



# Screening for perfluoroalkyl acids in consumer products, building materials and wastes



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## HIGHLIGHTS

- 15 target PFAAs were analyzed in 126 samples of consumer products and building materials.
- In 88% of samples at least one PFAA was detected; dominated by PFOS.
- The highest  $\Sigma_{15}$ PFAA concentration ( $77.61 \mu\text{g kg}^{-1}$ ) was found in a textile.
- Several materials contained high levels of unregulated short-chain PFAAs.
- C5–C8-chain PFCAs in wood-based building materials were identified for the first time.

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## ABSTRACT

Perfluoroalkyl and polyfluoroalkyl substances (PFASs) are a large group of important chemical compounds with unique and useful physico-chemical properties, widely produced and used in many applications. However, due to the toxicity, bioaccumulation and long-range transport potential of certain PFASs, they are of significant concern to scientists and policy makers. To assess human exposure to PFASs, it is necessary to understand the concentrations of these emerging contaminants in our environment, and particularly environments where urban population spend most of their time, i.e. buildings and vehicles.

A total of 126 samples of building materials, consumer products, car interior materials and wastes were therefore analyzed for their content of key PFASs - 15 perfluoroalkyl acids (PFAAs). At least one of the target PFAAs was detected in 88% of all samples. The highest concentration of  $\Sigma_{15}$ PFAAs was found in textile materials ( $77.61 \mu\text{g kg}^{-1}$ ), as expected, since specific PFAAs are known to be used for textile treatment during processing. Surprisingly, PFAAs were also detected in all analyzed composite wood building materials, which were dominated by perfluoroalkyl carboxylic acids with 5–8 carbons in the chain ( $\Sigma_4$ PFCAs up to  $32.9 \mu\text{g kg}^{-1}$ ). These materials are currently widely used for building refurbishment, and this is the first study to find evidence of the presence of specific PFASs in composite wood materials. Thus, in addition to consumer products treated with PFASs, materials used in the construction of houses, schools and office buildings may also play an important role in human exposure to PFASs.

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## 1. Introduction

Perfluoroalkyl and polyfluoroalkyl substances (PFASs) are a diverse and large group of chemicals which consists of various polymers and nonpolymer substances (Buck et al., 2011). PFASs are applied to a wide range of commercial and industrial materials in order to change their physico-chemical properties (Banks, 1994; Kissa, 2001) resulting in lowered interfacial or/and surface

tension and enhanced resistance to water, stains, oil, and fire. Major producers of PFASs are 3M, DuPont, Clariant, Daikin and Asahi Glass and applied in large volumes on a global scale: e.g. the total production of perfluorooctane sulfonyl fluoride from 1970 to 2002 was estimated to be about 100 000 metric tons (Paul et al., 2009). PFASs are specifically used in applications such as surface treatment of textiles (e.g. carpets, furniture materials and clothing), leather products, paper and packaging, coating additives, cleaning agents and firefighting foams (Paul et al., 2009; Prevedouros et al., 2006; Wang et al., 2014). In addition, specific PFASs are also used in the photographic industry, photolithography and manufacturing of

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semiconductors (OECD, 2002; EFSA, 2008). Since PFASs are typically used to treat surface layers they can be directly released from final products into the environment during their lifecycle. Wang et al. (2014) estimated global emissions of perfluoroalkyl carboxylic acids (PFCAs) (C<sub>4</sub> to C<sub>14</sub>) from direct and indirect sources between 1951 and 2015 of up to 21 400 metric tons.

The fate of PFASs in the environment as well as their biological activity are determined by their physico-chemical properties, which vary greatly with molecular structure (e.g. functional group and carbon chain length) (Kissa, 2001). In general, neutral PFASs (e.g. fluorotelomer alcohols, perfluorinated sulfonamides and their derivatives) may undergo long range atmospheric transport (Lai et al., 2016) and may be partially degraded into perfluoroalkyl acids (PFAAs) in the presence of OH radicals (Ellis et al., 2004). These PFAAs, such as PFCAs and perfluoroalkane sulfonates (PFASs) have been shown to persist in the environment (Scheringer et al., 2014) and have no known significant natural degradation mechanism (Sulbaek Andersen et al., 2003). Typically they can undergo long-range transport and therefore they are widespread in a range of environmental compartments around the globe (Butt et al., 2007; Giesy and Kannan, 2001). Humans are exposed to PFASs through various pathways (e.g. dust ingestion, drinking water, diet and inhalation) (Enault et al., 2015; Haug et al., 2011; Miralles-Marco and Harrad, 2015). Similarly to the legacy persistent organic pollutants (POPs, e.g. polychlorinated biphenyls, organochlorine pesticides), the neutral PFASs accumulate in lipid-rich tissues (Chu et al., 2016). In contrast, the ionic PFASs, particularly the longer chain PFAS, bind to blood proteins and accumulate in the liver, kidney and bile secretions (Ng and Hungerbühler, 2014). Almost all human blood samples collected around the world were found to contain ionic PFASs at levels of ng mL<sup>-1</sup> (Gebink et al., 2015; Miralles-Marco and Harrad, 2015; Wang et al., 2015b). Information about the effects on humans are primarily available for PFOA and PFOS and are related to early menopause, changes in a sperm quality and increased risk of cancer (Bonefeld-Jorgensen et al., 2011; Joensen et al., 2009).

