

Kristine Hunsbedt Ersdal

Per- and polyfluoroalkyl substances in clothing textiles, and their implications for the environment, health and fabric recycling

KJ2900 - Bachelor project in Chemistry

Bachelor's thesis in Analytical and Environmental Chemistry

Supervisor: Alexandros Asimakopoulos

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Abstract

This thesis investigates the uses and effects of per- and polyfluoroalkyl substances (PFAS) in clothing textiles, and a literary search was conducted on selected PFAS in clothing and functional textiles. The results showed that legacy PFAS such as perfluorooctanic acid (PFOA) and perfluorooctane sulfonate (PFOS) are still present in clothing today, with detection frequencies of 43% and 23%. It was also found possible precursors such as fluorotelomer alcohols (FTOHs), and that these precursors may transform during weathering through use. Clothing textiles contribute to the release of PFAS to the environment, and could potentially be a source for human uptake. PFAS compounds pose a challenge in textile recycling, as well as other methods of disposal, and a global restriction is needed in order to minimize future use.



Figure 0.0.1: Graphical abstract

Sammen drag

Denne oppgaven tar for seg bruk og effekter av per- og polyfluoralkylstoffer (PFAS) i klestekstiler, og et litteratursøk om utvalgte PFAS i klær og funksjonelle tekstiler ble utført. Resultatene viste at historiske og regulerte PFAS som perfluoroktansyre (PFOA) og perfluoroktansulfonat (PFOS) fortsatt finnes i klær i dag, og ble detektert i henholdsvis 43% og 23% av prøvene. Mulige forløpere som fluortelomeralkoholer (FTOH) finnes også, og disse forløperne kan transformeres gjennom slitasje ved bruk. Klestekstiler kan bidra til utslipp av PFAS i miljøet, og være en potensiell kilde til menneskelig opptak. PFAS utgjør en utfordring for tekstilgjenvinning og andre former for avfallshåndtering, og en global restriksjon trengs for å minimere framtidig bruk.

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Abbreviations

Table 0.0.1: General abbreviations

Abbreviation	Full name
BFR	Brominated flame retardant
CEAP	Circular Economy Action Plan
CIC	Combustion ion chromatography
DWR	Durable Water Repellent
ECHA	European Chemicals Agency
EDI	Estimated daily intake
EFSA	European Food Safety Authority
EPA	United States Environmental Protection Agency
FR	Flame retardant
FTOH	Fluorotelomer alcohol
6:2 FTOH	6:2 Fluorotelomer alcohol
8:2 FTOH	8:2 Fluorotelomer alcohol
GC	Gas chromatography
HFPO-DA/GenX	Hexafluoropropylene oxide dimer acid
HPLC	High performance liquid chromatography
LOQ	Limit of quantification
MS	Mass spectrometry
MS/MS	Tandem mass spectrometry
NIR	Near infrared spectroscopy

Abbreviation	Full name
OECD	Organisation for Economic Co-operation and Development
PBT	Persistent, bio-accumulative and toxic substance
PFAS	Per- and polyfluoroalkyl substances
PFSA	Perfluoroalkane sulfonic acid
PFAA	Perfluoroalkyl acid
PFCA	Perfluoroalkyl carboxylic acid
PFBS	Perfluorobutanesulfonic acid
PFHxS	Perfluorohexanesulfonic acid
PFNA	Perfluorononanoic acid
PFOA	Perfluorooctanoic acid
PFOS	Perfluorooctane sulfonate
PIGE	Particle induced gamma-ray emission
POP	Persistent Organic Pollutant
pyr-GC/MS	Pyrolysis-gas chromatography-mass spectrometry
RAC	The Committee for Risk Assessment
REACH	The Regulation on the Registration, Evaluation, Authorisation and Restriction of Chemicals
SDGs	Sustainable Development Goals
SEAC	The Committee for Socio-economic Analysis
SFP	Side-chain fluorinated polymers
SVCH	Candidate List of Substances of Very High Concern for Authorisation
TF	Total fluorine
THP	Total hydrolysable precursor
TOP	Total oxidizable precursor
WR	Water repellent

1 | Introduction

The textile industry has never been bigger, and we have never bought more clothes than today. Fabric manufacturing has increased by more than 80% since the 1980s [1], and is expected to continue to grow, as the average person today consumes 13 kg textiles annually [2]. The rise of "fast fashion", describing how clothing today is sold cheap, with new pieces constantly being released to keep up with rapidly changing trends, has altered how we consume textiles [3], and lead to a throw-away culture where clothing is easily disposable. This means that as well as being associated with poor working conditions, high water consumption, up to ten percent of global greenhouse gas emissions and environmental pollution during production, the fashion industry leads to enormous amounts of waste, both industrial and post-consumer [4]. Millions of tonnes, ca. five percent of total world wide waste, is caused by textiles. According to a report from 2017, only 23% of textiles are recycled, and less than one percent is made into new textile fibers in closed loop-recycling. The remaining 75% end up in landfills [5]. In the EU, an average consumer throws away 11 kg textiles each year [1], and global textile waste in 2030 is expected to reach 148 million tonnes [6].

One of the United Nations 17 Sustainable Development Goals (SDGs) from 2015, SDG 12, concerns "Responsible consumption and production", and has sub-goals regarding reducing waste through "reducing, reusing and recycling", as well as management of chemical usage and waste [7]. This means going from a linear economic model of consumption and disposal, to a circular economy, focusing on prolonging life cycles of products, saving resources and re-purposing [2]. Circular economy is also one of the main parts of the European Union's "European Green Deal", in the "Circular Economy Action Plan" (CEAP), aiming to prevent waste and create more sustainable products [8]. This includes strategies for sustainable and circular textiles, striving for recyclable fabrics without toxic substances.

Because clothing do not just consist of textile fibers, it is also associated with numerous classes of hazardous chemicals. Ever since the middle of the 1800s, when the first synthetic dyes were invented [9], chemicals have been used as additives in textiles, either added intentionally to achieve wanted properties in clothing, or as residual contaminants from production processes. Some of these include brominated flame retardants (BFRs), trace metals from dyeing or nano-particles used for antibacterial purposes. Aromatic azo dyes, benzothiazoles with derivatives used as UV-stabilizers and phtalate plasticizers are additional examples. Many of these additives are known to pose environmental or health-related challenges, and are regulated today [10]. Per- and polyfluoroalkyl substances (PFAS) is a well known group of longtime use additives in fabrics.

