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Characterisation of "flushable" and "non-flushable" commercial wet wipes using Micro-Raman, FTIR spectroscopy and fluorescence microscopy: To flush or not to flush

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ABSTRACT

The introduction to the market of wet wipes, advertised and labelled as "flushable", has been the subject of controversy due to their perceived potential to block sewer systems as observed with other nonwoven cloths such as traditional non-flushable wipes. Nonwoven cloths that enter wastewater systems can find their way into the aquatic environment via wastewater effluents and it has been suggested that the breakdown of the these fabrics can release materials such as microplastics into the environment. Worldwide research has revealed the alarming number of aquatic organisms affected by the presence of plastic debris in the aquatic environment harbouring a potential risk to humans through the introduction of microplastics into the food chains. However, the actual material composition of "flushable" wipes, their fate and impacts in the aquatic environment have not yet been scientifically studied. This paper investigates the fibre composition of "flushable" and non-flushable wipes, specifically with regards to synthetic polymer material, using Fourier transform infrared (FTIR) and micro-Raman spectroscopy along with fluorescence microscopy. The study demonstrated the presence of polyester (polyethylene terephthalate, (PET)), high density polyethylene (HDPE) and polyethylene/vinyl acetate (PEVA/EVA) in some "flushable" wipes and PET in all non-flushable. Other polymers such us polypropylene (PP), low density polyethylene (LDPE), expanded polystyrene (EPS) and polyurethane (PU) were also identified as potential components in the "flushable" material. Hence, commercially available wet wipes labelled as "flushable" could also be considered as a possible source of microplastics fibres in the wastewater streams and, if not retained, in the environment.

Keywords: MicroRaman; Microplastic; Wet wipes; Flushable; FTIR; Fluorescent microscopy

INTRODUCTION

Plastic pollution of the aquatic environment is of increasing global concern due to its impact on marine ecosystems and wildlife (Waller , et al., 2017; Cesa, et al., 2017; Sigler, 2014), and carries the potential risk of entering the human food chain (Rochman, et al., 2013). The effects of plastic debris in the oceans have been well documented, including physical harm to marine fauna, from zooplanktonic organisms to mammals, through entanglement or ingestion of the synthetic materials (Wright , et al., 2013; Wilcox, et al., 2016). Microplastics, which are defined as plastic particles between 1µm and 5mm, are a growing source of contamination in aquatic environments, through being carriers of chemical pollution (containing plasticisers and additives) as well as vehicle for chemicals adsorbed onto their surface (Coppock, et al., 2017). Microplastics can form as the result of degradation or mechanical breakdown of larger plastic items, but they are also manufactured for use in some items such as cosmetics, hygiene products and synthetic fabrics. These are then discharged and transported into sewer systems and eventually enter the aquatic environment via wastewater outflows (Ziajahromi, et al., 2016).

Wastewater Treatment Plant (WWTP) effluents have been identified as a significant pathway of microplastics in the environment, and much attention has been paid to the presence of microbeads coming from personal care products (Baldwin, et al., 2016; Eerkes-Medrano, et al., 2015; Estahbanati & Fahrenfeld, 2016; Talvitie, et al., 2015) As a result, these are now in process of being banned for sale in the US (FDA, 2017) and the UK (DEFRA, 2017). However, the release of synthetic fibres in aquatic ecosystems still remains an issue that requires further investigation (Ziajahromi, et al., 2016). Plastic fibres may enter the sewers from washing synthetic textile clothes. However, the new development of the intentional disposal of nonwoven wet wipes into the system is of particular concern. This is due to their potential plastic content and alarming figures found in the aquatic environment (Browne, et al., 2011; Cesa, et al., 2017; Nelms, et al., 2017). For example, the Marine Conservation Society report on the Great British Beach Clean 2015 (MCS, 2017) concluded that the number of wet wipes found in the UK coastline has increased by 94% in 2017 and a total 400% increment during the last decade (27.5 pieces of wet wipes for every 100m of beach cleaned).

