}$	13.0
DBDPE	Decabromodiphenylethane	84852-53-9	971	$1.2x10^{-12}$	13.64

Polybrominated diphenyl ether (PBDEs)					
BDE-17	2,2',4-Tribromodiphenyl ether	147217-75-2	407	$2.6x10^{-2}$	5.88
BDE-28	2,4,4'-Tribromodiphenyl ether	41318-75-6	407	$2.6x10^{-2}$	5.88
BDE-71	2,3',4',6-Tetrabromodiphenyl ether	189084-62-6	486	$1.5 x10^{-3}$	6.77
BDE-47	2,2',4,4'-Tetrabromodiphenyl ether	5436-43-1	486	$1.5 x10^{-3}$	6.77
BDE-66	2,3',4,4'-Tetrabromodiphenyl ether	189084-61-5	486	$1.5 x10^{-3}$	6.77
BDE-100	2,2',4,4',6-Pentabromodiphenyl ether	189084-64-8	565	$7.9 x10^{-5}$	7.66
BDE-99	2,2',4,4',5-Pentabromodiphenyl ether	60348-60-9	565	$3.9 x10^{-4}$	7.66
BDE-85	2,2',3,4,4'-Pentabromodiphenyl ether	182346-21-0	565	$7.9 x10^{-5}$	7.66
BDE-154	2,2',4,4',5,6'-Hexabromodiphenyl ether	207122-15-4	644	$4.2 x10^{-6}$	8.55
BDE-153	2,2',4,4',5,5'-Hexabromodiphenyl ether	68631-49-2	644	$4.2 x10^{-6}$	8.55
BDE-138	2,2',3,4,4',5'-Hexabromodiphenyl ether	182677-30-1	644	$4.1 x10^{-6}$	8.55
BDE-183	2,2',3,4,4',5',6-Heptabromodiphenyl ether	207122-16-5	722	$2.1 x10^{-7}$	9.44
BDE-190	2,2',3',4,4',5',6-Heptabromodiphenyl ether	83992-70-5	722	$2.1 x10^{-7}$	9.44
BDE-209	Decabromodiphenyl ether	1163-19-5	959	$2.8 x10^{-11}$	12.11
Phthalates					
DiBP	Di isobutyl phthalate	84-69-5	278	5.1	4.46
DnBP	Di-n-butyl phthalate	84-74-2	278	2.3	4.61
BzBP	Benzyl butyl phthalate	85-68-7	312	0.9	4.84
DEHP	Di (2-ethylhexyl) phthalate	117-81-7	391	$1.1x10^{-3}$	8.39
DiNP	Di isononyl phthalate	68515-48-0	419	$2.3x10^{-5}$	9.37

Table S2. Instrument detection limits (IDL) and limits of quantification (LOQ) of chemicals analyzed.

	IDL (pg or *ng)	**LOQ (pg or *ng)**
TnBP	20	67
TCEP	234	779
TCPP-1	94	314
TCPP-2	112	375
TCPP-3	115	385
TPhP	76	253
TDCiPP	14	48
EHDPP	83	276
ATE	1.1	3.8
PBBz	2.1	6.9
PBT	2.5	8.5
PBEB	3.0	9.9
HBB	3.7	12
TBB (EH-TBB)	13	44
TBPH (BEH-TEBP)	33	111
s-DP (s-DDC-CO)	2.3	7.7
a-DP(a-DDC-CO)	3.4	11
OBIND (OBTMPI)	11	38
DBDPE	209	696
BDE-17	2.8	9.3
BDE-28	2.1	6.9
BDE-71	5.1	17
BDE-47	5.5	18
BDE-66	5.8	19
BDE-100	13	44
BDE-99	5.6	19
BDE-85	11	36
BDE-154	6.8	22
BDE-153	7.6	25
BDE-138	12	42
BDE-183	5.8	19
BDE-190	16	55
BDE-209	36	119
DiBP*	0.15	0.50
DnBP*	0.15	0.49
BzBP*	0.27	0.89

DEHP*	0.24	0.79
DiNP*	1.9	6.5

IDL= calculated as amount of chemical that gives a signal to noise ratio of 3:1
LOQ= calculated as amount of chemical that gives a signal to noise ratio of 10:1

Specific surface area (SSA) measurements

Brenauer-Emmett-Teller (BET) adsorption method was used to measure specific surface area (Rouquerol et al. 2014). Briefly, fine pieces of fabrics (200-300 mg) were kept under vacuum (~10^{-6} bar) at 60°C for 16 hours to outgas moisture and other volatile compounds. An Autosorb-iQ gas sorption analyzer (Quantachrome, Boynton Beach, FL, USA) with a resolution of ~0.01 m^2/g was used to obtain adsorption isotherms. Adsorbate gas, Krypton (Kr, 99.999%), and purge gas, Helium (He, 99.999%), were purchased from MEGS (Quebec, Canada). Kr sorption isotherms were obtained at 77 K using a liquid nitrogen (N) bath at a relative pressure range of 0.01 to 0.99. Adsorption isotherms were obtained using Kr rather than N as the fabrics' SSA were < 5 m^2/g.

