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ATR-FTIR investigation of plastics in cosmetics, personal-care products and biodegradable materials

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Abstract
This paper presents the relevance of ATR-FTIR technique in the investigation of the presence of synthetic plastics in selected beauty and consumer goods. In the first step, we performed a vibrational analysis of different types of synthetic plastic consumer goods, i.e. PET, PP, PS, PE, HDPE and LDPE. Thus, we identified the vibrational assignments of each IR absorption band, as well as the quality of the plastic consumer goods samples. Secondly, we investigated, from point of view of the presence of plastics, some personal care products and cosmetics, i.e, a lipstick, a bio sunscreen emulsion for children, a sunscreen emulsion for adults, a toothpaste sample, a blush and five shampoo samples, using their ATR-FTIR spectra and the characteristic IR absorption bands of the analyzed synthetic plastics.

Keywords: ATR-FTIR; PE; PP; PET; plastics; personal care products; cosmetics

1. INTRODUCTION

Over the past decades, plastic pollution has become a major environmental problem. The different types of plastics that end up in water and soil are degraded over time by the action of the sun and wind, transforming into extremely small particles called microplastics. Also, microplastics are included in the composition of some personal care such as scrubs, body washes, toothpaste, lipstick etc., due to their abrasive, emollient, thickening and exfoliating properties [1–4]. It is important to mention that, due to very small sizes of microplastics, they can penetrate into living organisms and food items [5,6]. In addition, the persistence and accumulation of microplastics in water and soil constitutes a major challenge for environmental protection specialists who must find innovative methods to remedy this problem. Stopping this problem can be achieved through a good management of plastic waste based on very clear legislation regarding the environmental protection [7].

FTIR (Fourier Transform Infrared) spectroscopy can provide important information about the molecular structure of organic and inorganic components in a sample, being one of the most versatile analytical techniques for the chemical characterization of a sample [6, 8–11]. The ATR-FTIR spectroscopy, which uses an ATR (Attenuated Total Reflectance) accessory, is an efficient and non-destructive technique used for identifying of microplastics based on characteristic IR absorption bands to each type of synthetic polymer [3,11]. Consequently, the FTIR technique, together with an appropriate spectrum library or machine learning methods, can be used as an automated classification method of microplastics [12,13].

Firstly, in this paper we achieved a vibrational analysis of some plastic consumer goods such as PET (polyethylene terephthalate), PP (polypropylene), PS (polystyrene), PE (polyethylene), HDPE (high-density polyethylene) and LDPE (low-density polyethylene). Biodegradable plastics are good alternatives to synthetic plastics which have produced serious problems of waste pollution. For this reason, we analyzed the ATR-FTIR spectrum of a biodegradable bag in order to identify the kind of biodegradable plastic it belongs to.

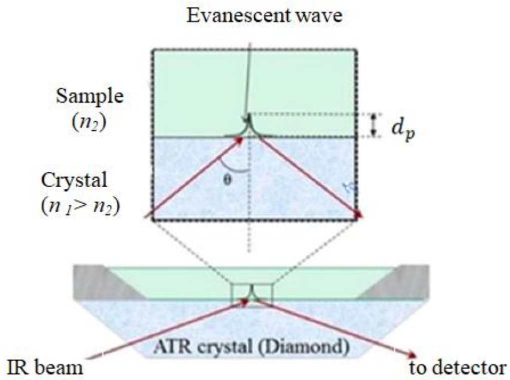
In the second part of the paper, research was conducted on the presence of synthetic plastics in some personal care products and cosmetics such as toothpaste, shampoo, lipstick, sunscreen emulsion and blush.

2. EXPERIMENTAL

The samples investigated in this paper are six synthetic polymer consumer goods representing the following types of plastic: PET, PP, PS, PE, HDPE and LDPE, and an unknown biodegradable bag sample. The plastic samples were examined in the solid phase without sample preparation using ATR-FTIR spectroscopic technique. Also, we recorded ATR-FTIR spectra for some personal-care products: a lipstick, a bio sunscreen emulsion for children, a sunscreen emulsion for adults, a toothpaste sample, a blush and five shampoo samples. The personal care samples analyzed are either in emulsion or powder form.

The ATR-FTIR technique provides structural information about the sample through the phenomenon of attenuated total reflection that occurs at the interface between the crystal (diamond) and the sample (Figure 1). At each point of incidence of IR radiation, an evanescent wave is generated through which the incident IR radiation is partially absorbed. Consequently, ATR-FTIR method is a superficial technique, the penetration depth of evanescent wave, d_p, being less than 5μm. In this case to obtain a very good quality of spectrum it is necessary to perform a very well contact between the ATR crystal and the sample. If the sample is liquid or emulsion, this contact is very well, but in the case of powder sample it is necessary to use a high-pressure clamp.