It is has been suggested that the situation is aggravated by the introduction of so-called "flushable" wipes to the market (Patchell, 2012). Currently, flushable wipes are made by bonding airlaid pulp with ion sensitive cationic polymer binders or by light hydroentanglement. Additionally, other types of flushable wipes consist of three layer composites with wetlaid pulp in the core supported by fibre webs, which can be made of either synthetic or natural fibres (Dipayan & Behnam, 2014). Manufacturers have claimed that these flushable products can be safely disposed through the sewage system, using labels such as "flushable tissues" and "biodegradable wipes". However, evidence from the water treatment industry has shown that some flushable wipes remain intact through the sewerage lines, causing blockages and damage to equipment in WWTP (WATER UK, 2016; Fam, et al., 2017). It is estimated that the cost of unblocking the system in the UK is around £88 million a year (Nelms, et al., 2017), and more than 50% of obstructions are caused by wipes and hygiene products. Even when wet wipes eventually break down, small amounts of plastics from the nonwoven fabrics may be released into the environment via sewage effluent (MCS, 2017).

The lack of regulations on flushability assessment and adequate labelling are additional concerns for environmental organizations and the water industry. The current guidelines on flushability have been published by the INDA (The Association of the Nonwoven Fabrics Industry) and its European partner EDANA (The European Disposables and Nonwovens Association). They include a series of tests such as toilet clearance and slosh box disintegration test, settling, biodegradation and sewage pump tests (INDA/EDANA, 2014). Nevertheless, the assessment is only voluntary and has been subject to criticism because it does not properly mimic the wastewater system (Flegenheimer, 2015; MCS, 2017). This issue has been discussed in the media, especially during recent years, but has not been approached scientifically yet with little environmental research applied to it (Mitchell, et al., 2017). Current studies on this matter have mainly focused on the disintegration of flushable products in wastewater systems (Karadagli, et al., 2012; Eren & Karadagli, 2012), from an industrial point of view, but there is still a gap in the literature on the actual composition of the materials and their potential to reach and harm the aquatic environment.

In this study we report the characterisation of several commercial wet wipes, including flushable and non-flushable wipes, in an effort to provide: (i) scientific evidence of the material composition, specifically the synthetic polymer material in wet wipes that are currently advertised as flushable, and (ii) to discuss the possible environmental impacts that flushing those wipes (as potential source of microplastics) may have in the aquatic environment. Fourier transform infrared (FTIR) and Raman spectroscopy are the gold standard techniques used in identification of microplastics from environmental samples (Lenz, et al., 2015; Löder & Gerdts, 2015; Song, et al., 2015). In this study, both techniques along with fluorescence microscopy were used as complementary tools to characterise accurately the fibres that make up the nonwoven wet wipes.

MATERIALS AND METHODS

Granular polyethylene terephthalate (PET) was obtained from Sigma Aldrich (429252). Acetonitrile (11373230), HNO_3 (N/2272/PB17), H_2O_2 (H/1750/15), tri-sodium citrate (S/3320/60) and citric acid (C/6160/62) were obtained from Fisher Chemical. Nile red (415710010) acetone (268310025), n-hexane (197360025) and sodium azide (190381000) were obtained from Acros Organics. All water used was passed through 0.45um filter (Purite).

Before analysis, wet wipes samples were cleaned by soaking in deionised water for 4h and then rinsed 3 times. After that, the wipes were soaked in acetonitrile and then air dried in a fume hood. For the FTIR bulk analysis, a Smith Detection HAZMATID Ranger FTIR instrument was used with a single bounce diamond ATR contact probe, 32 average scans, $4cm^{-1}$ resolution and $650\text{-}4000cm^{-1}$ range. All FTIR and Raman spectra shown were normalised to the highest intensity peak.