This paper will investigate what kind of PFAS are present in clothing textiles today, their role in environmental and human exposure and implications for fabric recycling in a circular economy.

2 | Theory

2.1 Clothing textiles and recycling

Clothing textiles are composed of fibers of natural, regenerated or synthetic polymers, or blends of different materials. Cotton, wool, linen and silk are examples of natural textile fibers, viscose of a regenerated one, whereas polyester, nylon and acrylic are man-made from petroleum oil, constituting about 63% of all textile fibers[2]. Knitting or weaving connects the fibers into fabric.

Because of their varying molecular polymer structures, textiles made from different fibers are recycled using separate methods. Most textile recycling today is "open-loop", meaning the fibers aren't used to produce new clothing apparel, but the goal is fiber-to-fiber closed loop recycling [1]. The two major textile recycling technologies today are mechanical and chemical. A drawback for both methods is that garments have to be manually sorted beforehand, as no way of automated identification through for instance NIR-spectroscopy, has proven sufficiently accurate or efficient. Through mechanical recycling, textiles are shredded, ground, and fibres respun, alternatively melted and extruded if synthetic thermoplastic polymers, without altering the chemical structures, and the technique is commonly used for natural fibers and polyester, but scarcely on blended fabrics [1]. A disadvantage of the method is that fiber length often deteriorates.

Chemical recycling is done by converting the textile polymers into monomers, and then using these to create new fibers, meaning that one can produce longer fiber lengths. However, it brings environmental concerns because of its use of hazardous chemicals. First dyes are removed through extraction or destruction, such as oxidation. Other pre-processing to try to remove contaminants may also be done. Fibers are then hydrolyzed, often via acid or alkaline hydrolysis and then re-polymerized. Chemical recycling is also suitable for blended fibers.

Incineration is a third alternative of disposing of textiles, bringing the opportunity for energy recovery, but also the risk of hazardous gas emissions [1]. Pyrolysis to thermo-chemically decompose waste anaerobically yielding pyrolytic liquid, gas and char is another method [11], but high oxygen content in natural fibers is a challenge [1].

2.2 Per- and polyfluoroalkyl substances

2.2.1 Chemical structures

The term per- and polyfluoroalkyl substances refers to a group of anthropogenic chemicals in use since the 1950s [12]. According to the United States Environmental Protection Agency's "CompTox Chemicals Dashboard" database, almost 15 000 different PFAS-compounds exist today [13] [14], and are used in a wide variety of consumer products and industrial processes. More than 200 different categories of use have been identified, such as firefighting-foams, cosmetics, paints, food packaging, ski wax, non-stick cookware, semiconductors and even guitar strings [15]. Industrially, PFAS is manufactured through electrochemical fluorination or telomerization [12].

Chemically, PFAS can be divided into multiple subcategories. In the perfluoroalkyl compounds, all H-atoms that are not part of the functional group have been substituted for fluorine atoms. Defined by Buck et al. in 2011, they contain the perfluoroalkyl moiety, $C_nF_{2n+1}-$. Examples include the perfluoroalkyl acids (PFAAs), such as PFOA, and PFOS, see Figure 2.2.1a and 2.2.1b, who are carboxylic and sulfonic acids respectively (PFCAs and PFSAAs). These were two of the earliest commercially available compounds [12] [16, s.3]. Furthermore, the PFAAs are often classified as "long or short chain", where a PFCA with more than seven, and PFSA with more than five C-atoms are described as "long chain" [12]. A polyfluoroalkyl compound on the contrary, does not have all H-atoms substituted, and fluorotelomer alcohols such as 8:2 FTOH, see Figure 2.2.1c, are important examples [12]. In addition to the non-polymeric classes, there are also several types of fluorinated polymers, containing fluorine in either the backbone of the polymer, or the side chains as sidechain fluorinated polymers (SFPs), shown in Figure 2.2.1d with six fluorinated carbon atoms in the side-chain [12]. In 2021, the OECD proposed a broader definition, classifying all substances containing a fully fluorinated methyl or methylene C-atom without any other halogenic substituents as a PFAS [17].

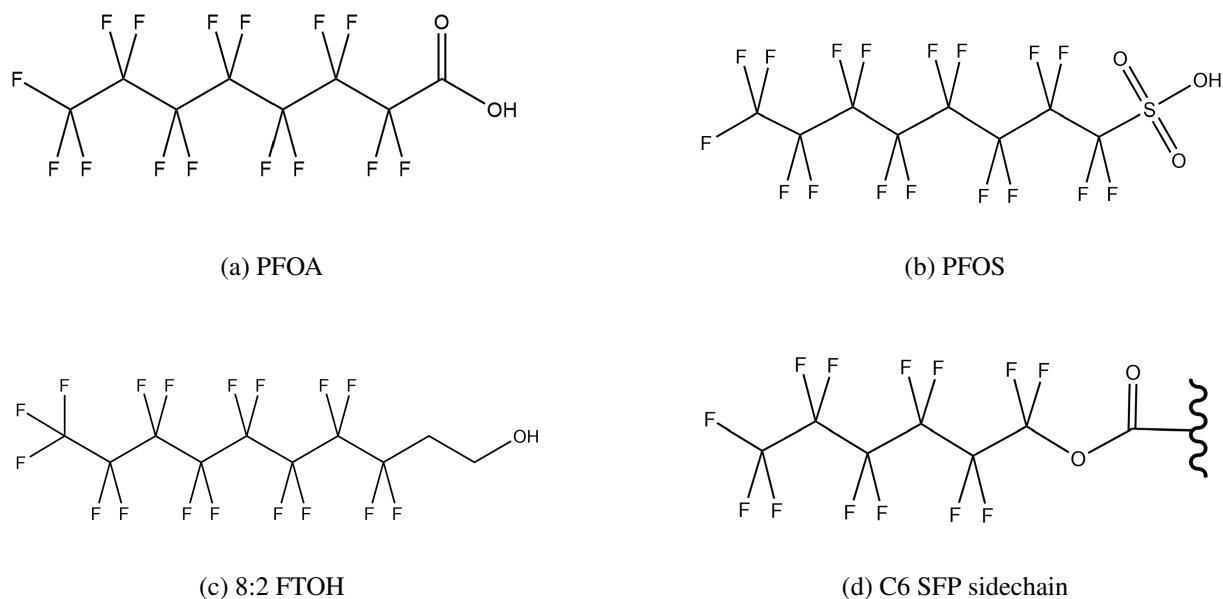


Figure 2.2.1: Four examples of different-category PFAS. Subfigure (a) shows the carboxylic acid PFOA, (b) the sulfonic acid PFOS, (c) 8:2 fluorotelomer alcohol, and (d) a SFP sidechain with six fluorinated C-atoms.