ATR-FTIR spectra of samples were recorded as a mean of 32 scans, at a resolution of 4 cm^{-1} in the 4000–400 cm^{-1} spectral domain. Before each individual analysis, the background spectrum was recorded. All spectra were obtained with a Bruker Tensor 27 FTIR spectrometer from the INPOLDE research center at Dunarea de Jos University of Galati, Romania.



$$d_p = \frac{\lambda}{2\pi\left(n_1^2 sin^2\theta - n_2^2\right)^{1/2}}$$

n_1 – refractive index of the sample
n_2 – refractive index of the diamond
λ – wavelength
θ – incidence angle

Fig. 1. The schematic principle of the ATR system

3. RESULTS AND DISCUSSION

3.1. Vibrational analysis of synthetic plastic consumer goods sample

In the figures 2, 3, 4 and 5 are presented the ATR-FTIR spectra of PET, PP, PS, PE, HDPE and LDPE plastic consumer goods samples. Using the literature [14–17], we performed a vibrational analysis of these spectra and the results of the analysis are included in the Tables 1 and 2. It is important to mention that, in order to identify the presence of syntetic plastic in personal care products and cosmetics, it is necessary to know very well the representative peaks of each type of synthetic plastic.

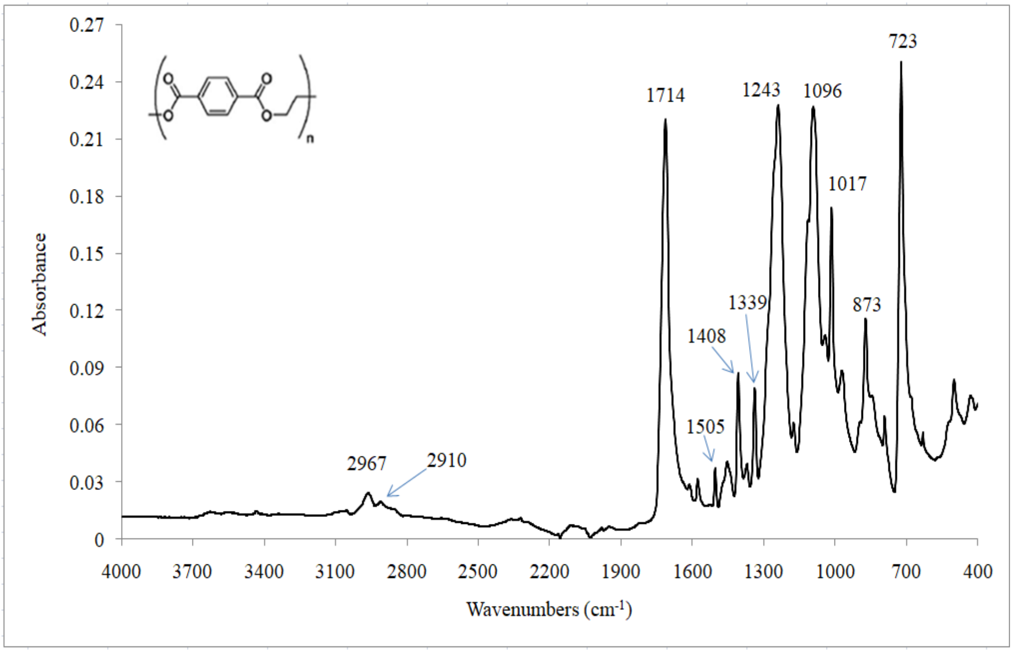


Fig. 2. ATR-FTIR spectrum of PET plastic consumer goods sample.

Analyzing the Tables 1 and 2, we can emphasize the fact that the IR absorption bands attributed to the asymmetric and symmetric stretching vibrations of aliphatic CH_2 chemical group are located in the (3000-2800) cm^{-1} spectral domain. The presence of the aromatic ring in the molecular structure of some plastics is highlighted by the following IR bands: the (3100-3000) cm^{-1} spectral range which is attributed to stretching vibrations of aromatic CH_2 chemical group, the (1600-1450) cm^{-1} spectral range associated with the stretching vibrations of C – C aromatic and the (850-690) cm^{-1} spectral range attributed to the out-of-plane bending vibration of aromatic CH_2.

The most intense and representative IR absorption bands of PET plastic sample (Figure 2) are located in the fingerprint region at the following wavenumbers: 1714 cm^{-1} associated with the C = O stretching vibrations, 1243 cm^{-1} associated with C – C(O) – O stretching vibrations, 1096 cm^{-1} associated with C – O stretching vibrations and 723 cm^{-1} associated with the aromatic CH_2 out-of-plane bending vibration.