We used an in-house modified Raman microscope. A Witec CRM200 microscope with reflected light illumination was modified with a silver coated, N-BK7 right angle mirror (Edmund Optics, 89627) to deliver a 785nm laser from a Thermo Scientific First Defender RMX Raman handheld spectrometer (250 to 2875 cm^{-1} range and 7 cm^{-1} resolution) via the atomic force fibre optic port (resulting on a spot size of 20µm using 100X objective, Figure S4). Two infinity corrected objectives were used: Nikon (20X/040 wd 3.8) and Zeiss Ec Epiplan (100x/0.8). An Olympus objective micrometer (OB-MM-1/100, 10um per division) was used to measure laser spot size. The Raman spectra obtained were matched against the instrument database (version RM9_ENU) which contains 10,110 chemicals including plastic polymers. The database matching software does not report values analogous to correlation or similar "Hit Quality Indices". Instead, the processing software rules out library records if they are spectroscopically inconsistent. For those library records that cannot be ruled out, it reports posterior probabilities: the probability, based on the data, that the measured material is library material A versus B, etc. (Chalmers, et al., 2012)

Nile red staining was performed according to Shim et al, (2016). Briefly, a small amount of wipe sample was placed in a glass microscope slide and incubated with Nile red for 30 minutes (5mg/L in n-hexane, diluted from a 50mg/L stock solution in acetone). For image acquisition an Olympus BX41 microscope with fluorescent illumination, a mercury lamp and the following filters U-MNU (blue, ex 360-370, em 420-460), FITC 41001 (green, ex 480/40, em 535/50), Propidium iodide 41005 (red, ex 535/50, em 645/75) (Shim, et al., 2016). A Srate, MC500 5MP ccd camera was used to capture all images.

The hydrolysis of PET using microwave digestion has been reported for the recovery of terephthalic acid with high yield (98%). In this study we used such method as an additional mean to confirm the presence of PET in wipes (Cata , et al., 2017). For acid digestion, cleaned and dried wipes (<0.5g) were placed in 50mL Teflon tubes and digested in a Mars Xpress microwave by adding 9mL HNO_3, 2ml H_2O_2 @150^0C for 10min. Toilet paper was used as negative control. The digested mixture was centrifuged and washed with deionised water 3 times. The white precipitate was dried at 70^0C overnight and was ready to analyse.

Wet wipes used in this study are listed in Table 1 along with the ingredients, biodegradability and recycling instructions as stated by the manufacturer label.

RESULTS

Bulk FTIR analysis of different flushable and non-flushable wet wipes is shown in Figure 1A and 1B respectively. There were noticeable differences between the bulk FTIR spectra of flushable and non- flushable wipes (Figure 1C). However bulk analysis was not enough to match the samples to any of the two groups, this was because the spectra associated with some of the wipes samples such as sample F2 contained different peaks (Figure 1A, peaks at 1731, 1369 and 1235cm-1). The non-flushable wipes indicated good similarity with the spectrum of polyester (PET, Figure 1B). We subtracted the spectrum of cellulose (Toilet paper, Figure S3) from all spectra. The resulting spectrum was flat for all samples with the exception of F2 . The residual peaks for F2 after subtraction (Figure 1D), matched (0.812) the polymer polyethylene/vinyl acetate (PEVA/EVA). Using PEVA/EVA polymers to make fibres is not likely. However, coating fibres with PEVA/EVA polymers can increase fibres softness and flexibility (Olabisi & Adewale, 2016).

Fig 1 FTIR spectra of different flushable A), non-flushable B), cellulose based fibres subtracted from sample F2 matched to polyethylene/vinyl acetate (PEVA/EVA) C) and flushable/non-flushable comparison of wet wipes taken in bulk D), 32 average scans, $4cm^{-1}$ resolution and $650\text{-}4000cm^{-1}$ range

Bulk Raman analysis was not possible to perform due to the high fluorescence signal of the samples. Therefore micro-Raman spectra of flushable and non-flushable single fibres were taken and results are shown in Figure 2A and 2B respectively (microscopy images are shown in Figure S1 and S2). All non-flushable fibres contained polyester (PET). However, sample NF7 (Figure 2A), contained a different type of fibre (not matching PET), this fibre spectrum was similar but not identical to that of flushable wipe samples. Only one sample (F2) was highly fluorescent and no spectrum could be obtained in this way. According to visual inspection, all flushable samples contained two different types of fibres. The difference was mainly dependant on the relative fibres thickness along their length (Löder & Gerdts, 2015).