Details of laundering, drying, extraction and analysis

Laundering. Fabrics were laundered in 500 mL glass bottles with polypropylene caps (Pyrex, Fisher Scientific). Bottles and caps were pre-washed with a soap solution followed by baking glass bottles for 12 hours at 250°C. Both caps and bottles were rinsed with hexane, DCM and methanol before use. For fabric laundering, 500 mL of HPLC grade water (Fisher Scientific), stored at room temperature, was added to the bottles along with 2–3 drops of Natural 2X concentrated liquid laundry detergent (Seventh Generation, Burlington, VT, USA). A list of detergent ingredients is given below. No target chemicals were detected in the laundry detergent which is consistent with Schreder and La Guardia (2014). Bottles were manually shaken to mix the soap and water before adding fabrics. Once fabrics were added (one fabric in one bottle), capped bottles were shaken using a wrist action shaker (Burell Scientific, LLC., USA) for 30 minutes to imitate mixing by a washing machine. After fabrics were laundered,

laundry water was transferred to Teflon separatory funnels (TSF) for liquid-liquid extraction. Bottles containing fabrics were rinsed twice with 250 mL of HPLC grade water and the rinse water, along with laundry water obtained from squeezing the fabrics (using tweezers), were combined with the laundry water in the TSF for extraction.

Laundry soap ingredients of Seventh Generation detergents (as obtained from http://www.seventhgeneration.com/natural-laundry-detergent?v=31): water, laureth-6 (plant-derived cleaning agent), sodium lauryl sulfate (plant-derived cleaning agent), sodium citrate (plant-derived water softener), glycerin (plant-derived enzyme stabilizer), sodium chloride (mineral-based viscosity modifier), oleic acid (plant-derived anti-foaming agent), sodium hydroxide (mineral-based pH adjuster), calcium chloride (mineral-based enzyme stabilizer), citric acid (plant-derived pH adjuster), protease, amylase, and mannanase (plant-derived enzyme blend soil removers), and benzisothiazolinone and methylisothiazolinone (synthetic preservatives).

Drying (in a dryer). Group 3 fabrics were dried in a five year-old LG electric dryer. Before drying, lint was removed from the lint trap and the internal surface of the dryer (stainless steel tub) and lint trap were wiped with isopropanol (HPLC grade, Fisher Scientific). Two sets of blanks (dried and not dried in dryer) were collected before drying test samples. In the first set, two pieces of pre-cleaned cotton and polyester squares were separately swirled thrice inside the dryer and were wrapped in clean aluminium foil to dry in a desiccator before extraction. The second set of blanks consisted of two pre-cleaned squares of cotton and one of polyester that were dried in the dryer and then wrapped in clean aluminium foil for further extraction. The drying cycle of the dryer was set at medium heat for 20 minutes. Cotton and polyester fabrics (group 3, 10 pieces of each fabric) were dried separately that had been laundered after deployed for 30 days. Lint was collected from the lint trap after the drying cycles of cotton and polyester fabrics. Dried fabric and single lint samples (each of cotton and polyester weighing 0.02 and 0.23 g, respectively) were wrapped in clean aluminium foil until extraction.

Extraction. ASE operating conditions: temperature: 70°C, pressure: 1500 psi, heat time: 5 min, static time: 4 min, flush volume: 60%, purge time: 60 s, and static cycles: 3.

GC-MSD operating conditions: OPE (except TDCiPP) and phthalate analysis was performed using a 30 m DB-5 MS column (Agilent Technologies, 0.25 mm i.d. and 0.25 µm film thickness) operating in EI mode. For OPEs, the oven temperature program was: initial at 75°C hold for 1 min, 15°C min^{-1} to 180°C and hold for 1 min, 6°C min^{-1} to 270°C, 20°C min^{-1} to 310°C and hold for 4 min. For phthalates, the oven temperature program was: initial at 75°C hold for 3 mins, 10°C min^{-1} to 320°C and hold for 3 min. BFRs (PBDEs and NFRs) and TDCiPP analysis was performed using 15 m DB-5 MS column (Agilent Technologies, 0.25 mm i.d. and 0.25 µm film thickness) operating in electron capture negative ion mode with methane as the regent gas with the following oven temperature program: initial at 100°C hold for 1.5 min, 12°C min^{-1} to 250°C, then 60°C min^{-1} to 290°C, hold for 3 min and finally 40°C min^{-1} to 320°C, hold for 11 min.

Results

Table S3. Average concentrations of chemicals (ng/dm^2 fabric planar surface area) accumulated by cotton and polyester fabrics after 30 day deployment (group 1).

Chemicals	**Cotton**	**Polyester**
	Average ± Standard deviation (Detection frequency)	
DiBP	650±157 (100%)	70±14 (70%)
DnBP	914±312 (100%)	179±38 (90%)
BzBP	370±53 (100%)	421±125 (100%)
DEHP	1363±492 (90%)	1091±460 (90%)
DiNP	178±95 (90%)	187±62 (70%)
PBBz	0.1±0.05 (100%)	0.2±0.05 (100%)
PBT	0.4±0.1 (90%)	0.4±0.1 (90%)
HBB	1.0±0.2 (90%)	0.9±0.3 (90%)
EH-TBB	1.0±0.5 (100%)	1.3±0.8 (100%)
BDE-17	1.0±0.3 (100%)	0.84±0.1 (100%)
BDE-28	2.2±0.6 (100%)	2.3±0.8 (100%)
BDE-47	46±11 (100%)	53±18 (100%)
BDE-66	0.7±0.2 (100%)	0.8±0.3 (100%)
BDE-100	2.6±0.7 (100%)	3.5±1.2 (100%)
BDE-99	10±2.9 (100%)	15±4.5 (100%)
TnBP	22±9 (90%)	3.2±1.3 (50%)
TCEP	57±49 (70%)	62±41 (70%)
TCPP-1	588±422 (80%)	109±88 (100%)
TCPP-2	347±191 (80%)	80±55 (100%)
TCPP-3	115±91 (80%)	19±7.8 (100%)
TPhP	32±13 (90%)	21±7.5 (100%)
TDCiPP	36±25 (100%)	13±7.6 (90%)
EHDPP	9.3±5.5 (100%)	3.8±1.7 (100%)

Table S4. Average concentrations of chemicals ± standard deviation transferred to laundry water (ng/L.dm^2 fabric) and remaining sorbed to cotton and polyester fabrics (ng/dm^2 fabric). Note: group 2 fabrics were dried in desiccator whereas group 3 fabrics were dried in an electric dryer. All concentrations normalized to planar surface area.