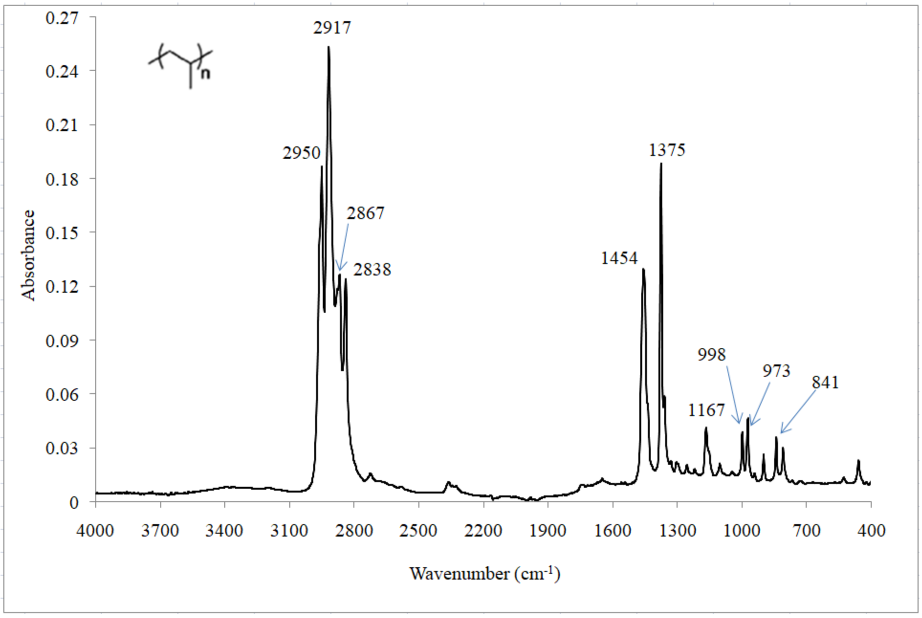


Fig. 3. ATR-FTIR spectrum of PP plastic consumer good sample.

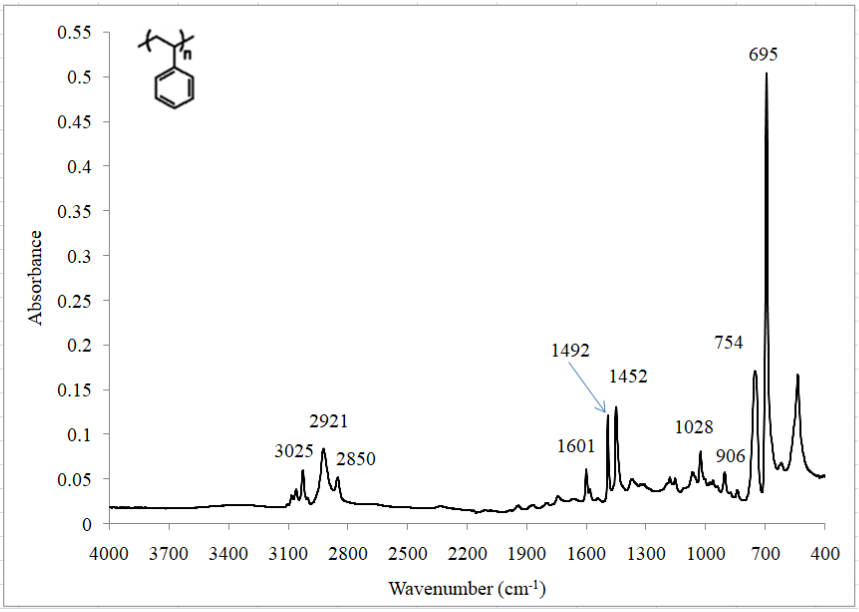


Fig. 4. ATR-FTIR spectrum of PS plastic consumer good sample.

The ATR-FTIR spectrum of PP plastic sample (Figure 3) has several IR bands with high intensity such as: the absorption bands (2950, 2917, 2867 and 2838 cm^{-1}) attributed to the asymmetric and symmetric stretching vibrations of aliphatic CH_2 and CH_3 groups and, the 1454 and 1375 cm^{-1} absorption peaks associated with in-plane bending vibrations of CH_2 and CH_3 chemical groups.