None of the spectra shown in Figure 2A (Figure S2) matched any entry on the instrument database (10,110 chemicals). It has been reported that complete masking of the polymer type during Raman spectroscopy analyses can occur. This is due to the addition of additives and other coatings during the manufacturing process of fibres (Lenz, et al., 2015). However, when a cellulose based spectrum (F6) was subtracted from sample NF7, the residual peaks matched the spectrum of amorphous titanium dioxide (142, 400, 514 and 635 cm^{-1}) (La Notte, et al., 2012)

For this reason we used Nile red staining along with green and red fluorescence as well as auto-fluorescence (blue emission) in an attempt to identify the remaining fibres in flushable wipes (Maes, et al., 2017).

Fig 2 Micro-Raman spectra of different flushable as well as second fibre found in sample NF7 wipes A), non-flushable B), and cellulose based (F6) subtracted from sample NF7 C) all spectra taken on single fibres at 100X magnification, spot size 20um, 785nm laser, 250 to 2875 cm^{-1} range and 7 cm^{-1} resolution. Polyethylene terephthalate (PET)

Results for Nile red staining of flushable wipes and controls (non-flushable wipes and Toilet paper) are shown in Figure 3. The results obtained using the red filter were not conclusive and therefore not used since the negative control (Toilet paper, Figure 3H3) did show red fluorescence.

Nile red fluorescence using a green filter has been reported for positive identification of polypropylene (PP), low density polyethylene (LDPE), high density polyethylene (HDPE), expanded polystyrene (EPS), polyurethane (PU) and polyethylene vinyl acetate (PEVA/EVA) (Shim, et al., 2016). Any one of such mentioned polymers, reported to become visible under green-filtered Nile red staining, may be components of those wipe products where positive test results were observed, namely, F2 , F4 and F5 , (Figures 3B2, 3D2, 3E2 respectively). It is worth mentioning that it is unlikely to find EPS as material for making fibres. This is because temperature affects its flexibility and fibres can also break at low temperatures. However, natural fibres reinforced with polystyrene composites do show improved mechanical properties (Singha & Rana, 2012).

Dim green fluorescence glow has been reported for polyvinyl chloride (PVC) and polyester (PES) (Shim, et al., 2016). We found dim glow in samples: F1, F3, F6, NF7 and NF1 Figures 3A2, 3C2, 3F2, 3G2 and 3I2 respectively. As expected Toilet paper samples did not show green fluorescence (Figure 3H2). Interestingly samples NF7 and NF1, Figures 3G2 and 3I2 respectively may contain PVC or PES in addition to polyester (PET), already identified.

Fig 3 Nile red staining analysis of different flushable and non-flushable fibres: A) F1, B) F2, C) F3, D) F4, E) F5, F) F6, G) NF7, H) Toilet paper , I) NF1 (1 no filter, 2 FITC green, ex 480/40, em 535/50, 3 PI red, ex 535/50, em 645/75)

Blue auto-fluorescence (in unstained samples) has been reported for polyamide (PA), PES and PET (Shim, et al., 2016). However, in our study we found bright blue fluorescence on the Toilet paper sample (negative control), this fact stopped us from drawing any conclusions about this method. We therefore do not recommend such methods in the identification of microplastics since one of the most common types of fibre in the environment (cellulose) can give false positive blue fluorescence.

Fig 4 Auto-fluorescence (blue U-MNU Ex 360-370, em 420-460) analysis of different flushable and non-flushable fibres: A) F1, B) F2, C) F3, D) F4, E) F5, F) F6, G) NF7, H) Toilet paper , I) NF1 (1 no filter, 2 blue)

The main limitation in the identification of microplastics using Nile red staining is the co-staining of natural organic material (Shim, et al., 2017). It is therefore advisable to remove or separate the synthetic and natural material.

