

Sorption of polycyclic aromatic hydrocarbons from tobacco smoke on different fabric materials

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Abstract. Third-hand tobacco smoke was evaluated through chemical analysis of polycyclic aromatic hydrocarbons (PAHs) adsorbed on different fabrics (cotton, silk, polyester and acetate) by ultrasonic extraction with n-hexane and subsequently analyzed using gas chromatography-mass spectroscopy. The total PAHs concentration adsorbed on the different fabrics ranged from 63–119 $\mu\text{g g}^{-1}$ tobacco (dry weight) with the lowest level of total PAHs observed in silk and the highest level in polyester. Total naphthalene and alkyl naphthalene concentration ranged from 50 to 100 $\mu\text{g g}^{-1}$ tobacco, with silk having adsorbed the lowest level of naphthalene and the highest level observed in polyester. On the other hand, acetate adsorbed the highest level of benzo(a)pyrene (0.038 $\mu\text{g g}^{-1}$). A positive correlation was observed between the fabric's chemical structure and the adsorption behavior of PAHs. PAH compounds exhibited high adsorption to less polar fabrics like acetate and polyester, while silk has lower affinity to PAHs due to the presence of polar amino acid side chains.

Key Words: third-hand tobacco smoke, polycyclic aromatic hydrocarbon, benzo(a)pyrene.

Introduction. Tobacco/cigarette use was reported by the World Health Organization (2010) as the leading cause of preventable death and has estimated to kill more than 6 million people each year worldwide. Tobacco smoke contains several thousand different chemical compounds, many of which are known carcinogens (US EPA 1992). However, non-smokers could also be at risk of lung cancer as a result of their exposure ("passive smoking") to tobacco smoke that occurs in various environments where smoking is permitted. Environmental Tobacco Smoke (ETS), also referred to as second-hand smoke (SHS), is dilute compared with the mainstream smoke (MS) inhaled by active smokers, but it contains similar chemical compounds, some of which could be carcinogenic and toxic (WHO 2010).

Another smoke hazard from residual tobacco smoke contamination that remains after the cigarette is extinguished is the so-called third-hand smoke (Winickoff et al 2009). Third-hand smoke (THS) consists of tobacco smoke pollutants that adsorbed to surfaces, fabrics and in dust, and may persist for months (Matt et al 2008). The THS could be reemitted and re-suspended back into the air, or react with oxidants and other compounds in the environment to yield secondary pollutants (ASH Scotland 2011), which may be inhaled or ingested by humans (Matt et al 2008; Ueta et al 2010).

In a study by Vello & Bryant (1975) the smoke components responsible for the biological activity of smoke are formed in the pyrolysis zone of the cigarette cone and the cigarette smoke condensate resides in the neutral smoke fraction and more specifically within the subfraction which contains the polycyclic aromatic hydrocarbons (PAHs). These specific components (PAHs) of tobacco smoke merit special attention inasmuch as the ones that are present in cigarette smoke condensates are believed to be carcinogens and have been shown to be partially responsible for the tumorigenicity of cigarette smoke condensate upon animal testing (Wynder & Hoffmann 1967).

Toxicity of tobacco smoke could be attributed to one of its major components released in the air during tobacco combustion, the PAHs. PAHs are a large class of organic compounds containing two or more fused aromatic rings made up of carbon and hydrogen atoms. PAHs are well known toxic and hazardous pollutants and highly potent carcinogens that have been established to cause tumors in rodents (ATSDR 1995). Several hundreds of PAHs and their derivatives having carcinogenic effects have been identified by Bjorseth & Ramdahl (1985) making them the largest single class of chemical carcinogens.