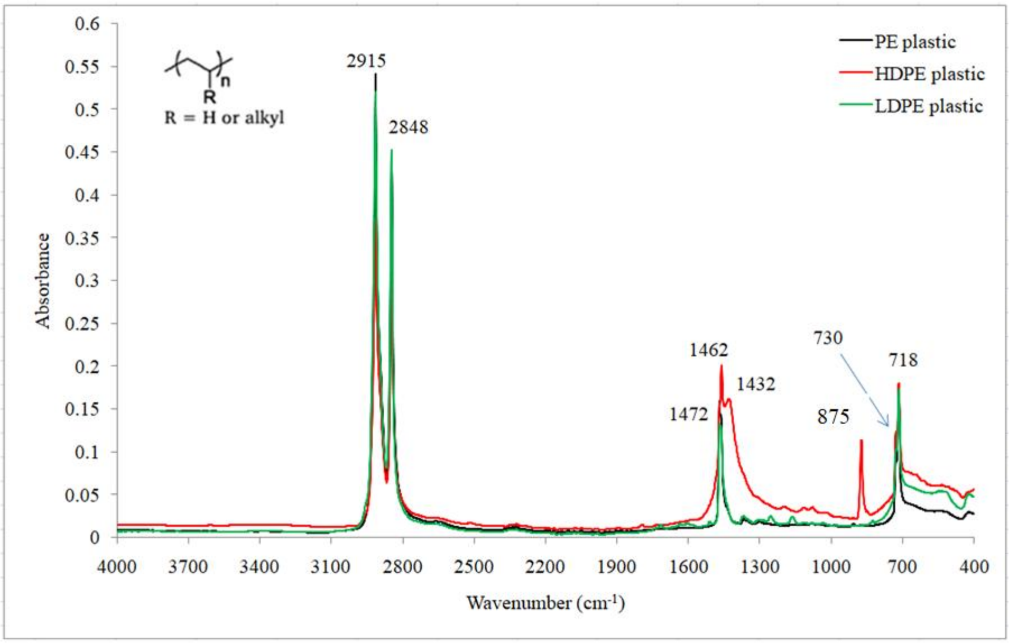
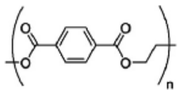
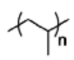
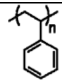


Fig. 5. ATR-FTIR spectra of PE, HDPE and LDPE plastic consumer goods samples.

Table 1. Experimental IR wavenumbers and their vibrational assignments for PET, PP and PS plastic consumer goods.

Polyethylene terephthalate PET		**Polypropylene PP**		**Polystyrene PS**	
					
Wavenumber (cm^{-1})	Assignment [14,16]	Wavenumber (cm^{-1})	Assignment [14,15,17]	Wavenumber (cm^{-1})	Assignment [14, 15]
2967	$\nu_{as}(CH_2)_{al}$	2950	$\nu_{as}(CH_3)_{al}$	3082	$\nu(CH)_{ar}$
2910	$\nu_{sym}(CH_2)_{al}$	2918	$\nu_{as}(CH_2)_{al}$	3060	$\nu(CH)_{ar}$
1714	$\nu(C = O)$	2888	$\nu_{sym}(CH_3)_{al}$	3025	$\nu(CH)_{ar}$
1578	$\nu(C–C)_{ar}$	2838	$\nu_{sym}(CH_2)_{al}$	2921	$\nu_{as}(CH_2)_{al}$
1505	$\nu(C–C)_{ar}$	1454	$\beta(CH_2)$	2850	$\nu_{sym}(CH_2)_{al}$
1408	$\beta(CH)_{al}$	1375	$\beta(CH_3)$	1601	$\nu(C–C)_{ar}$
1339	$\beta(CH)_{al}$	1167	$\beta(CH_3)+ \nu(C–C)$	1583	$\nu(C–C)_{ar}$
1243	$\nu(C–C(O)–O)$	998	$\beta(CH_3)+ \gamma(–CH_2–)$	1492	$\nu(C–C)_{ar}$
1096	$\nu(–O–C–)$	973	$\beta(CH_3)+ \nu(C–C)$	1452	$\nu(C–C)_{ar}$
1017	$\beta(CH)_{ar}$	898	$\beta(–CH_2–)$	1028	$\beta(=CH)$
873	$\gamma(CH)_{ar}$	841	$\beta(–CH_2–)$	906	$\gamma(=CH)$
723	$\gamma(CH)_{ar}$	809	$\nu(C–C)_{backbone}$	695	$\gamma(CH)_{ar}$

Legend: *ν* – stretching vibration, *β* – in-plane bending vibration, *γ* – out-of-plane bending vibration, *as/sym* – asymmetric/symmetric, *ar* – aromatic, *al* – aliphatic.

In the case of ATR-FTIR spectrum of PS plastic sample (Figure 4), it is only one absorption peak with very high intensity at 695 cm^{-1} attributed to the aromatic CH out-of-plane bending vibrations.

The ATR-FTIR spectra of PE, HDPE and LDPE plastic samples (Figure 5) are very simple and contain four peaks (2915, 2848, 1462 and 718 cm^{-1}, Table 2) except for the HDPE plastic which has an additional peak at 875 cm^{-1}, as well as a double degenerate absorption band at 1462 cm^{-1}.

Table 2. Experimental IR wavenumbers and their vibrational assignments for PE, HDPE and LDPE plastic consumer goods.