Samples of flushable wet wipes were acid digested and the resulting products precipitated in an attempt to positively identify their composition. In this way any natural digested organic material will be completely soluble. Bulk Raman spectra of the white precipitate were collected and results are shown in Figure 5. Positive controls (PET standard, NF2 , NF1 , NF7 and NF6) were analysed in the same way. Flushable samples F1 and F4 gave positive identification for terephthalic acid (Figure 5A) which is one of the main precursors of PET. White powder obtained from sample F5 gave positive identification to white paint (Figure 5B, First defender database). The main two peaks in such spectra matched the spectra of titanium dioxide (Anatase, 444 and 610cm-1) (Lenz, et al., 2015).

Single fibres were picked up with tweezers under a stereo microscope; several fibres collected in this way were put together and placed on the diamond ATR to obtain FTIR spectra. Results for this analysis are shown in Figure 5C and 5D. Samples F4 and F1 gave positive identification for HDPE (Figure 5C, 0.788 and 0.832 respectively). Despite the fact that single fibres picked in this way looked identical under the microscope, once we subtracted the spectrum of HDPE for each sample spectrum, the residual gave positive identification for polyester (PET, Figure 5D, 0.913 and 0.855 respectively).

Fig 5 Bulk Raman spectra of different flushable wipes taken after microwave assisted acid digestion (9mL HNO_3, 2ml H_2O_2 @150 C for 10min), centrifuged and washed with deionised water A) database matched to terephthalic acid B) databased match to white paint, C) FTIR spectra of few single fibres collected from samples F4 and F1 D) FTIR spectra of samples in C after the spectrum of HDPE was subtracted

DISCUSSION

This study has demonstrated that some flushable wet wipes contain polyester (PET), HDPE and PEVA/EVA as well as potentially other plastics such as PP, LDPE, EPS and PU (Figure 3, only Nile Red stain indication was found), and therefore may contribute to the increasing problem of microplastics in the aquatic environment. This is particularly important since consumers have been encouraged to discard these products down the toilet. Once the nonwoven fabrics make their way into the sewage works and following their eventual breakdown, small amounts of the listed polymers can be released into the environment (Estahbanati & Fahrenfeld, 2016; Mintenig, et al., 2017).

As mentioned in the introduction, there are no clear regulations for the use and disposal of wet wipes. The current voluntary labelling ‘Code of Practice’ developed by the nonwovens industry (INDA/EDANA, 2014) is not completely adequate since it only describes flushability in terms of blockages but does not address the issue of wider environmental impact found in our study. It is for this reason, we believe that the lack of clear legal regulations regarding disclosure of fibre composition in flushable wet wipes, the lack of clear information about disposal on labels and the widespread use of these products, promote and encourage the disposal of all types of wet wipes (including non-flushable wipes) into the drainage system thus aggravating the negative environmental impact. More accurate standards for assessment of flushability are also required in order to control the increase of these mislabelled “flushable” products on the market.

From the wastewater treatment industry perspective, the main issue they faced with wet wipes, i.e. blockages in the sewer system and damages to WWTP facilities (Mitchell, et al., 2017), have been addressed with the introduction of the flushable alternatives. To date, little scientific research has been carried out to investigate further environmental repercussions specifically concerning the presence of plastic materials in wipes displayed as "flushable" and their fate in the environment.