2.2.2 Environmental and health concerns

2.2.2.1 Persistence and distribution

The carbon-fluorine chemical bond is very strong [16, s.3], as fluorine is the most electro-negative element, and make PFAS highly chemically and thermally stable [18]. This chemical stability makes the per- and polyfluoroalkyl substances persistent in the environment, meaning they have long degradation half lives. Degradation is possible via incineration [19], but requires high temperatures such as 900°C [20], thus there is no effective biotic or abiotic way to cleave the C-F bonds in a natural environment. It is known that SFPs can hydrolyse and release their side chains as nonpolymeric PFAS, and that polyfluoroalkyl substances can serve as precursors for perfluoroalkyl ones, as the non-fluorinated part of the molecules can react [18]. Fluorotelomer alcohols may for instance degrade to PFCAs, both in the atmosphere [21], and through biotransformation in humans and animals such as fish and rats [22]. However, the final perfluoroalkyl moieties will persist, and PFAS are thus often called "forever chemicals" [18].

The persistence of PFAS has been in focus since the early 2000s following detection in human and wildlife blood, and it has been argued that the persistence itself should be a sufficient requirement for regulation of the substances, since one does not yet know all long term-effects of

contamination already in the environment, even though new emissions are reduced [19]. PFAS are released both as industrial waste, including the textile industry, and from use of consumer products, and is thereafter distributed throughout the environment, both via air and water [23]. PFAS are ubiquitous today, even detected in remote locations such as the Arctic and Antarctic [24]. Volatile PFAS such as FTOHs have estimated lifetimes of about 20 days in the atmosphere, and can be atmospherically transported over long distances [25]. Long chain PFAS are generally less volatile and less mobile in water than short chain-ones, but can undergo long range transport by adsorption to particles in air or water [23].

2.2.2.2 Bioaccumulation and toxicity

Long chain PFAS in particular have been shown to bioaccumulate in both humans and animal species by binding to blood proteins such as human serum albumin [26], as well as biomagnify in food chains [18]. Estimations of half lives of PFAS in humans vary, but range from 1.5- 5 years for PFOA and 2-6 years for PFOS [27]. Animal PFAS exposure has among other effects shown to delay brain development in mice [18], and exposure to humans can also lead to a variety of detrimental health effects, such as being carcinogenic and linked to breast- and testicular cancer. They are also known endocrine disruptors and immunotoxic, leading to lowered responses to vaccines [28], and are associated with liver and cardiovascular disease. Exposure via contaminated drinking water [23], ingestion, inhalation and dermal uptake from consumer products are known pathways for human exposure, where dietary exposure is considered the principal pathway [29]. The European Food Safety Authority (EFSA) has set a safe weekly intake limit of 4.4 ng/kg·body weight/week in 2020, for PFOA, PFOS, perfluorononanoic acid (PFNA) and perfluorohexanesulfonic acid (PFHxS), which they assume to have similar toxicological effects [18].

2.2.2.3 Regulatory work

Due to the concerns regarding detrimental effects, several PFAS are subject to restriction under different regulations in different regions of the world. The European Chemicals Agency (ECHA) added PFOA to its "Candidate List of substances of very high concern (SVCH) in 2013, and the EU has set a limit for the compound in articles at < 25ng/g [30]. Later on, other PFAS such as long-chain C9-C14 PFCAs, perfluorobutanesulfonic acid (PFBS) and PFHxS, which are sulfonic acids, and hexafluoropropylene oxide dimer acid also known as GenX, a short-chain substitution for PFOA, have also been added to the list. PFOS is also restricted in the EU's POPs regulation [31] [32], and a limit of 10 µg/g in articles or 1 µg/m² for coated textiles has been set

[30]. A few PFAS are also regulated internationally through the Stockholm Convention. These are registered as persistent organic pollutants (POPs), meaning they meet the criteria of being persistent, bioaccumulative and toxic organic compounds (PBT) [19]. As of today, PFOA and PFHxS are listed to be eliminated, while PFOS is listed for restriction [33].

Due to their restrictions, PFOA and PFOS are often called "legacy PFAS", but less is known about health and environmental effects of emerging and unregulated PFAS synthesised to replace old ones [25]. In 2023, the Scandinavian countries along with Germany and the Netherlands, proposed via ECHA to restrict all PFAS under REACH [34]. If approved, this will be the first legislation to place restrictions on a whole class of chemicals based on structure, as opposed to individual compounds. Currently the proposal is being evaluated by RAC and SEAC based on feedback, and a meeting concerning the use of PFAS in textiles is scheduled for September this year [35]. Still, this proposal would only concern European countries, and production of restricted PFAS in other parts of the world will still be a source of new contamination [23].

2.2.3 Use in textile production

As PFAS compounds contain a hydrophilic head from the functional group, and a hydrophobic tail from the fluorinated part [16], they are useful as surface-protectants. Surfaces coated with the fluorochemicals obtain a very low surface energy, meaning water or oil will not spread [16, s.3-4], and this can be utilised in textile production to manufacture water- and stain repellent garments [15]. In a review article from 2023, it was found 72 different PFAS in 381 textile samples, with a mean concentration of 2.48 $\mu\text{g/g}$ [36]. In clothing textiles, these types of coatings are often called "Durable Water Repellent" (DWR), and many use SFPs with FTOH side-chains, even though other alternatives such as silicone and hydrocarbon based ones exist. DWRs are commonly utilised in outdoor clothing, often with six carbon (C6) and eight carbon (C8) based coatings [37].

2.2.4 Methods of analysis

PFAS can be complicated to analyse because there are so many compounds, and different methods are needed. Some are extractable, while others, such as SFPs often used in textiles, are not [38]. There might also be a lack of available standards for unknown PFAS and precursors [39]. Still, non-targeted and suspect analysis are performed for several groups of consumer products [36]. Extraction followed by a type of chromatography coupled with mass spectrometry is common, for instance high-performance liquid chromatography–tandem mass spectrometry (HPLC-MS/MS) is

commonly used for ionic PFAS such as PFAAs, while gas chromatography (GC) can be used for volatile neutral PFAS such as FTOHs [39] [40]. Pyrolysis-gas chromatography-mass spectrometry (pyr-GC/MS) may also be utilised without extraction [41].