Polyethylene PE		**High-density polyethylene HDPE**		**Low-density polyethylene LDPE**	
Wavenumber (cm^{-1})	Assignment [14,15]	Wavenumber (cm^{-1})	Assignment [14,15]	Wavenumber (cm^{-1})	Assignment [14,15]
2915	$\nu_{as}(CH_2)$	2915	$\nu_{as}(CH_2)$	2915	$\nu_{as}(CH_2)$
2848	$\nu_{sym}(CH_2)$	2848	$\nu_{sym}(CH_2)$	2848	$\nu_{sym}(CH_2)$
1472	$\beta(CH)$	1462, 1432	$\beta(CH)$	1472	$\beta(CH)$
719	$\beta(CH_2)n \geq 6$	730, 718	$\beta(CH_2)n \geq 6$	718	$\beta(CH_2)n \geq 6$

Legend: *ν* – stretching vibration, *β* – in-plane bending vibration, *as/sym* – asymmetric/symmetric.

The conclusion we can draw after analyzing the ATR-FTIR spectra is that the quality of the plastic consumer goods samples is very similar to standard plastics found in the literature [14–17].

3.2. Vibrational analysis of a biodegradable bag sample

In the figure 6 we present the ATR-FTIR spectrum of a biodegradable bag and in the Table 3 we included the assignments of absorption IR bands obtained from the vibrational analysis.

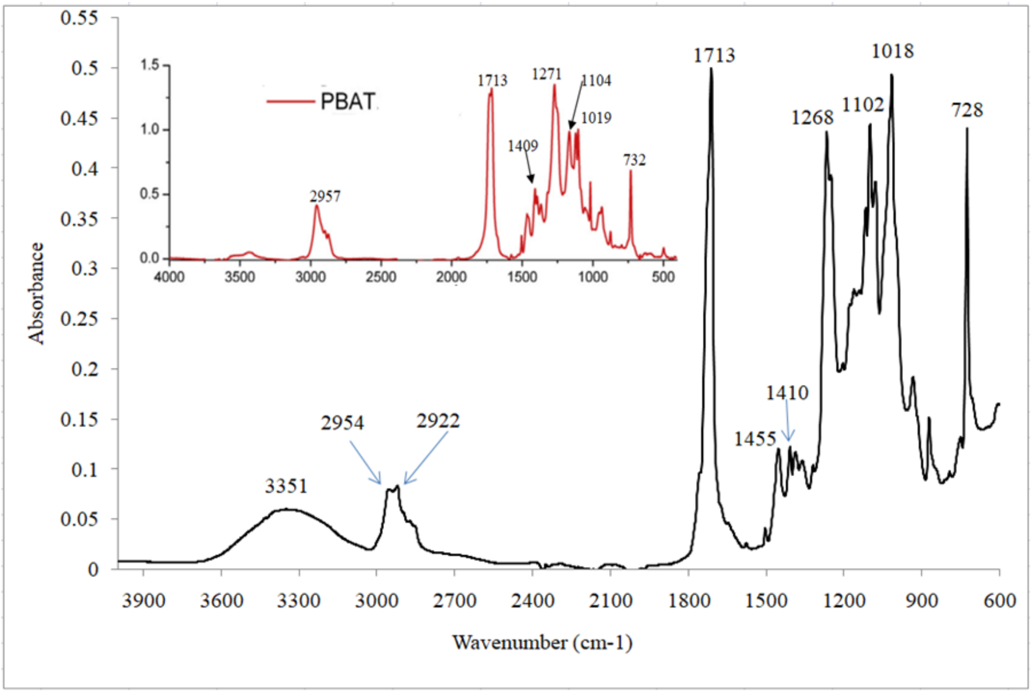


Fig. 6. ATR-FTIR spectrum of a biodegradable bag. The superposed IR spectrum is for PBAT biodegradable plastic [18].

By comparing the ATR-FTIR spectrum of biodegradable bag (unknown type) with that of the PBAT polymer [18] it was found that both spectra are very similarly. Consequently, our unknown sample is the poly(butylenes-adipate-co-terephthalate), a synthetic polymer with high flexibility, very well impact strength, melt processibility and fully biodegradability [19–21]. Due to these properties, the PBAT polymer is used more and more in everyday life.

Table 3. Experimental IR wavenumbers and their assignments for the biodegradable bag.

Wavenumber (cm^{-1})	Assignment [18, 20]	Wavenumber (cm^{-1})	Assignment [18,19]	Wavenumber (cm^{-1})	Assignment [18–20]
3351	ν(OH)	1578	$\nu(C–C)_{ar}$	1268	ν(C–C(O)–O)
2953	$\nu_{as}(CH_3)_{al}$	1505	$\nu(C–C)_{ar}$	1102	ν(C-O)
2923	$\nu_{as}(CH_2)_{al}$	1455	$\beta(CH_2)$	1019	$\beta(CH)_{ar}$
2874	$\nu_{sym}(CH_3)$	1410	$\beta(CH_2)$	873	$\gamma(CH)_{ar}$
1714	ν(C=O)	1364	$\gamma(CH_2)$	728	$\gamma(CH_2)_4$

Legend: *ν* – stretching vibration, *β* – in-plane bending vibration, *γ* – out-of-plane bending vibration, *as/sym* – asymmetric/symmetric, *ar* – aromatic, *al* – aliphatic.