Our analysis of individual fibres in the nonwoven wipes has revealed the presence of mainly polyester (PET) fibres in non-flushable wipes and a combination of cellulose and polyester (PET), HDPE and PEVA/EVA fibres in some of the "flushable" wipes. Different international studies on concentration and characterisation of microplastics in WWTP discharges have concluded that synthetic fibres are the dominant type of wastewater-based microplastics (Browne, et al., 2011; Dris, et al., 2015; Ziajahromi, et al., 2017). Browne et al. (2011), found that the most common microplastics in three different WWTP final effluents in Australia were PET fibres. Similarly, Dris et al. (2015), identified fibres as the main type of microplastic in secondary effluent from a WWTP in France. In a Scottish study, Murphy et al. (2016) found fibres to be one of the principal contributors to microplastics after plastic flakes in final effluent, the most common polymer being polyester (including PET, 32%) and the least polystyrene (4%). The recurrence of synthetic fibres has been generally attributed to washing of textile products. However, from the findings of this study, it can be stated that wet wipes may also be a substantial source of synthetic fibres. It has been reported that in 2015 the market of flushable wipes had a significant increase to 55,720 tonnes and $1.4 billion in sales compared to $796 million in 2010, estimated to double by 2020 (Smithers Pira, 2015). Due to the increasing demand for these products, added to the consumer misconception regarding what should or should not be flushed down the pipes, more attention needs to be directed to this matter at all levels (scientific, social and political).

Although some wastewater treatment plants have been reported to efficiently remove microplastics from final effluents, with 90% removal after tertiary treatment (Ziajahromi, et al., 2017) and up to 98% after advanced filtration (Mintenig, et al., 2017), wastewater related microplastics are still an important pathway considering the large volumes of effluent that reach the aquatic environment. Another important issue is the fate of the plastic fibres in the wastewater treatment plants. Studies have demonstrated that the synthetic fibres are mostly retained in sewage sludge (Leslie, et al., 2017) which in some countries is used as conditioner and soil fertilizer, transferring the microplastic pollutants into the soil matrix (Salvador Cesa, et al., 2017). Moreover, there is evidence that plastic fibres transfer through daily wastewater effluents into receiving waters and disperse in aquatic ecosystems, where they can cause harm to a variety of organisms through ingestion (Ziajahromi, et al., 2017). Leslie et al., (2017), found synthetic fibres to be present in four out of five species examined from the Dutch coast, with oysters and mussels having the highest concentration of microplastics (up to 105 particles g^{-1}). In another study, fish from the UK coast were found to have sewage-related microplastics in their digestive tracts, which were mostly in fibre form (Ivar do Sul & Costa, 2014). In order to mitigate these impacts, it is crucial to minimise the entries of plastic material into the sewage system. This is an important issue as there is increasing evidence that not only "flushable" but also non-flushable wipes are disposed through the sewage pipes (WATER UK, 2016; Fam D, Turner A, Latimer G, Liu A, Guirco D, & Sta, 2017).

It is alarming that although some manufacturers list as many as 26 ingredients in their wet wipes (Table 1), none of them disclose the fabric material they use in either their flushable or non-flushable wipes. Another issue with labelling is that some manufacturers advertise their wipes as biodegradable although they contain synthetic non-degradable polymers. We found that one manufacturer stated instructions for flushability and non-flushability within the same packaging, thus creating confusion to users.

CONCLUSIONS

The presence of plastic fibres was confirmed in 7 different brands of non-flushable wet wipes and in 3 brands of so called "flushable" wipes, including baby wipes, cleaning wipes, and personal hygiene wipes. Polyester (PET) was the most common polymer in all fibres followed by HPDE and we found one sample containing PEVA/EVA. Additionally, any one of the polymers reported to become visible under green-filtered Nile red staining (PP, LDPE, HDPE, EPS and PU) may be present in all 6 brands of flushable nonwoven wipes.

Previous recent studies regarding microplastic pollution from wastewater treatment effluents in the aquatic environment have shown that plastic fibres, mainly associated with washing of synthetic clothing, are the most predominant type of wastewater-based microplastic. Our findings confirm that wet wipes should also be considered as an important and relevant source of plastic fibres.

Major issues still remain regarding the commercialization of wet wipes and require urgent action. They relate to the absence of national and international regulations on wet wipes labelling/advertising regarding flushability and disposal, as well as the need for standard procedures to assess flushability from a wider perspective including environmental consequences.

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