In a mixture of more than 70 PAHs in cigarette smoke, benzo(a)pyrene (BaP) is considered as one of the most strongly carcinogenic (ATSDR 1995). Grimmer et al (1988) described that a sidestream smoke contains 100 ng BaP per cigarette. While WHO (1987) reported that the mean total BaP content in the main stream smoke of one cigarette was 35 ng before 1960 and 18 ng in 1978-1979 while modern "low tar" cigarette delivers 10 ng BaP per stick and the concentration of BaP in a room extremely polluted with cigarette smoke is 22 ng m⁻³. Menzie et al (1992) observed that smokers consuming one pack of non-filtered cigarettes per day may have an estimated additional intake of 1–5 µg BaP day⁻¹ or 1000–5000 ng BaP day⁻¹.

Hence, this study assessed and compared the level of total PAHs, B(a)P and the B(a)P toxicity equivalents of other toxic PAHs adsorbed onto different fabrics: cotton, silk, polyester and acetate.

Materials and Methods

Preparation of materials. Unbranded local tobacco was purchased at the local public market of Miagao, Iloilo, Philippines. The fabrics (cotton, silk, acetate and polyester) used in the study were purchased from Shell Canvas Fabrics and Furnishing House-SM North EDSA Branch, Quezon City, Manila, Philippines. The fabric samples used were of 100% composition. The fabrics were pre-cleaned with n-hexane (Scharlau) using ultrasonic bath (Cole Palmer) for 30 min repeated three times for complete extraction to remove PAHs and other compounds initially present in the fabric.

Experimental set-up. Upon completion of the needed materials for the study and preliminary test was run, the experiment was conducted on January 2013 to February 2013. The pre-cleaned 10-cm square of each fabric was placed in a home-made pre-washed 18-Liter glass box following the method used by Ueta et al (2010). About 1 gram tobacco (exact weight recorded) was placed at the bottom of the box. An inlet was made at the bottom of the box that will provide enough air to sustain the tobacco combustion. During the tobacco combustion, the box was filled with side stream smoke. The fabrics were exposed to the smoke for 20 minutes and were immediately subjected to extraction. The experimental conditions, such as volume of the glass box and the surface area of the fabrics, were held constant all throughout the experiment.

Extraction, processing and analysis. The evaluation of third-hand smoke was done by quantitation of PAH concentration adsorbed to the fabric surfaces. Fabric samples were added with n-hexane for extraction of PAHs using ultrasonic bath for 30 minutes and repeated thrice. All solvents used are of HPLC grade (Merck). The extracts were pooled and concentrated to 5 mL using rotary evaporator set at 40°C with rotating speed of 50 rev per min. Furthermore, the extracts were cleaned up by eluting with 3 fractions of 30-ml n-hexane in a silica gel column. The eluates were then concentrated to 5 mL using the rotary evaporator and diluted to 10 mL by n-hexane. The PAH standards: 1,6,7-trimethyl naphthalene, 2-methyl anthracene, benzo[a]pyrene, fluoranthene, 1,12-dimethyl benz[a]anthracene, 1,4,5,8-tetramethylnaphthalene, 1,7-dimethyl naphthalene, 1-methyl naphthalene, 2,8-dimethyl dibenzothiophene, 3,6-dimethyl phenanthrene, 6-methyl chrysene, 9,9-dimethyl 9H fluorene, acenaphthene, acenaphthylene, anthracene, benz[a]anthracene, benzo(k)fluoranthene, fluorine, methyl dibenzothiophene, naphthalene, phenanthrene and pyrene were obtained from Supelco (Bellefonte, PA, USA).

PAH analysis using GC-MS. A Clarus 600 Gas Chromatograph (GC) with a split/splitless injection port and Clarus 600 T Mass Spectrometer (MS) detector was used for analysis of PAH. The injections were done in splitless mode. GC separation was performed on an Elite-5 silica capillary column of 60 m × 320 μm inner diameter. Helium was employed with a carrier gas flow of 2 mL min⁻¹ and the injector was maintained at 230°C. The GC-MS interface temperature was set at 280°C. The column temperature was started at 90°C for 1.0 min, and then programmed to 290°C at a rate of 10°C min⁻¹. The mass spectrometer was operated with a *m/z* range of 50 to 450. The data collection and quantitation was done with the TurboMass™ GC/MS software.