3.3. Identification of plastics in personal care products and cosmetics using the ATR-FTIR spectra

Common ingredients that make up lipsticks are: waxes, oils, colouring agents and chemical preservatives. The basic composition, about (40 – 70) % consists of oils, *e.g.* castor oil, lanolin oil and mineral oil, followed by the vaxes in percentages about (8 – 15)%, *e.g.* carnauba wax, candelilla wax, bees wax and paraffin wax, and (0.5 – 8)% pigments such as rhodamine, erythrosine, titanium dioxide and tartrazine, and, finally, additives like antioxidants, preservatives, emollients and perfumes in very small quantities [22,23]. The natural and synthetic waxes are an important component of lipstick production. Among the synthetic waxes, we are mentioning the presence of polyethylene [24] as an ingredient which contributes to the structure and smooth spreadability of lipstick.

In the figure 7, which shows the ATR-FTIR spectra of a lipstick and PE sample, we can notice that the characteristic absorption IR peaks of PE, *i.e.* 2918, 2850, 1462 and 720 cm^{-1}, have a correspondent in the spectrum of lipstick. Thus, we can conclude that in the composition of investigated lipstick sample it found PE polymer.

Generally, the sunscreen composition consists of (40–65) % water, one or more known ultraviolet absorbing agents, polyethylene homopolymer having a low molecular weight and a low density or a copolymer (*e.g.* styrene/acrylates copolymer, acrylates copolymer, acrylates/C10-30 alkyl acrylate crosspolymer) and vinyl acetate, emollients, emulsifiers, preservatives, antioxidants, fragrances and coloring agents [25,26].

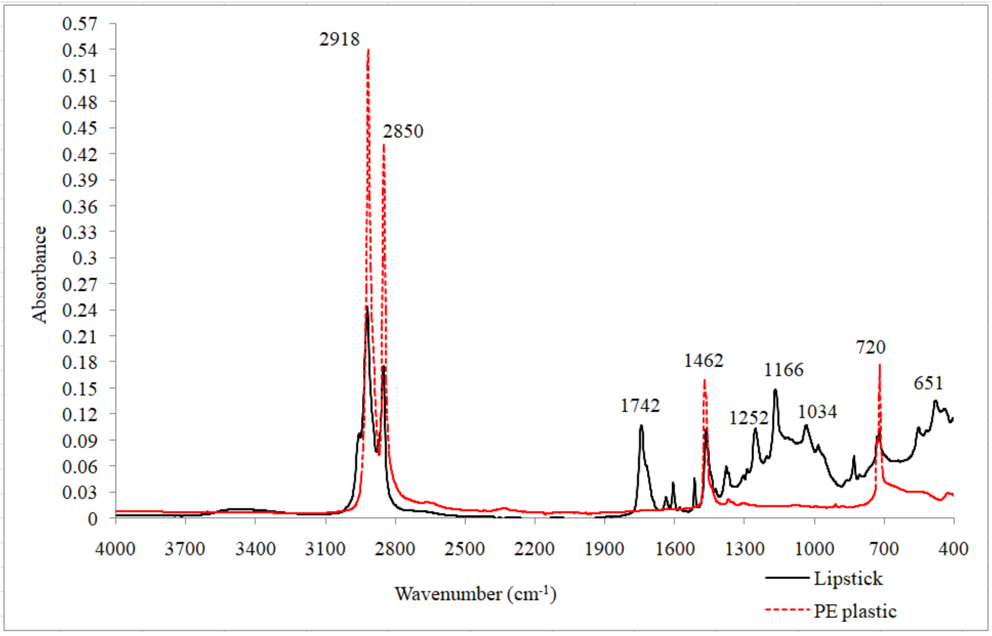


Fig. 7. ATR-FTIR spectra of a lipstick sample and PE plastic consumer goods.