In 2009, PFOS and related compounds were listed under Annex B of the Stockholm Convention on Persistent Organic Pollutants, which restricts manufacturing and use to a few specific applications (Stockholm Convention, 2009). Within the European Union, the use of PFOS is regulated by European Commission Regulation No. 122/2006 (EU, 2006a), 552/2009 (EU, 2009) and 757/2010 (EU, 2010) Intermediate products or articles with concentrations of PFOS greater than 0.1% by weight are banned since 2007. For textiles or other coated materials, the amount of PFOS must be lower than 1 µg m<sup>-2</sup>. Moreover, PFOA, other C<sub>9</sub>-C<sub>14</sub> PFCAs and their salts and precursors have been identified by the European Chemicals Agency as a Substance of Very High Concern due to their irreversible effects on the environment and human health (ECHA, 2015) and presence of PFOA in articles on the European market at levels higher than 0.1% must be reported (EU Regulation No. 1907/2006 (EU, 2006b)). The group of regulated PFAAs includes also several other identified precursors and intermediates (OECD, 2007), which are currently being replaced by the manufacturers with alternative PFASs; e.g. PFAAs with shorter (C<sub>4</sub> to C<sub>6</sub>) fluorinated chains or PFASs with hetero atoms (N, S, O) in their carbon chains (EPA, 2012; Wang et al., 2013). Use volumes and the environmental and human impact of these alternative PFASs are currently under scientific focus (Blum et al., 2015).

Both regulated and non-regulated PFASs are frequently found to be present in consumer products used on a daily basis in homes, schools and workplaces. While intensive research has focused on food packaging materials (Begley et al., 2005; Gallart-Ayala et al., 2013; Herzke et al., 2012; Martinez-Moral and Tena, 2012; Poothong et al., 2012; Sinclair et al., 2007; Trier et al., 2011a, 2011b;

Vestergren et al., 2008; Zafeiraki et al., 2014), information regarding levels found in other daily-use consumer products is limited (Herzke et al., 2012; Liu et al., 2014; Trier et al., 2011a; Vestergren et al., 2015; Ye et al., 2015) and no information regarding levels in building materials is currently available. However, the indoor environment is a potential source of human PFAS exposure, especially in view of the fact that people in urban areas spend more than 20 h per day in indoor spaces (Klepeis et al., 2001).

Quantifying PFAS exposure is a complex and challenging process, not least due to a lack of knowledge concerning the exact PFAS composition in materials. Thus, a necessary component of quantifying PFAS exposure is obtaining information on their concentrations in broad range of materials types, since these materials release PFASs and contribute to their elevated levels in indoor air and dust. In this study we analyzed 126 individual samples of building materials, consumer products, car interior materials and wastes in order to provide insight into the distribution and amounts of PFASs added to indoor materials and to identify the potential sources of specific PFASs to the indoor environment.

## 2. Experimental

### 2.1. Sample collection

The aim of this study was to obtain PFAA levels in a representative selection of different types of materials. Both new and used materials were included to cover the widest range of materials used in the construction of buildings, household equipment, interior of cars and wastes. These included mainly construction materials used in the past three decades, new and used electrical devices, flooring, fabric, upholstery and other daily-use materials. Samples of recycled materials were also included, as their contamination from primary materials remains unclear. New materials were purchased while older and used materials were collected from various sources. These materials were also analyzed for flame retardants, presented elsewhere (Vojta et al., in prep).

A total of 126 samples were split into four categories according to use and composition. The first category included household equipment; i.e. (1A) textiles, (1B) floor coverings, (1C) electrical & electronic equipment (EEE), and (1D) plastics. The second category included building materials; i.e. (2A) oriented strand board (OSB), other composite wood and wood, (2B) insulation materials, (2C) mounting and sealant foam, (2D) facade materials, (2E) polystyrene and (2F) air conditioner components. Some building materials were supplied by a company dealing with the construction of low-energy houses. In the third category were car interior materials. The final group consisted of wastes of electrical & electronic equipment (WEEE) collected at a sorting plant. See Supplementary Materials Table SI.2 for a detailed list of sample categorization.

### 2.2. Sample preparation and extraction

Samples of solid materials were crushed, chopped or ground while samples of foam and fabrics were cut into small pieces. After grinding or cutting, 5 g of each sample was extracted with methanol with the addition of ammonium acetate (final concentration 5 mM) using warm Soxhlet extraction (60 min warm Soxhlet followed by 30 min of solvent rinsing) in a B-811 extraction unit (Büchi, Switzerland). Following extraction, samples were cleaned up according to the procedures for PFAAs analysis described in detail elsewhere (Karásková et al., 2016). Briefly, concentrated extracts were filtered using a syringe filter (nylon membrane, 13 mm diameter and 0.45 µm pore size). The filtrate was concentrated using a stream of nitrogen in a TurboVap II (Caliper LifeSciences, USA) concentrator unit to 500 µL and transferred to a minivial.

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