For non-extractable PFAS such as SFPs and precursors, one can use the total oxidizable precursor (TOP)-assay before analysis. The method was developed in 2012 [39], and makes it possible to quantify unidentified precursors by oxidising them to same-length PFAAs [42]. A variant of this is the Photo-TOP assay [38]. In a similar way, FTOH residues in polymers may be detected after use of a total hydrolysable precursor (THP)-assay [39] [43]. Total fluorine (TF) in a textile can be measured by particle induced gamma-ray emission (PIGE)-screening or combustion ion chromatography (CIC) [38].

3 | Results

3.1 Selected PFAS in clothing textiles

Table 3.1.2 show compiled results from a literature search on several studies and reports determining selected PFAS in a variety of clothing and functional textiles in recent years, such as shirts, outdoor apparel and infant clothing. Some analyses of functional-, outdoor- and home textiles such as firefighter gear, raw fabrics, tents and pillowcases have been included as supplement due to limited studies done on apparel. PFOA, PFOS, 6:2 FTOH and 8:2 FTOH as well as sum of total PFAS where reported, were selected as analytes to include, due to them commonly being targeted in analyses. Legacy PFAS such as PFOA and PFOS are still relevant to investigate even though they are restricted, as old clothing from before regulations could still be in use. Total PFAS is reported seeing as many other PFAS that could not be included due to the limited scope of the thesis, may be present. TF has also been included where screened for. Where samples may have been re-analysed, due to aging experiments or treatment such as TOP- or THP-assay, these results have been included under the original samples. Abbreviations for fiber materials are shown in Table 3.1.1.

Table 3.1.1: Fiber materials in the investigated samples

Abbreviation	Fiber type
CT	Cotton
EL	Elastane
LT	Leather
NY	Nylon
OCT	Organic cotton
O	Other
PAC	Polyacrylic
PA	Polyamide
PE	Polyester
PU	Polyurethane
RPE	Recycled polyester
VN	Vinyl

Table 3.1.2: Determined concentrations of selected PFAS and sum of total PFAS in clothing textiles in previous studies. Reported in median concentration unless stated otherwise, where n refers to number of samples.

Garment type	Fiber	Unit	PFOA	PFOS	6:2 FTOH	8:2 FTOH	ΣTotal PFAS (TF)	Year	Study
Outdoor textiles (n=5)	n.r.	μg/m ²	0.431 (n=3)	9.5 (n=3)	6.5 (n=4)	44.2 (n=4)	102.7	2015	[44]
Gloves (n=3)	n.r.	ng/g	0.93	1.5	8.2 (n=1)	53.2 (n=1)	125.7	2015	[44]
School uniform shirts/pants (n=9)	n.r.	ng/g	-	-	n.d.	406	-	2015	[45]
Leather jackets (n=2)	LT, CT	μg/m ²	0.0185	n.d.	0.71 (n=1)	0.6 (n=1)	-	2015	[46]
Shell jacket (n=1)	PU	μg/m ²	0.087	n.d.	n.d.	n.d.	-	2015	[46]
Bathrobe (n=1)	PE	μg/m ²	n.d.	n.d.	373	n.d.	-	2015	[46]
Rain jackets (n=7)	PE, NY, PA, PU	μg/m ²	0.22	n.d.	9.95 (n=2)	13,2	21,1	2016	[40]
For re-analysis (n=1)			1.45	n.d.	1.29	39.5	57.9		
3 years later			1.28	n.d.	<LOQ	3.98	10.36		
Hardshell jackets (n=7)	NY, PA, PU, O	μg/m ²	0.67	0.025 (n=4)	<LOQ (n=6)	35.3	41.9	2016	[40]
For re-analysis (n=3)			1.03	n.d.	<LOQ (n=6)	65.4	76.2		
3 years later			1.95	n.d.	<LOQ (n=2)	8.77	21.32		
Softshell jacket (n=1)	PE, EL	μg/m ²	0.68	0.01	<LOQ	1.70	5.68	2016	[40]
Work jacket (n=1)	O, CT	μg/m ²	171	0.54	<LOQ	14.8	458	2016	[40]
Diapers (n=2)	n.r.	μg/m ²	0.86a	0.04a	-	-	-	2016	[47]
Shirt (n=2)	n.r.	μg/m ²	0.04a	0.57a	-	-	-	2016	[47]
Pants (n=3)	n.r.	μg/m ²	0.15a	1.65a	-	-	-	2016	[47]
Footwear (n=2)	n.r.	μg/m ²	0.18a	1.63a	-	-	-	2016	[47]
Uniforms (n=3)	n.r.	μg/m ²	0.21a	1.37a	-	-	-	2016	[47]
Outdoor jackets (n=11)	n.r.	μg/m ²	0.175 (n=8)	0.25 (n=2)	130 (n=7)	56 (n=1)	106 (n=9)	2016	[48]
Outdoor trousers (n=8)	n.r.	μg/m ²	0.58 (n=5)	0.17 (n=1)	150 (n=7)	129 (n=2)	150.5	2016	[48]
Shoes (n=7)	n.r.	μg/m ²	2.88	1.07 (n=1)	1300 (n=5)	550 (n=3)	1400	2016	[48]
Backpacks (n=8)	n.r.	μg/m ²	0.44	0.09 (n=1)	n.d.	54.5 (n=2)	1.31	2016	[48]
Sleeping bags (n=2)	n.r.	μg/m ²	7.10 (n=1)	-	41 (n=1)	52 (n=1)	60.1	2016	[48]
Tents (n=2)	n.r.	μg/m ²	0.36	-	n.d.	12 (n=1)	7.07	2016	[48]
Outdoor jackets (n=3)	n.r.	ng/g	3.15 (n=2)	n.d.	1405 (n=1)	n.d.	4.6	2017	[49]
Windstopper (n=1)	n.r.	ng/g	6.6	n.d.	n.d.	n.d.	12.3	2017	[49]
Shoes (n=2)	n.r.	ng/g	n.d.	n.d.	n.d.	n.d.	0.55	2017	[49]

- not analysed; n.d. not detected; n.r. not reported; a mean concentration; b range; c read from graphical results