	Group 2				**Group 3**			
	Cotton		**Polyester**		**Cotton**		**Polyester**	
	In laundry water	**Sorbed to fabric**	**In laundry water**	**Sorbed to fabric**	**In laundry water**	**Sorbed to fabric**	**In laundry water**	**Sorbed to fabric**
DiBP	454±79	<LOQ	59±20	<LOQ	322±51	154±54	37±15	<LOQ
DnBP	565±137	113±57	118±57	46±15	418±139	149±36	68±28	108±47
BzBP	196±52	60±26	155±31	144±30	194±83	69±16	169±97	37±34
DEHP	362±296	757±345	339±322	641±205	348±114	709±139	168±91	591±317
DiNP	<LOQ	141±62	<LOQ	200±67	<LOQ	239±54	<LOQ	272±268
PBBz	<LOQ	0.06±0.02	<LOQ	0.10±0.04	<LOQ	0.08±0.02	<LOQ	0.26±0.09
PBT	<LOQ	0.24±0.1	<LOQ	0.23±0.08	<LOQ	3.1±2.5	<LOQ	4.5±2.5
HBB	<LOQ	0.56±0.17	<LOQ	0.59±0.15	<LOQ	0.62±0.12	<LOQ	1.2±0.38
EH-TBB	<LOQ	0.84±0.3	<LOQ	1.1±0.05	<LOQ	0.71±0.19	<LOQ	0.9±0.45
BDE-17	0.05±0.02	0.50±0.2	0.01±0.0	0.69±0.3	0.04±0.01	0.60±0.17	<LOQ	0.99±0.16
BDE-28	0.06±0.02	1.3±0.6	0.02±0.01	2.3±0.78	0.08±0.05	1.5±0.32	<LOQ	3.1±0.69
BDE-47	0.67±0.2	33±15	0.81±0.8	61±15	0.66±0.34	33±7.4	0.24±0.13	84±18
BDE-66	<LOQ	0.57±0.3	0.02±0.01	0.86±0.23	<LOQ	0.33±0.10	<LOQ	1.3±0.69
BDE-100	0.04±0.04	1.8±0.85	0.29±0.27	3.7±1.1	0.06±0.03	1.5±0.29	0.11±0.04	5.0±1.0
BDE-99	0.22±0.12	7.4±3.8	1.1±0.8	18±6.4	<LOQ	6.0±1.2	0.44±0.4	21±6.8
TnBP	16±5	<LOQ	1.5±1.5	<LOQ	17±5.0	0.95±0.39	1.1±0.34	<LOQ
TCEP	439±197	<LOQ	118±65	28±13	338±137	2.6±2	110±59	<LOQ
TCPP-1	1684±771	1.9±1.5	168±73	6.2±2.5	1517±729	3.1±1.5	155±68	0.6±0.9
TCPP-2	694±301	3.0±4.3	106±39	4.5±2.8	636±286	2.5±1.2	93±38	0.8±1.2
TCPP-3	104±42	2.2±1.6	26±7.3	1.1±0.8	100±42	5.9±2.3	24±14	0.3±0.08
TDiCPP	18±11	<LOQ	9.6±7.0	<LOQ	11±4.7	5.0±2.7	6.6±7.7	<LOQ
TPhP	10±4	10±6	12±7.3	12±2.8	11±3.8	5.1±1.3	9.7±2.5	0.8±0.55
EHDPP	0.4±0.2	5.9±4.2	0.4±0.1	2.9±1.6	0.29±0.10	4.1±1.4	0.29±0.15	4.2±6.3