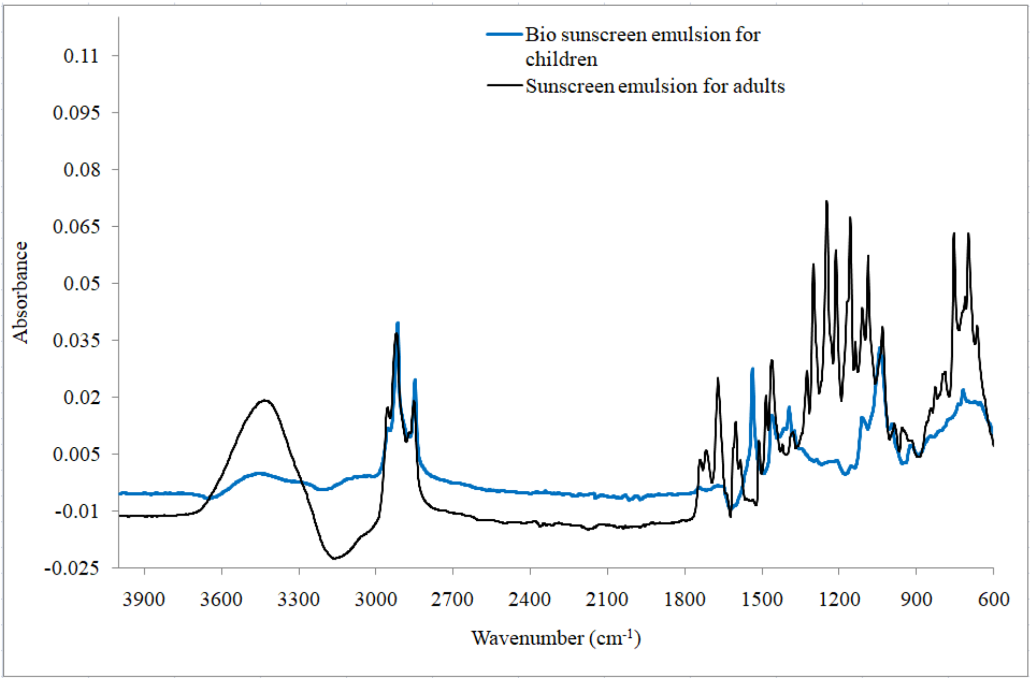


Fig. 8. ATR-FTIR spectra of the bio sunscreen emulsion for children and the sunscreen emulsion for adults.

As we stated above, the sunscreen emulsion samples, *i.e.* bio sunscreen for children and sunscreen for adults, contain a significant percentage of water, for this reason we substracted the water spectrum from the samples' spectra (Figure 8) in order to distinguish clearly the IR absorption bands.

At the first analysis of the spectra of the two emulsions (Figure 8), we have observed that the ATR-FTIR spectrum of the sunscreen for adults contains much more IR bands than that of the bio sunscreen for children. This fact leads to the idea that the sunscreen for adults has a much more complex composition than the children's sunscreen emulsion. Thus, we can emphasize that the ATR-FTIR technique confirms the known fact that the sunscreen emulsions from the *bio* category have a much simpler composition.

Regarding the presence of synthetic polymers in the sunscreen emulsion for adults, we have performed the superposition of ATR-FTIR spectrum of emulsion with that of PE sample (Figure 9) and with that of PS sample, respectively (Figure 10). In this situation, it is very visible the fact that the characteristic IR bands i.e. 2918, 2850, 1462 and 718 cm^{-1}, of the PE sample (Figure 9) are present in ATR-FTIR spectrum of the sunscreen emulsion for adults. Also, in the case of comparing the ATR-FTIR spectrum of the sunscreen emulsion with that of the PS sample (Figure 10), we remarked that it is a very well correspondence between IR peaks i.e. 2918, 2850, 1601, 1492, 1452, 1028, 754 and 695 cm^{-1}, of PS and those of sunscreen sample. Finally, we can conclude that in the sunscreen emulsion for adults it was identified the presence of PE and PS plastics.