Statistical design and analysis. The four (4) fabric materials (cotton, acetate, polyester and silk) with three (3) replicates each, were exposed to side stream smoke one after the other following a complete block design. A solvent blank (negative control) was included in the experiment. One-Way Analysis of Variance and Duncan's Multiple Range Test (DMRT) using SPSS 14.0 Software for Windows™ was used to determine significant differences between the concentrations of PAHs adsorbed on the different fabrics and specifically identify which fabric differ from the other, respectively.

Results and Discussion

PAHs concentration adsorbed on fabrics. Among the 16 priority environmental PAHs considered by US EPA (2001), and also cited by Yan et al (2004) eleven (11) of which were determined and quantified in this study: acenaphthylene, acenaphthene, anthracene, benz[a]anthracene B[a]A, benzo[a]pyrene (B[a]P), benzo[k]fluoranthene (B[k]F), fluoranthene, fluorene, naphthalene, phenanthrene, and pyrene and some of their alkylated homologs. Figure 1 presents the concentration of PAH compounds adsorbed on cotton, polyester, silk and acetate fabrics and the total concentration of the 11 PAHs (Σ PAHs) and their alkylated homologs adsorbed on the different fabrics ranged from 63-119 μg g⁻¹ tobacco with the lowest level detected in silk and highest level in polyester (Table 1).

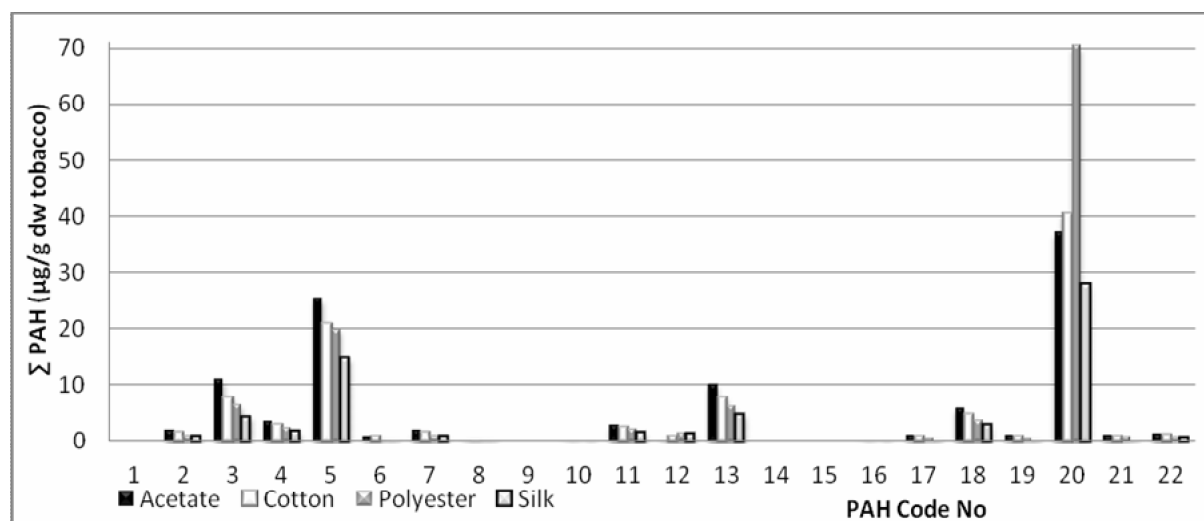


Figure 1. Mean PAHs concentration in different fabrics tested
 (1 - 1 12 dimethyl benz[a]anthracene; 2 - 1 4 5 8 tetramethyl naphthalene;
 3 - 1 6 7 trimehtyl naphthalene; 4 - 1 7 dimethyl naphthalene; 5 - 1 methyl naphthalene;
 6 - 2 8 dimethyl dibenzothiophene; 7 - 2 methyl anthracene; 8 - 3 6 dimethyl phenanthrene;
 9 - 6 methyl chrysene; 10 - 9 9 dimethyl 9H fluorene; 11 - acenaphthene; 12 - acenaphthylene;
 13 - anthracene; 14 - benz[a]anthracene; 15 - benzo[k]fluoranthene; 16 - benzo[a] pyrene;
 17 - fluoranthene; 18 - fluorene; 19 - methyl dibenzothiophene; 20 - naphthalene;
 21 - phenanthrene; 22 - pyrene.