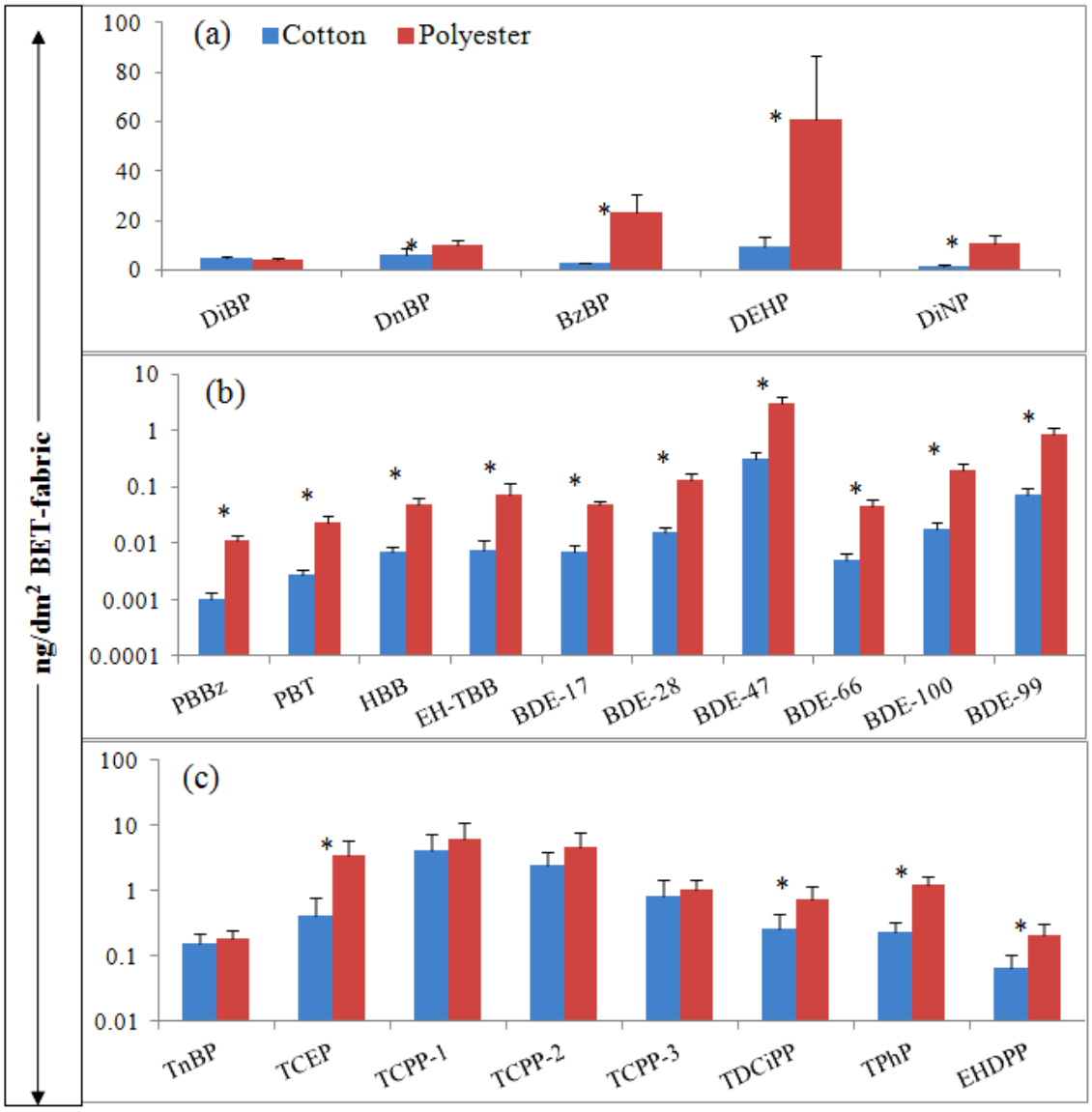

Figure S1. Averaged concentration of phthalates (a), BFRs (b), and OPEs (c) accumulated expressed as ng/dm^2 BET-SSA of fabric. Error bars indicate standard deviation. Note: Y-axis uses a log scale for BFRs and OPEs but is linear for phthalates. * represents a significant difference between cotton and polyester ($p<0.05$). Note: TCiPP is referred as TCPP-1.

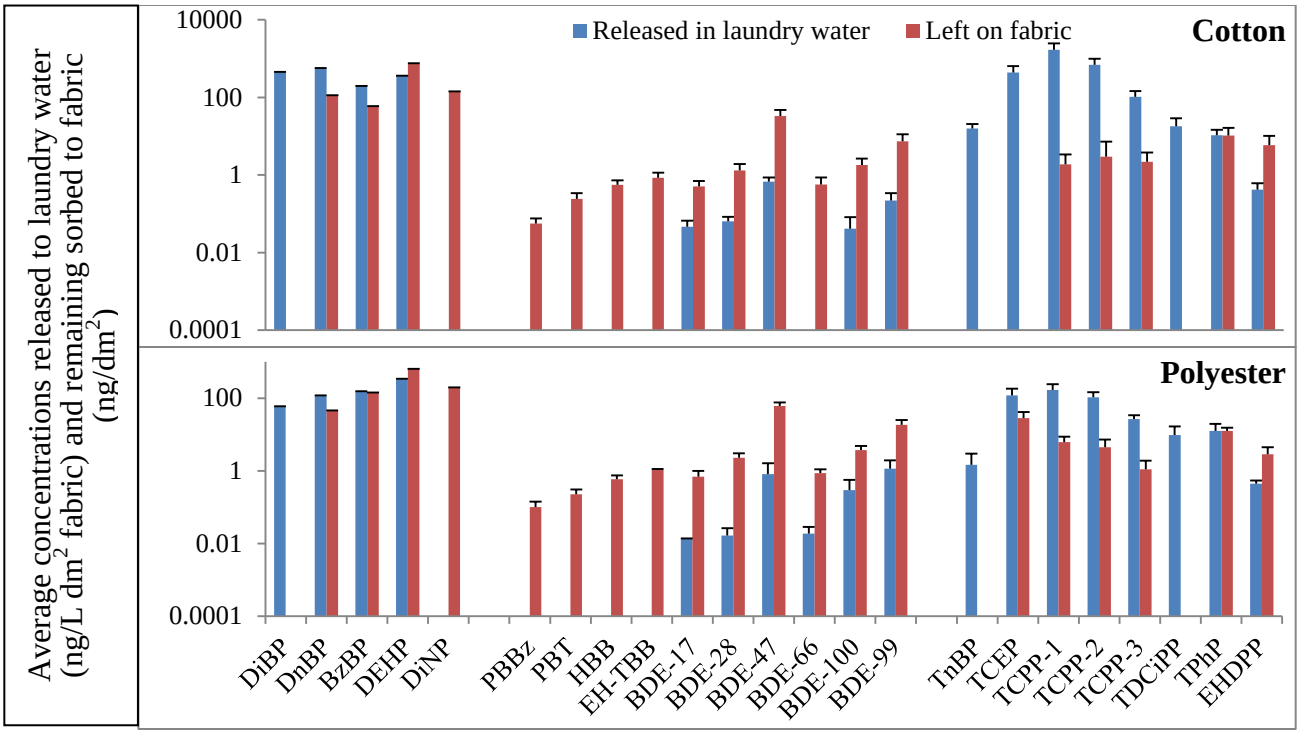

Figure S2. Average concentrations of SVOCs released in laundry water (ng/L.dm^2) and remaining on cotton (top) and polyester (bottom) (ng/dm^2).