Garment type	Fiber	Unit	PFOA	PFOS	6:2 FTOH	8:2 FTOH	ΣTotal PFAS (TF)	Year	Study
Childrens textiles (n=2)	n.r.	μg/m ²	<LOQ	<LOQ	7.0 (n=1)	101	368 (530 (n=1))	2017	[50]
Produced by TOP			9.45 (n=2)						
Jackets (n=4)	n.r.	μg/m ²	0.45 (n=1)	<LOQ	370 (n=3)	255	1046.6(970)	2017	[50]
Produced by TOP			7.35 (n=4)						
Hat (n=1)	n.r.	μg/m ²	n.d.	0.69	n.d.	22	23.4	2017	[50]
Produced by TOP			5.9						
Pillowcase (n=1)	n.r.	μg/m ²	3.3	n.d.	11	1300	1548.1(1600)	2017	[50]
Produced by TOP			22						
Used children's swimsuit (n=1)	n.r.	ng/g	-	-	<LOQ	81	81	2018	[51]
Used children's vest (n=1)	n.r.	ng/g	-	-	130	1300	1830	2018	[51]
Jacket (n=1)	n.r.	ng/g	-	-	180000	-	180000	2018	[51]
Used firefighter jacket (n=1)	n.r.	ng/g	-	-	300	1800	2770	2018	[51]
Infant clothes (n=81)	CT, PE, NY	ng/g	n.d.-0.748b (n=4)	n.d.-0.570b (n=4)	-	-	0.953	2020	[39]
After TOP			n.d.-1.70b (n=17)	n.d.-28.6b (n=48)			8.93		
WR textiles (n=56)	PE, CT, NY, VN	ng/g	n.d.	n.d.-33.9b (n=15)	-	-	2.78	2020	[39]
After TOP			n.d.-6.56b (n=9)	n.d.-1349b (n=52)			53.1		
FR textiles (n=23)	PE, CT, NY, VN	ng/g	n.d.-0.480b (n=1)	n.d.-146b (n=6)	-	-	7.83	2020	[39]
After TOP			n.d.-3.70b (n=2)	n.d.- 880b (n=9)b			247		
New firefighter jacket (n=2)	n.r.	ng/g	306 (n=1)	n.d.	-	-	1061.8-91455.8b	2020	[52]
Used firefighter jackets/pants (n=2)	n.r.	ng/g	37-1018	2-7b	-	-	412.6-105284.8b	2020	[52]
New inner thermal liners (n=16)	n.r.	μg/g	-	-	-	-	(50)	2020	[52]
New outer thermal liners (n=18)	n.r.	μg/g	-	-	-	-	(105)	2020	[52]
New outer shells (n=20)	n.r.	μg/g	-	-	-	-	(21500)	2020	[52]
Used inner thermal liners (n=9)	n.r.	μg/g	-	-	-	-	(72)	2020	[52]
Used outer thermal liners (n=9)	n.r.	μg/g	-	-	-	-	(145)	2020	[52]
Used outer shells (n=10)	n.r.	μg/g	-	-	-	-	(15700)	2020	[52]
Used thermal liners (n=3)	n.r.	ng/g	n.d.	4-11b	-	-	276-1904b	2020	[52]
After TOP			16-37b	4-12b			1526-4950b		
Used moisture barriers (n=3)	n.r.	ng/g	22-64b	7-96b	-	-	499-5748b	2020	[52]
After TOP			48-1300b	6-99b			2883-11802b		
Used outer shells (n=3)	n.r.	ng/g	24-190b	6-9b	-	-	359-2891b	2020	[52]
After TOP			38-237b	7-11b	-	-	1419-8562b		

- not analysed; n.d. not detected; n.r. not reported; a mean concentration; b range; c read from graphical results

Garment type	Fiber	Unit	PFOA	PFOS	6:2 FTOH	8:2 FTOH	ΣTotal PFAS (TF)	Year	Study
Outdoor trousers (n=1)	RPE	μg/m ²	0.11	n.d.	n.d.	4.6	12.91	2020	[53]
After aging			0.28	n.d.	45	62	163.54		
Jackets (n=4)	CT, PE, PA	μg/m ²	0.46 (n=3)	n.d.	76.5	14 (n=3)	196.7	2020	[53]
After aging			4.5 (n=3)	n.d.	102	69 (n=1)	189.34		
Outdoor textiles (n=8)	PE, CT, PA	μg/m ²	1.66 (n=4)	3.24 (n=1)	29	2.2	111.7	2020	[53]
After aging			0.26 (n=7)	0.235 (n=2)	114	260.2 (n=2)	689.5		
Childrens garments (n=19)	CT	ng/g	0.704 (n=13)	0.466 (n=2)	n.d.	n.d.	3.27	2020	[54]
Diapers (n=10)	OCT	ng/g	0.610 (n=9)	n.d.	n.d.	n.d.	4.02	2020	[54]
Dress, onesie, pants, shirt (n=20)	CT, PE	ng/g	0.662 (n=13)	0.229 (n=1)	n.d.	n.d.	3.46	2020	[54]
Onesie, socks, shirt, pants (n=18)	CT, SP	ng/g	0.517 (n=12)	n.d.	1.84 (n=1)	10.4 (n=3)	3.19	2020	[54]
Diapers, onesie (n=11)	PE	ng/g	0.739 (n=7)	n.d.	17.3(n=3)	n.d.	3.28	2020	[54]
Coat, onesie, pants (n=8)	NY	ng/g	0.671 (n=4)	n.d.	149 (n=3)	56.7 (n=5)	86.9	2020	[54]
School uniforms (n=25)	Several	ng/g	0.161 (n=5)	0.0247 (n=6)	445 (n=19)	9.50 (n=5)	728	2022	[55]
Pre-TOP/THP (n=5)			0.287 (n=1)	-	16200 (n=4)	9.49 (n=1)	16200		
After TOP			4.97 (n=1)	-	225 (n=4)		105000		
After THP					1220000 (n=4)	19.6 (n=1)			
Outdoor wear (n=16)	Several	ng/g	0.802 (n=12)	0.0719 (n=12)	30.4 (n=11)	16.8 (n=9)	111	2022	[55]
Mixed (n=16)	Several	ng/g	0.431 (n=9)	0.0521 (n=7)	6.74 (n=6)	13.8 (n=7)	35.5	2022	[55]
Awning fabric (n=1)	PAC,PE	μg/m ² (μg F/g)	n.d.	n.d.	n.d.	n.d.	(579)	2023	[38]
After Photo-TOP/dTOP			35/14d				600d		
After THP						300d			
Imitation linen (n=1)	PE,PU	μg/m ² (μg F/g)	n.d.	n.d.	n.d.	n.d.	(179)	2023	[38]
After Photo-TOP			15c				144		
After THP						70c			
Outdoor textile (n=1)	PAC,PE	μg/m ² (μg F/g)	n.d.	n.d.	n.d.	n.d.	(2064)	2023	[38]
After Photo-TOP/dTOP							273		
After THP					68				
Shower curtain (n=1)	PE	μg/m ² (μg F/g)	n.d.	n.d.	n.d.	n.d.	(19.1)	2023	[38]
After Photo-TOP/dTOP							178		
After THP					48				
Textiles C6 (n=5)	PA, PE, CT	μg/g	-	-	-	-	(931)	2024	[41]
Textiles (Fluorine-free) (n=5)	PA, PE, CT	μg/g	-	-	-	-	(21)	2024	[41]
Backpack (n=1)	n.r.	μg/g	-	-	-	-	(310)	2024	[41]