The result shows that naphthalene and alkylated naphthalene derivatives are the dominant PAH compounds extracted from the fabrics exposed to third-hand tobacco

smoke (Table 1). It is also evident that the highest level of total naphthalene and alkyl naphthalene was adsorbed to the polyester (100.59 $\mu\text{g g}^{-1}$ tobacco) and the lowest level of total naphthalene and alkyl naphthalene was on silk (49.68 $\mu\text{g g}^{-1}$ tobacco). Since naphthalene has higher vapour pressure and remains mostly in gaseous form, naphthalene and alkylated naphthalene found adsorbed onto fabrics may pose hazard when these compounds are desorbed and released to the air. Its presence in third hand smoke implies high exposure risk by inhalation. Some regulations and recommendations were established to protect individuals from the possible health effects of naphthalene in the environment. The Occupational Safety & Health Administration (OSHA) (cited in ATSDR 2005) has set a limit 0.2 milligrams of PAHs per cubic meter of air and safe limit of 10 parts per million (ppm) for the level of naphthalene in air over an 8-hour exposure.

Table 1
PAHs concentration in thirdhand smoke adsorbed to the different fabrics

Compound	PAH Concentration ($\mu\text{g g}^{-1}$ tobacco)			
	Acetate	Cotton	Polyester	Silk
Acenaphthene	2.960±0.429	2.500±0.096	2.156±0.505	1.582±0.320
Acenaphthylene	0.522±0.122	0.943±0.218	1.661±0.607	1.288±0.720
Anthracene	10.257±1.395	7.790±0.359	6.309±1.735	4.782±0.857
Benz[a]anthracene	<0.0012 LOD	<0.0012 LOD	<0.0012 LOD	<0.0012 LOD
Benzo[k]fluoranthene	<0.0027 LOD	<0.0027 LOD	<0.0027 LOD	<0.0027 LOD
Benzo[a]pyrene	0.038±0.008	0.027±0.010	0.022±0.008	0.016±0.002
Fluoranthene	1.055±0.107	0.888±0.177	0.687±0.486	0.519±0.213
Fluorene	5.976±0.574	4.770±0.244	3.804±0.959	2.863±0.571
Naphthalene	37.265±5.066	40.359±2.203	70.475±20.199	27.887±7.660
Phenanthrene	1.131±0.285	0.991±0.226	0.788±0.263	0.406±0.085
Pyrene	1.434±0.299	1.174±0.176	0.888±0.562	0.651±0.128
1,12-dimethyl benz[a]anthracene	<0.0044 LOD	<0.0044 LOD	<0.0044 LOD	<0.0044 LOD
1,4,5,8-tetramethyl naphthalene	2.069±0.311	1.536±0.053	1.200±0.371	0.949±0.170
1,6,7-trimethyl naphthalene	11.132±1.063	7.771±1.124	6.471±1.609	4.334±1.438
1,7-dimethyl naphthalene	3.567±0.634	2.864±0.186	2.435±0.551	1.752±0.366
1-methyl naphthalene	25.397±3.592	20.744±0.585	20.012±5.920	14.751±3.699
2,8-dimethyl dibenzothiophene	0.935±0.187	0.837±0.048	0.526±0.348	0.422±0.119
2-methyl anthracene	2.067±0.379	1.461±0.185	1.173±0.576	0.878±0.144
3,6-dimethyl phenanthrene	0.450±0.015	0.499±0.070	0.524±0.156	0.300±0.101
6-methyl chrysene	<0.0002 LOD	<0.0002 LOD	<0.0002 LOD	<0.0002 LOD
9,9-dimethyl 9H fluorene	0.317±0.102	0.209±0.024	0.096±0.086	0.121±0.052
1-methyl dibenzothiophene	1.025±0.196	0.882±0.041	0.649±0.301	0.508±0.112
Total	106.531±13.8	95.164±4.04	118.716±35.2	63.078±16.7

*LOD = limit of detection.