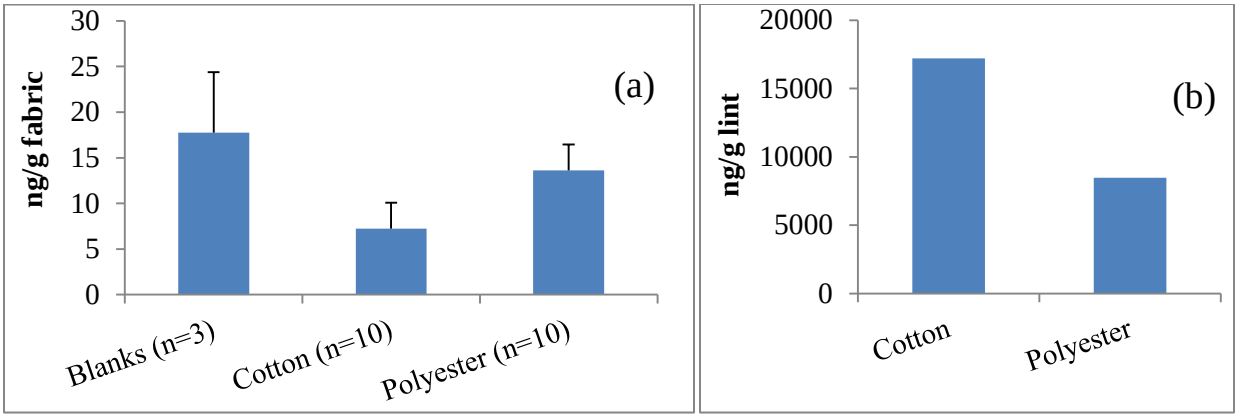

Figure S3. Concentrations of DBDPE in pre-cleaned and deployed fabrics dried for 20 minutes in an electric dryer (a), and lint collected from the lint trap of dryer (b). Note: A single lint sample was collected separately for each of cotton and polyester.

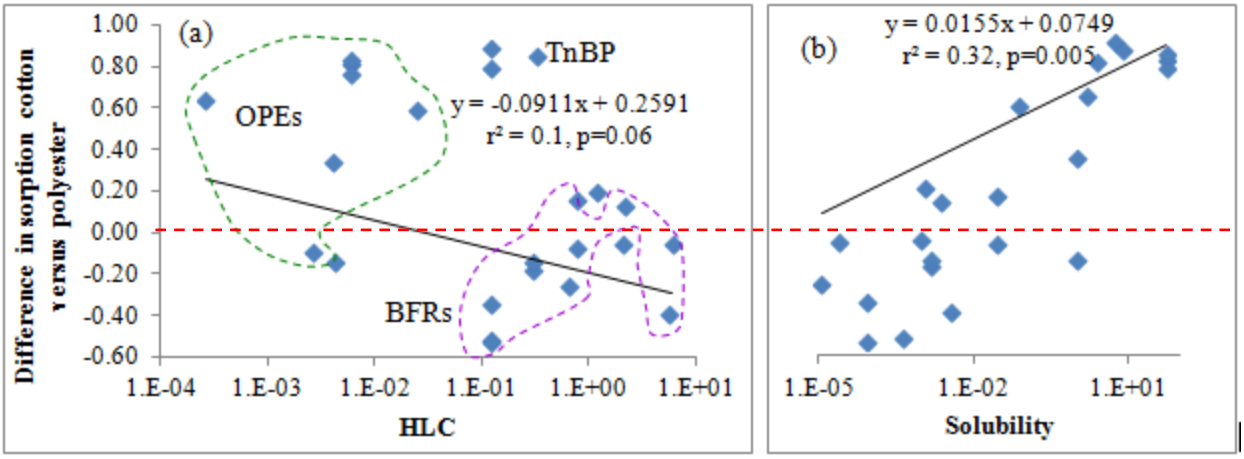

Figure S4. Difference in chemical accumulation, (C_{cotton} – $C_{polyester}$)/C_{cotton}, normalized to planar surface area plotted against the (a) Henry's law constant (HLC, Pa-m^3/mol), and (b) solubility (mg/L). TCEP, an outlier, was excluded from the solubility graph. In (a), green and purple ellipses indicate OPEs (except TCEP and TnBP) and BFRs, respectively, whereas remaining markers are phthalates. Red dotted line indicates zero on vertical axis.