- not analysed; n.d. not detected; n.r. not reported; a mean concentration; b range; c read from graphical results

3.2 Estimated dermal uptakes from clothing

Table 3.2.1 gives an overview of previously calculated estimated daily dermal intakes (EDI_{dermal}) of PFAS along with corresponding reference values, using different Equations 3.1 and 3.2. Equation 3.1 used to estimate dermal uptake from hand wipes [29], is given below,

$$EDI_{dermal} = \frac{Q_{hw} \cdot t_{exp} \cdot F_{uptake-dermal}}{bw} \quad (3.1)$$

where Q_{hw} refers to the mass of PFAS on hands estimated from wipes (pg), t_{exp} being exposure time (day^{-1}), $F_{uptake-dermal}$ being uptake absorption of PFAS through the skin as a fraction [29] and bw being body weight (kg) [56]. The other expression Equation 3.2 used to determine uptake from clothing [55], is given as

$$EDI_{dermal} = \frac{C \cdot SA \cdot F_{contact} \cdot F_{mig} \cdot F_{pen} \cdot T \cdot N}{bw} \quad (3.2)$$

where the new variables C refers to extractable PFAS concentration (ng/m^2), SA is the total body surface area in m^2 , $F_{contact}$ is the contact area between skin and textile as a fraction, F_{mig} refers to the daily migration rate to skin from textile and F_{pen} the penetration fraction into skin. T is the contact duration time and N the daily mean number of events [55]. Equation 3.2 may also be used with a concentration per mass (ng/g), by multiplying the numerator by the textile density D , as done by Zheng et al. [54].

Table 3.2.1: Previously estimated daily dermal intakes from different studies

Equation	Reference values	Age group	EDI (pg/kg·bw/day)	Study
3.1	$t_{exp} = 1 \text{ day}^{-1}$ $F_{uptake-dermal} = 0.48$ $bw = \text{individual}$	Adults	0.03 (median) 0.01-13 (range)	[29]
3.2	$SA = 1.08 \text{ m}^2$ $F_{contact} = 0.824$ $F_{mig} = 0.001 \text{ day}^{-1}$ $F_{pen} = 0.5$ $T = 0.42 \text{ day}$ $N = 1 \text{ day}^{-1}$ $bw = 31.8 \text{ kg}$	Children (6-11 years)	1003 (median) 0.2-222000 (range)	[55]
3.2	$SA = 2543 \text{ cm}^2$ $F_{contact} = 1$ $F_{mig} = 0.001 \text{ day}^{-1}$ $F_{pen} = 0.5$ $T = 1$ $N = 1 \text{ day}^{-1}$ $bw = 6.8 \text{ kg}$	Infants	9.74 (mean) 89.9 (max) 69.9 (mean, after TOP) 622 (max, after TOP)	[39]
3.2*	$SA = 2660, \text{ cm}^2$ $F_{contact} = 1$ $F_{mig} = 0.005 \text{ day}^{-1}$ $F_{pen} = 0.01$ $T = 1 \text{ day}$ $N = 1 \text{ day}^{-1}$ $bw = 7.4 \text{ kg}$ $D = 23.4 \text{ mg/cm}^2$	Infants (3-6 months)	1.39 (mean, non-NY) 37.6 (mean, NY) 0.0 (washed)	[54]

*Using fabric density

4 | Discussion

4.1 PFAS and precursors in textiles

Results in Table 3.1.2 show that different PFAS are present in many types of clothing and other functional textiles, and found in both decade old studies and more recent ones. Due to different units in different studies, some being reported by square-meter fabric and other per gram, and lack of reported densities for possible conversion, directly comparing the analytical concentrations becomes tricky, as different fibers do not have the same densities.

Still, it is evident that even though both PFOA and PFOS are restricted via the Stockholm Convention for elimination and restriction, they are regularly detected in apparel in varying concentrations, even as soon as in 2022 [55]. Out of 425 original samples analysed for PFOA without use of TOP- or THP-assay, the compound had a detection frequency of 43% (182). None of the median concentrations reported per gram were above the EU Commission limit, except for samples of used and new firefighter gear [52]. Since there is no area based limit for PFOA, but the mass based one is lesser than for PFOS, it is logical to assume that an area based one at least would not be higher than for PFOS. Using $1 \mu\text{g}/\text{m}^2$ as a theoretical limit for PFOA as well, several samples exceeded this, for instance sleeping bags, outdoor jackets and shoes [48]. PFOS was detected in a lower frequency of 23%, though a few samples exceeded the EU threshold limit for coated materials, for instance samples of outdoor textiles with a median of $9.5 \mu\text{g}/\text{m}^2$ in 2015 [44], almost ten times more than the limit, and one in 2020 with $3.24 \mu\text{g}/\text{m}^2$ [53].

Ideally there would not be any traces of PFOA or PFOS, but sources could be residues or contaminants from production processes or come from textiles from outside the EU. They could also come from known or unknown precursors in the textiles, and the results from the TOP and THP assay support this. Robel et al. [50] showed a substantial increase of detection frequency

and concentration of PFOA in all samples after oxidization, increasing in a range of 6-16 fold as seen in Table 3.1.2. The same was the case for the firefighter gear samples, and for Zhu et al. [39], where for instance the PFOS concentration of WR textiles increased from 33.9 to 1349 ng/g at most, and the total PFAS concentration also increased for all samples. Analysis of school uniforms [55] also showed increase of PFOA and total PFAS, but a decrease from 16200 to 225 ng/g of 6:2 FTOH. The subsequent THP-assay showed that the FTOH concentration increased, especially for 6:2 FTOH to 1.22 mg/g, strengthening the argument that FTOHs could be released via hydrolysis from SFPs and subsequently oxidized to PFAAs. The most recent study from 2023 also shows agreeing increases after TOP and THP. Total fluorine screenings done show measurable amounts of fluorine before the assays even though no extractable PFAS were detected, indicating the presence of other PFAS compounds than those included in Table 3.1.2 [38].