LPAH vs HPAH. Analysis of the extract indicated that the low molecular weight PAHs (LPAHs) of 2 or 3 ring groups such as naphthalene, fluorene, phenanthrene and anthracene, are more abundant than higher molecular weight PAHs (HPAHs) in cotton, acetate, silk and significantly in polyester (Figure 2). Moir et al (2008) observed a similar trend in their analysis of side stream cigarette smoke showing higher percentage of LPAHs than HPAHs. PAHs which tend to have low vapor pressures are usually adsorbed on particulate matter in the atmosphere. The vapor pressure of PAH is inversely proportional to the number of rings it contains.

Parent and alkylated PAHs. The abundance of alkylated PAHs is also worth noting. Although there is less toxicity information available for most of the alkyl PAHs than for their parent compounds, most alkyl PAHs appear to be as toxic or hazardous as the parent compound. However, the result of analysis shows the predominance of the parent PAHs relative to the alkylated PAH, except in the acetate fabric where the alkylated

naphthalene concentration is found higher than the parent naphthalene (Figure 3). The presence of alkylated PAHs poses risk in exposure. Alkyl naphthalenes pose similar hazards or worse hazards than naphthalene. It was reported by the US National Toxicology Program that exposure to 1-methylnaphthalene is also associated with increase in respiratory tumors as observed in mice (NTP 1992). Within an aromatic series, acute toxicity increases with increasing alkyl substitution on the aromatic nucleus (Irwin et al 1997). Alkylated PAHs generally persist for a longer time than their parent compounds which suggests that third-hand smoke exposure risk is further increased by the presence of alkylated PAHs.

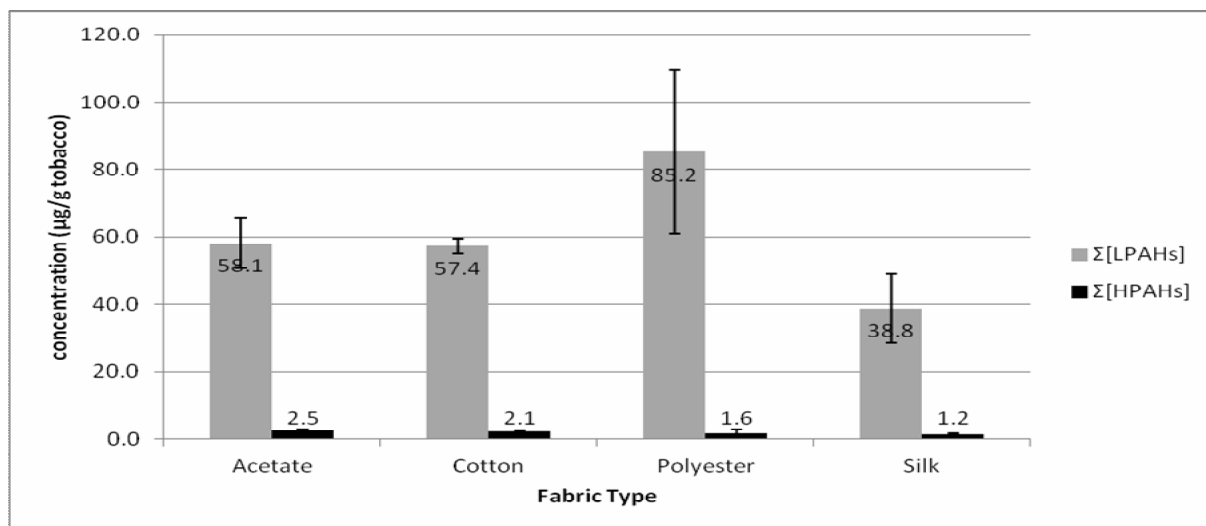


Figure 2. Σ[LPAHs] vs Σ[HPAHs] in third hand smoke.