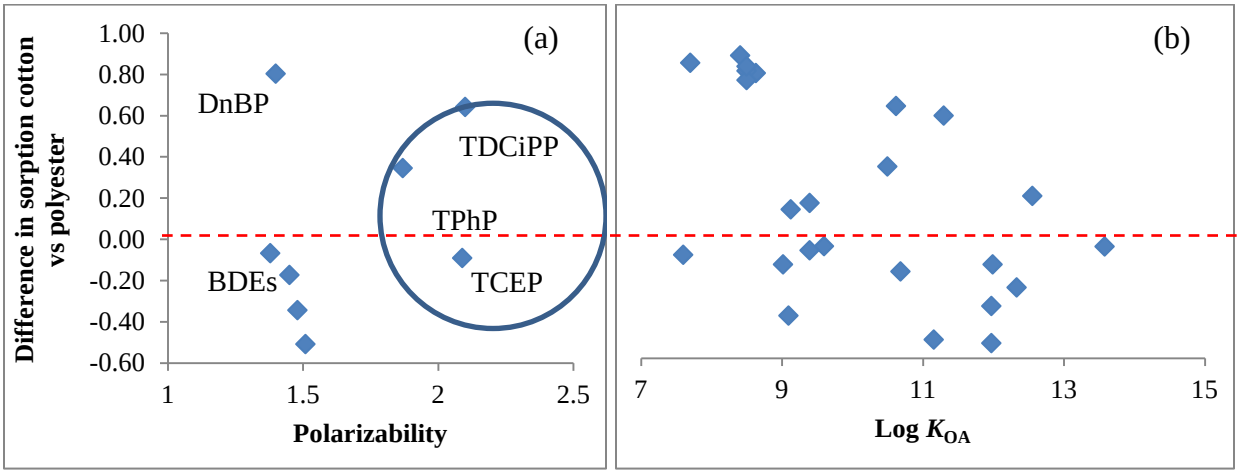

Figure S5. Difference in chemical accumulation, (C_{cotton} – $C_{polyester}$)/C_{cotton}, normalized to planar surface area, plotted against (a) polarizability (latter values not available for all chemicals), and (b) K_{OA}. Red dotted line indicates zero on vertical axis.

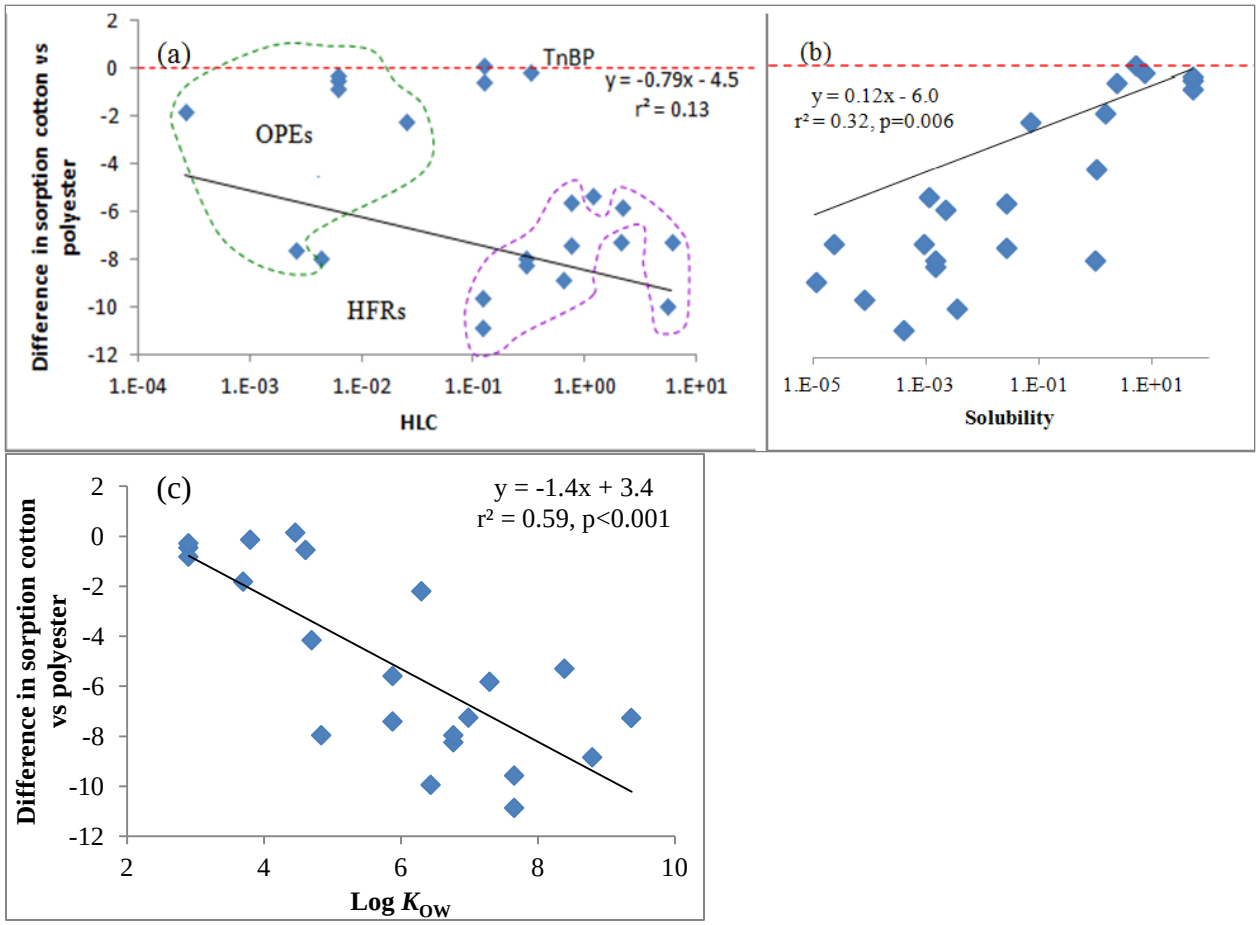

Figure S6. Difference in chemical accumulation, (C_{cotton} – $C_{polyester}$)/C_{cotton}, normalized to BET-SSA plotted against the (a) Henry's law constant (HLC, Pa-m^3/mol), (b) solubility (mg/L) and (c) octanol-water partition coefficient (Log K_{OW}). TCEP, an outlier, was excluded from solubility and Log K_{OW} graphs. In (a), green and purple ellipses indicate OPEs (except TCEP and TnBP) and BFRs, respectively, whereas remaining markers are phthalates. Red dotted line indicates zero on vertical axis.