4.2 Weathering during use and laundry

Results from van der Veen et al.'s study on weathering of DWR clothing textiles [53] (Table 3.1.2) show that concentrations of PFAAs and volatile PFAS increased in outdoor trousers, jackets and textiles after simulated weathering. In the trousers analysed, the median concentration of both PFOA, 6:2 FTOH, 8:2-FTOH and total PFAS increased, where total PFAS went from 12.91 to 163.54 $\mu\text{g}/\text{m}^2$. For the jackets, PFOA concentrations increased, while median total PFAS went down and 8:2-FTOH was found only found in one garment after aging. In the outdoor textiles, the median went down but detection frequency up for PFOA and PFOS. The 8:2 fluorotelomer alcohol was only found in two out of eight samples after aging, whereas it was found in all before, while median 6:2 FTOH increased four times. Total PFAS increased by about six times in the raw textiles [53].

These concentration changes could be due to hydrolyzation of FTOH-sidechains from SFPs, which again may be transformed to PFCAs. It is also possible that there are unknown precursors or non-extractable fluorine in the coatings used, that oxidize to PFAAs. These transformations could explain the increased detection of PFOA, even where it was not detected originally. The estimates for the volatile FTOHs is a bit uncertain, as it is unknown how much of the compounds may have been volatilized and emitted to the air, how much is transformed and how much is being released from SFPs [53].

In a follow up study [37], the effect of laundering was also investigated, analysing textiles deliberately coated with C6 and C8 DWR by the authors. The results supported that PFAAs are formed from aging textiles, and that FTOHs can vary, potentially increasing or decreasing. Washing the textiles resulted in decreased concentrations, meaning that laundering can be a potential source of PFAS to the environment through waste water [37]. The same study also found an increased concentration in poly-amide after aging, compared to polyester treated with the same DWR coating. The authors contributed this to different weaving of the textiles, and difference in hydrophobicity, showing that fiber material is also a factor in the release of PFAS [37].

In the same way, Schellenberger et al. [57] conducted an outdoor aging study on self treated DWR fabric samples, finding that fibres were degraded, releasing micro fibers from the samples, and total fluorine decreased after 6 months of weathering. PFAAs showed a 100-fold increase, some exceeded the EU-limit afterwards, indicating precursors [57]. Gremmel et al., who analysed outdoor jackets, re-analysed some samples after three years of storage. They found that the FTOH concentration, including 8:2-FTOH (Table 3.1.2) and total PFAS concentration had decreased significantly. For the rain jacket, the PFOA-concentration also decreased, while the median increased for hardshell jackets. [40].

This supports the argument that PFAS-concentrations in apparel will not stay constant during its life-time, but it can be hard to predict an overall effect due to many interacting factors. This is especially true for volatile PFAS, who may be emitted to air, transform to PFAAs and themselves be formed from unknown precursors, as seen from data that FTOH-concentrations may both lower and increase over time. Still, it is likely that clothing is a source of PFAS to the environment, and that compounds that were not present in the original garment, are formed and released during normal use, including regulated PBT ones such as PFOA and PFOS.

4.3 Potential human uptake from apparel

Following up, a natural next question could be if clothing and other skin-contact textiles may be a significant contributor to uptake of PFAS in humans, and thus cause toxic effects. However, even though possible detrimental health effects of PFAS are well researched, there is a very limited knowledge of dermal uptake as a potential pathway [58]. Hardly any studies on pathways have investigated, but one from 2020 estimated a daily dermal uptake in adult from hand wipe samples to be 0.3% of total exposure using Equation 3.1 [29]. The median daily dermal uptake was calculated

as 0.03 pg/kg·bw/day, see Table 3.2.1, out of a total daily uptake of 8.1 pg/ kg·bw/day, and it was thus concluded that dermal uptake did not constitute a significant pathway [29]. However, this study did not take into account any other part of the skin which could be in direct contact with clothing for an extended period of time, and that the hands constitute only a small part of the of the skin surface area of about 2 m² for an adult human [58].

Xia et al. who analysed school uniforms and other children's textiles [55] (Table 3.1.2), estimated a median daily dermal intake (EDI) of 1.03 ng/kg·bw/day for children (Table 3.2.1), using Equation 3.2. This sums up to 7.21 ng/kg bw per week, well above the EFSA's limit, though the authors acknowledge this could be an overestimate due to taking volatile FTOHs into consideration for the penetration rate [55].

In contrast, Zhu et al. calculated dermal exposure from infant clothing using the same expression, but different values for SA and T , finding that the mean EDI_{derm} for PFAAs was 9.74 pg/kg·bw/day before TOP-assay and 69.9 pg/kg·bw/day after, not above the threshold limit (Table 3.2.1). The maximum result after TOP-assay however, adds up to 4.35 ng/kg·bw/week [39]. Zheng et al. incorporated the density of the fabric into the calculations, and found a mean EDI_{derm} of 1.39 and 37.6 pg/kg·bw/day for infants for non-nylon and nylon textiles respectively 3.2.1, and also found that there was no detectable concentration after washing [54].

Calculating the EDI_{derm} for a set of children textiles from [50] (Table 3.1.2), with a total PFAS concentration of 368 $\mu\text{g}/\text{m}^2$, using the same reference values as Xia et al., gives an estimated daily intake of 2.16 ng/kg·bw/day. In all, different estimations vary greatly, because found concentrations of PFAS vary quite a lot between individual samples. Due to the aforementioned effects of weathering and laundering, it can be hard to estimate the general uptakes from clothing. Individual factors concerning skin such as pH and temperature also play a part [58]. But depending on the garment, dermal uptake could contribute to total daily intake, though washing the garment could reduce the potential. Its also worth noting that a lot of the apparel where DWR is applied is outdoor wear, meaning that the garments probably are not in direct contact with the skin as one would wear layers underneath. One also probably does not wear a rain coat or outdoor trousers continuously for several hours every day, such that the EDI_{derm} for these types of garments would be lower. These could still contribute to inhalation as a pathway by leaching of volatile PFAS.