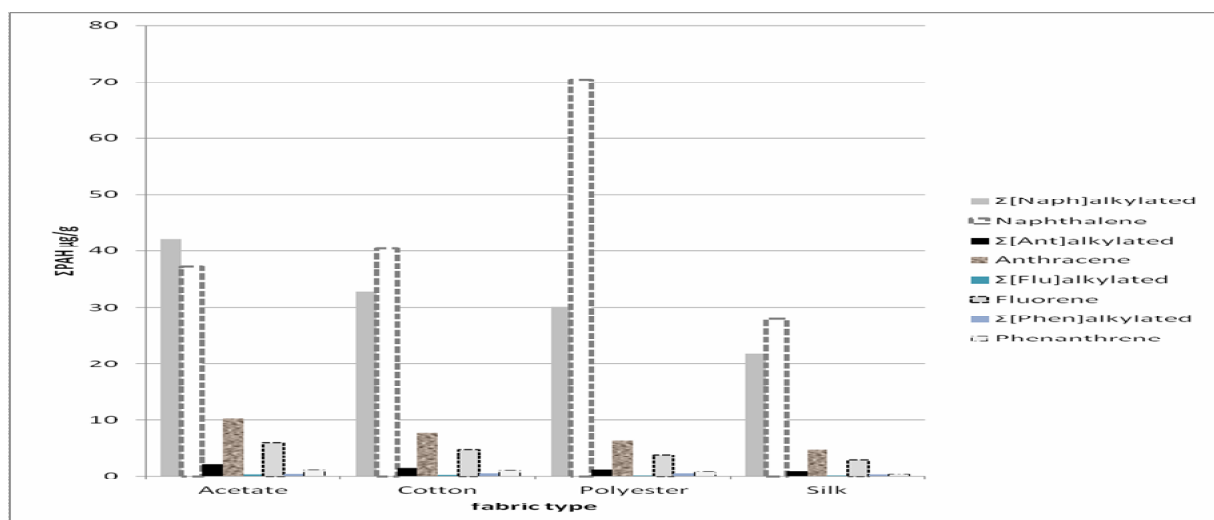


Figure 3. Concentration of Σalkyl [PAH] vs parent PAH.

Benzo[a]pyrene toxicity. In a mixture of PAHs, benzo(a)pyrene is of greatest concern because of its established toxicity (Jung et al 2010). Figure 4 presents the concentration of BaP present in the different fabrics ($0.016\text{--}0.038\ \mu\text{g BaP g}^{-1}$ tobacco in a space volume of $0.018\ \text{m}^3$), with acetate having the highest adsorption level ($0.038\ \mu\text{g BaP g}^{-1}$ tobacco). One-way analysis of variance ($p < 0.05$), shows that there is a significant difference among BaP concentration present in the four fabrics and DMRT analysis showed that acetate has significantly adsorbed higher BaP concentration than cotton, polyester and silk. The China Tobacco Control Report (2007) presented that a tobacco smoke contains 180 ng of benzopyrene released in a $30\ \text{m}^3$ room, is equivalent to a benzo(a)pyrene density of $6\ \text{ng m}^{-3}$, that is six times the permissible health standard of $1\ \text{ng BaP m}^{-3}$ (WHO 2010). If an average low tar cigarette would release 22 ng BaP (WHO

2010) in a 108 m³ room, the smoke of 5 cigarettes would be equivalent to about 1 ng BaP m⁻³. However, in this study, the 0.038 µg BaP g⁻¹ tobacco in a space volume of 0.018 m³ is equivalent to 2,111 ng BaP m⁻³ and is >2000 times the permissible health standard.