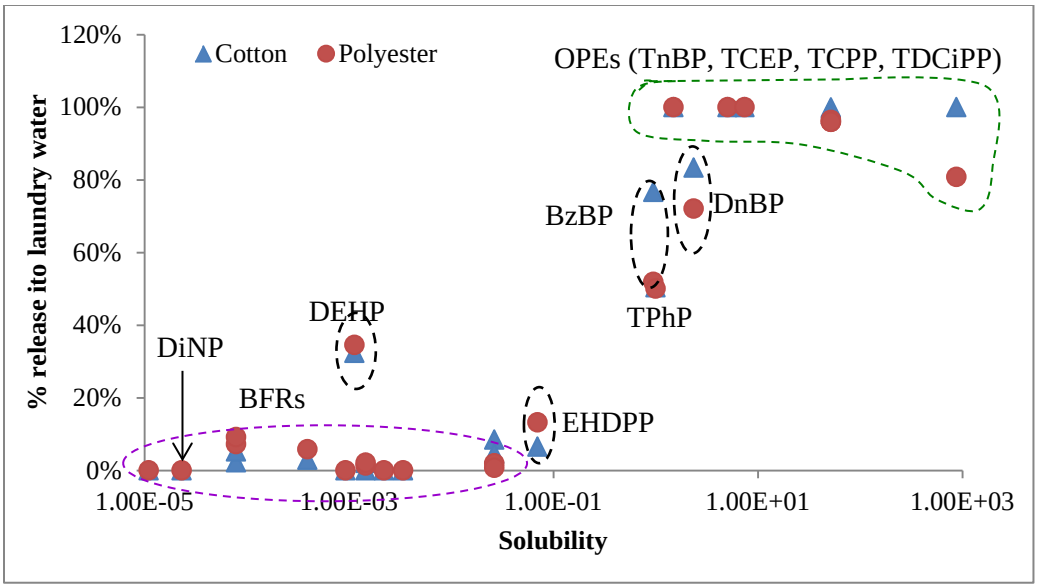

Figure S7. Percentage of accumulated chemical released to laundry water as a function of water solubility (mg/L). Black, green and purple ellipses indicate phthalates, OPEs and BFRs, respectively.

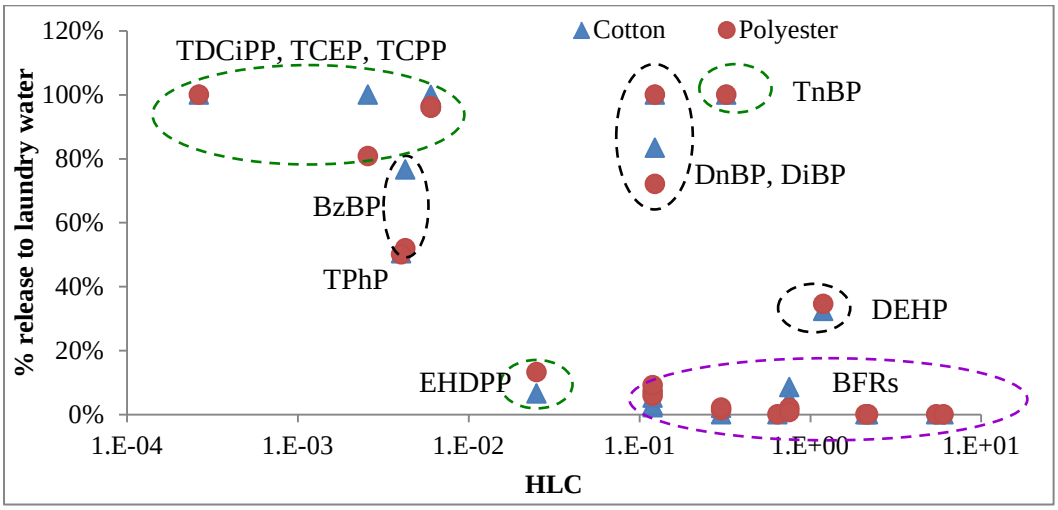

Figure S8. Percentage of accumulated chemical released to laundry water as a function of Henry's law constant (HLC, Pa-m^3/mol).