4.4 Implications for recycling and future outlook

Seeing as the results show that textiles containing PFAS may leach volatile and ionic compounds into the environment with aging, land-filling these garments as post-consumer waste is not ideal. But what should be done with them if we don't want to use them anymore due to their potential for human exposure? Mechanical recycling of PFAS-containing clothing will not transform the chemical compounds present in the textile, but PFAS might be carried over to new fibers. While one might suspect that an hardshell outdoor jacket is DWR-coated and thus may contain PFAS, one would probably not expect the same from bathrobes, infant onesies or shirts, where they were also detected (Table 3.1.2). Therefore there is a risk of unknowingly introducing PFAS into new garments made of mechanically recycled polymers.

According to Baloyi et al., there is a limited knowledge on the impact of chemical additives in chemical recycling [1], but a challenge regarding PFAS could be that many processes utilise hydrolysis. As seen by the THP-assay analysis, hydrolysis of DWR clothing containing SFPs may release side chains, meaning that one potentially could end up accumulating FTOHs or other precursors, which may then transform into PFCAs later. In total, this could be creating a higher PFAS concentration than present in the original garment. The fact that oxidation is a possible way of dye-destruction could be such a pathway for transformation. Even if one is successful in removing all PFAS from textile waste without contamination of new fibers, one has really just relocated the problem from one matrix to another, now having to get rid of chemical PFAS-waste instead. Disposal is thus a cyclical problem, and if one chooses to landfill the processing waste, PFAS would again leach out into the environment [59]. The only way to get rid of the PFAS as of today is via incineration, but the method might release unknown byproducts, transformation products and gases such as chlorofluorocarbons [59]. To consider pyrolysis as a possible option, more research on the effects of additives to textile waste is needed [11].

What could then be done about new emissions in the future? Cousins et al. [60] argued that a "concept of essential use" could be utilised to decide when PFAS should be phased out. They classified use of PFAS in waterproof clothing as substitutable, and in other leisure apparel as "non-essential". Silicon and hydrocarbon alternatives were deemed better options, as they are less persistent, even though research is needed to determine if these could have other adverse effects [60]. Protective clothing such as medical gowns and firefighter gear were deemed essential, as they

impact health and safety of workers, but with those exceptions, there is no need to continue using PFAS in textiles, seeing as they have potential for release into the environment, may contribute to human uptake, and are hard to recycle. Regulation of non-polymeric PFAS isn't enough, as SFPs, who were marketed as less problematic than traditional PFAS, may also leach. It is also evident from the results that even the regulated legacy PFAS are sometimes still used. In all, the proposed European PFAS restriction seems reasonable in regards to consumer clothing textiles. However, regulations, and monitoring of new and emerging PFAS are still needed on a world-wide scale, as the contamination is a global issue. Development of new technology for recycling and disposal is also necessary.

The analytical methods used in the reviewed studies could also be extended to other chemical classes. PFAS is just one of many groups of chemical additives in textiles who might also have detrimental effects, and knowledge is needed on these as well. There is a possibility in the future of expanding the TOP- and THP-assay, in order to better detect oxidizable compounds, for example in benzothiazole derivatives, or phtalates, leading to a better understanding of the chemical hazards present in our everyday clothing.

5 | Conclusion

PFAS in clothing textiles is a challenge, due to the wide variety of compounds used, and possible unknown precursors. Legacy PFAS such as PFOA and PFOS are still found in clothing today, and TOP- and THP-assay show the presence of precursors, which could be transformed to PFAAs through aging of the textiles. Experiments also show that PFAS is released to waste-water during laundering. There are varying opinions on whether dermal uptake could be a significant contributor for human intake, as this varies greatly with samples, but it is a plausible pathway. Neither mechanical nor chemical recycling is fit for PFAS-containing textiles due to carry-over or accumulation of the compounds in new fibers, but incineration and land-filling also comes with environmental challenges. A world-wide restriction is needed to prevent future use of non-essential PFAS in clothing, and alternative DWR coating methods should be utilised instead. The topic of PFAS in textiles should be investigated further, and the used methods of analysis also opens up for studies on other additives in clothing.

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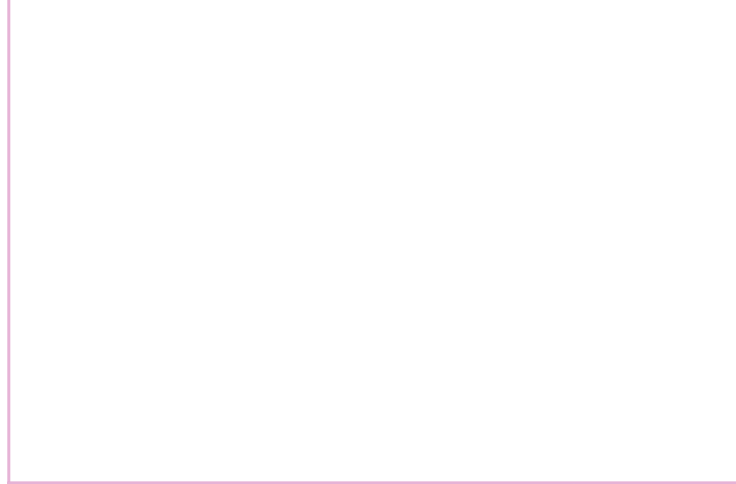
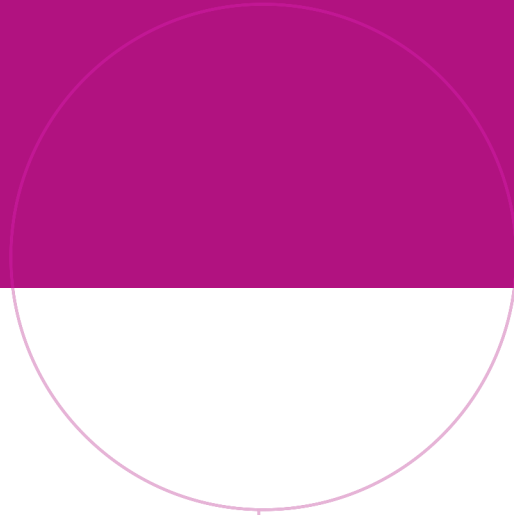
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