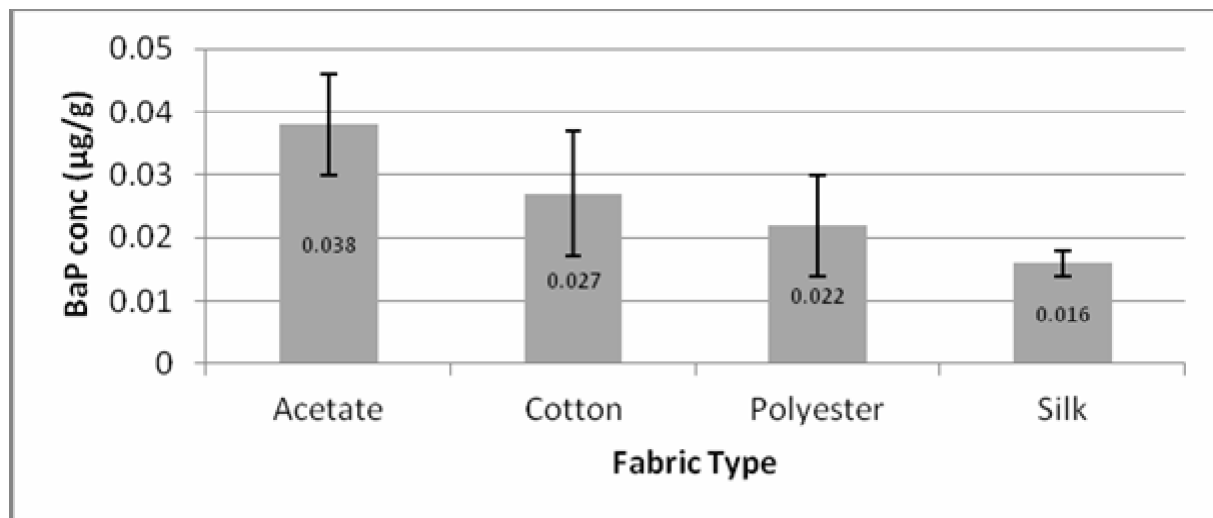


Figure 4. Benzo[a]pyrene concentration adsorbed on different fabrics.

BaP-toxicity equivalent factors for other PAHs. To account for the toxicity of other toxic PAHs, the toxicity equivalency factor (TEF) for each PAH was used as an estimate of the toxicity equivalence of some PAH compounds relative to benzo(a)pyrene. The total benzo[a]pyrene relative potencies contributed by individual PAHs and the BaP-TEQ, the carcinogenic equivalent calculated from the cancer potency relative to BaP (TEF) as the sum of unsubstituted PAH are calculated based on the equation derived by Jung et al (2010): $(\text{BaP-TEQ})_{\Sigma_{12}\text{PAH}} = \Sigma([\text{PAH}] \times \text{TEF})$. Using the BaP toxicity equivalent factors of USEPA (1993), the total BaP carcinogenic equivalents adsorbed on the four fabrics are presented in Table 2. The result showed that the total BaP carcinogenic equivalents is also equal to the benzo(a)pyrene value since the other toxic PAHs were almost nil (below the detection limit of the method and the instrument).

Table 2
Toxicity equivalent factors and BaP toxicity equivalent concentration (µg g⁻¹ tobacco)

Compound	US EPA TEF* unitless	Acetate	Cotton	Polyester	Silk
		mean±SD	mean±SD	mean±SD	mean±SD
Acenaphthene	-				
Acenaphthylene	-				
Anthracene	-				
Benz[a]anthracene**	0.1	0.0001	0.0001	0.0001	0.0001
Benzo(k)fluoranthene**	0.01	0.0003	0.0003	0.0003	0.0003
Benzo[a]pyrene	1	0.0381±0.008	0.0270±0.0096	0.0221±0.008	0.0156±0.0018
Fluoranthene	-				
Fluorene	-				
Naphthalene	-				
Phenanthrene	-				
Pyrene	-				
Σ[BaP-TEQ]		0.0385±0.008	0.0274±0.009	0.0225±0.008	0.0160±0.002

*TEFs values from USEPA (1993).