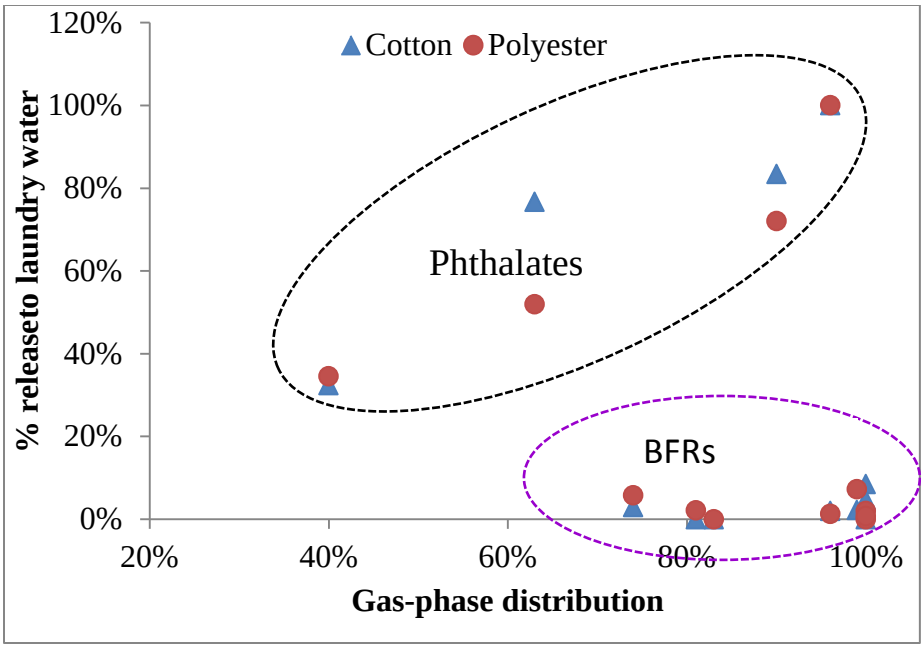

Figure S9. Percentage released of accumulated chemical to laundry water as a function of percentage in the gas phase measured by Saini et al. (2015, 2016).

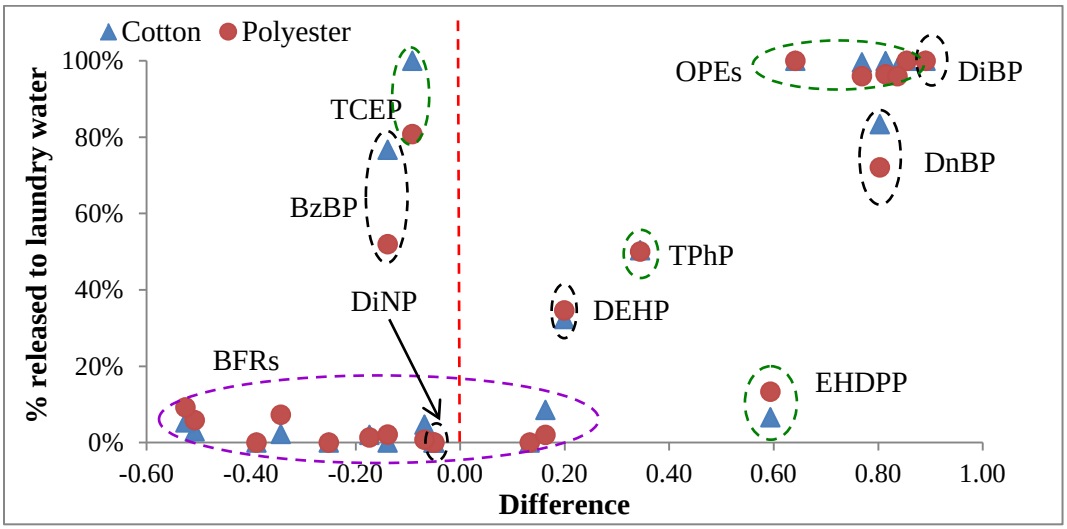

Figure S10. The difference of chemical accumulation from air ($C_{cotton} - C_{polyester}$)/$C_{cotton}$, normalized to planar surface area of fabric, plotted against the percentage released to laundry water. The dotted red line indicates zero on horizontal axis.

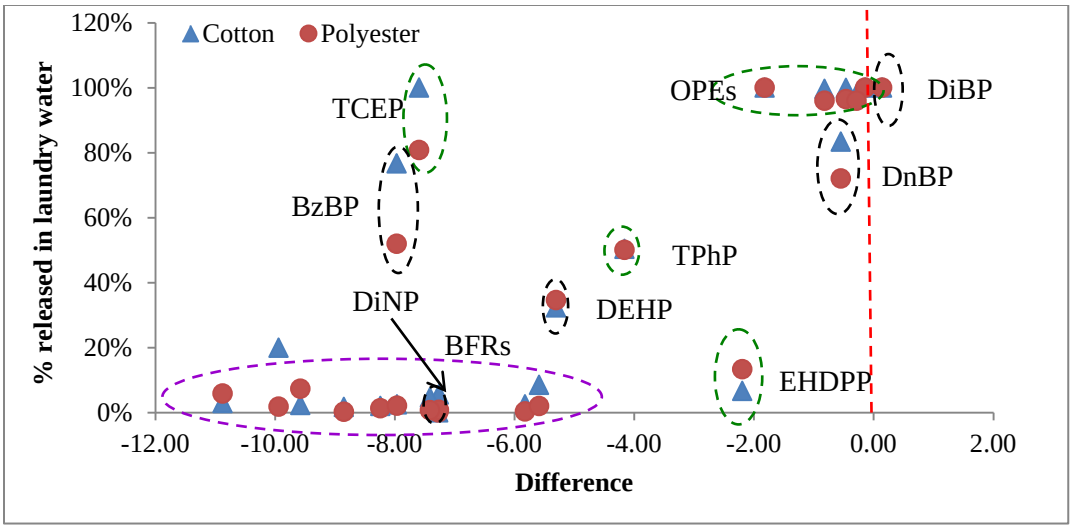

Figure S11. The difference in chemical accumulation, $(C_{cotton} - C_{polyester})/C_{cotton}$, normalized to BET-SSA plotted against the percentage released to laundry water. The dotted red line indicates zero on horizontal axis.

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