** Toxic PAH with TEF equivalent to BaP.

Adsorption of PAHs in relation to the fabric's chemical composition. Silk was found to have the lowest adsorption of PAHs which is almost half of the total PAHs adsorbed on polyester. Silk is a natural protein fiber excreted by the moth larva *Bombyx mori* (silkworm). The fibroin silk fibers are composed predominantly of glycine, alanine, tyrosine, and serine. Silk is a polar molecule due to the presence of polar amino acid side

chains (Needles 1986) and has low affinity for non-polar compounds such as PAHs. The total PAHs adsorbed on the cotton is about 1.5 times that adsorbed on silk and a little less than the PAHs adsorbed on acetate, which is a modified cellulose. Cotton fabric is a hydrophilic molecule owing to the -OH in its chemical structure. The β -1-4-D glucopyranose is the basic building block of the cotton cellulose repeated thousands of times. These long chains are restricted or have limited rotational liberty at the anhydrogluco-pyranose C-O-C link that makes the fiber more rigid and less flexible. There are many hydroxyl groups present at different points that are prone to hydrogen bonds. The long linear chains of cellulose permit the hydroxyl functional groups on each anhydroglucose unit to interact with hydroxyl groups on adjacent chains through hydrogen bonding and van der Waals forces (Needles 1986). Acetate and polyester are hydrophobic molecules and theoretically have affinity for nonpolar compounds, was found congruent with the results obtained in this study. Ueta et al (2010) also reported that among common materials (i.e. acetate, cotton, linen, silk, polyester), acetate can hold the most amount of volatile organic compound from cigarette smoke.

Probable risk associated to third-hand tobacco smoke. PAHs from third-hand smoke adsorbed to fabric materials may possibly be released to the air due to desorption of these volatile compounds. This process of desorption and risk of exposure to the desorbed PAHs may lead to possible toxicity due to inhalation. The safe exposure standard for naphthalene is 52 mg m^{-3} (equivalent to 10 ppm) (Workplace Health and Safety of Queensland 2012). The inhalation Reference Concentration (RfC) that considers toxic effects for both the respiratory system (portal-of-entry) and peripheral to the respiratory system (extra-respiratory effects) studies of the National Toxicology Program reported NOAEL for naphthalene as $3 \times 10^{-3} \text{ mg m}^{-3}$ and LOAEL(HEC) the human equivalent concentration for naphthalene as 9.3 mg m^{-3} (National Toxicology Program 1992; US EPA 1992).

Conclusions. Among the four fabric materials tested, acetate and polyester had the highest affinity to adsorb PAHs and silk has the lowest adsorption capacity. Naphthalene and alkyl naphthalene were found to be the predominant compound in the tobacco smoke. Benzo(a)pyrene, which is of greatest concern due of its established toxicity was found highest in acetate. The toxicity equivalence of other toxic PAHs was also calculated by using the toxicity equivalency factor for each PAH relative to benzo(a)pyrene. However, benz[a]anthracene and benzo(k)fluoranthene with 0.1 and 0.01 factor (unitless) for BaP toxicity equivalence factor were almost nil. As such, in this study the toxicity is mostly due to benzo(a)pyrene with the $0.038 \text{ } \mu\text{g BaP g}^{-1}$ tobacco in a space volume of 0.018 m^3 and is equivalent to $2,111 \text{ ng BaP m}^{-3}$ (equivalent to $2.11 \times 10^{-3} \text{ mg m}^{-3}$). Hence, PAHs from third-hand smoke adsorbed onto fabric materials may possibly be released to the air due to desorption of these volatile compounds. This process of desorption and risk of exposure to the desorbed PAHs may lead to possible toxicity through inhalation.

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