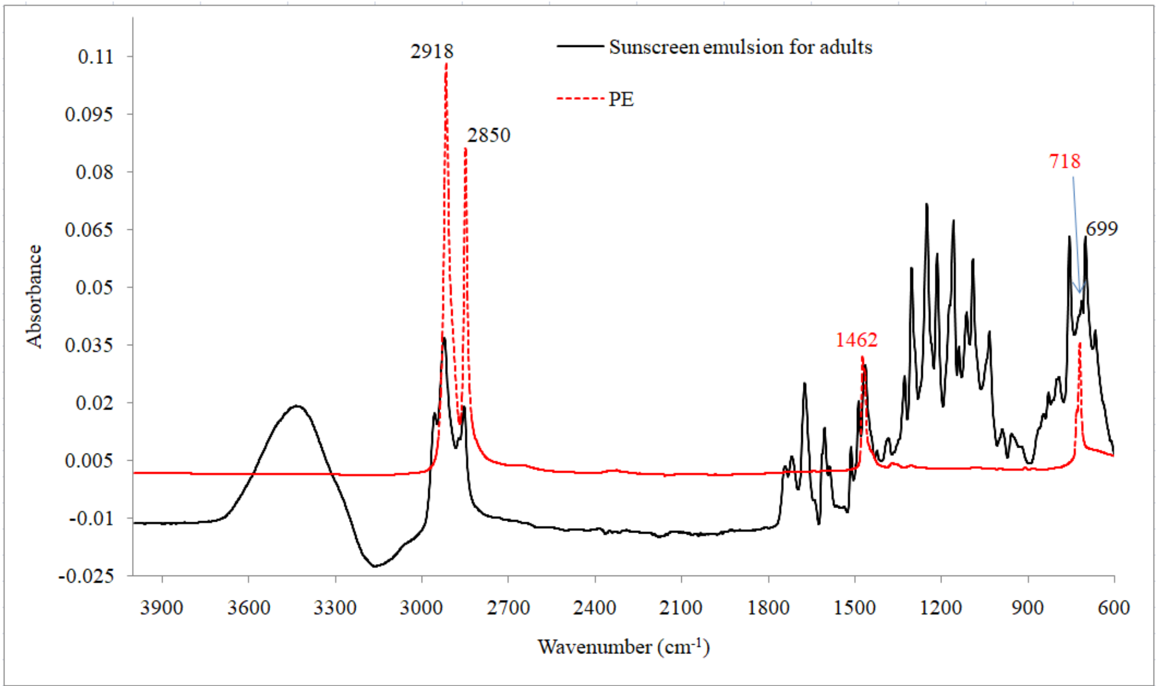


Fig. 9. ATR-FTIR spectra of the sunscreen emulsion for adults and PE sample.

In the case of toothpaste sample, it is known that its components are the following: water (20-40)%, abrasive agents (about 50%), calcium carbonate, silicates, hydroxyapatite, detergents, etc. The ATR-FTIR spectrum from Figure 11 represents the result obtained after subtracting the water spectrum from initial spectrum of toothpaste sample. Thus, we can see on spectrum the IR absorption bands attributed to the silicates at 1072 cm^{-1} and 448 cm^{-1}, calcium carbonate at 1403 cm^{-1} and 889 cm^{-1}, hydroxyapatite at 1072 cm^{-1} and 1038 cm^{-1} and, detergents at 2921 cm^{-1} [10,27]. Although toothpastes, generally, contain plastics, we did not identify their presence in our toothpaste sample.

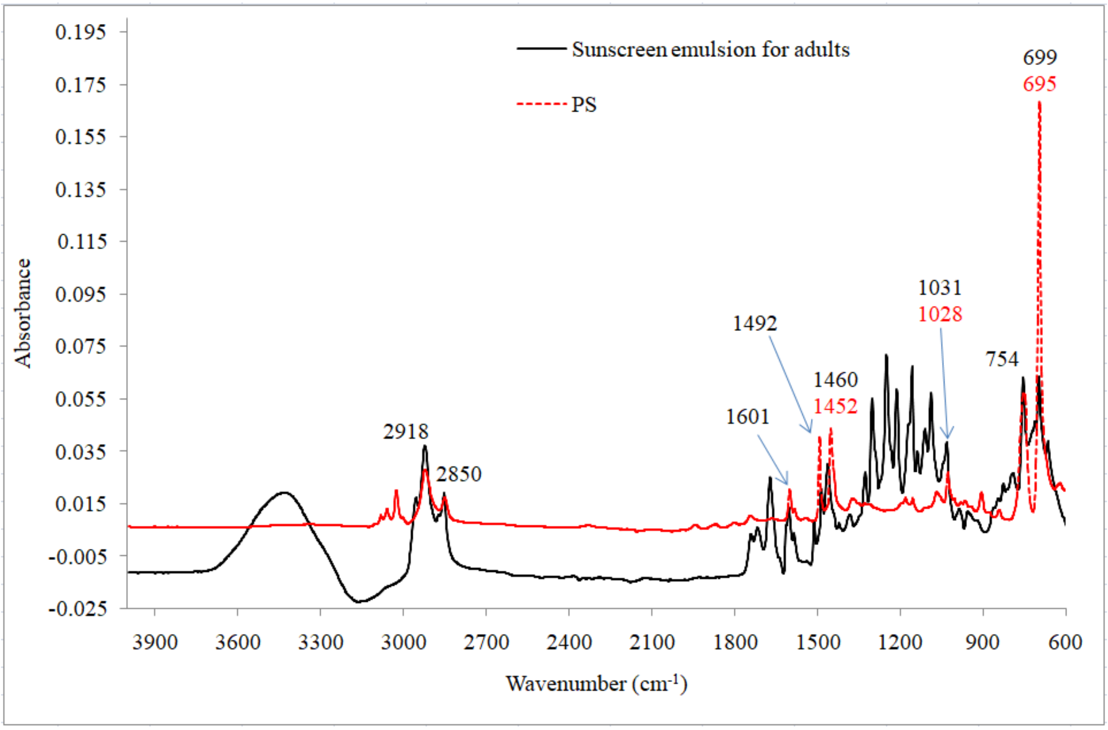


Fig. 10. ATR-FTIR spectra of the sunscreen emulsion for adults and PS sample.

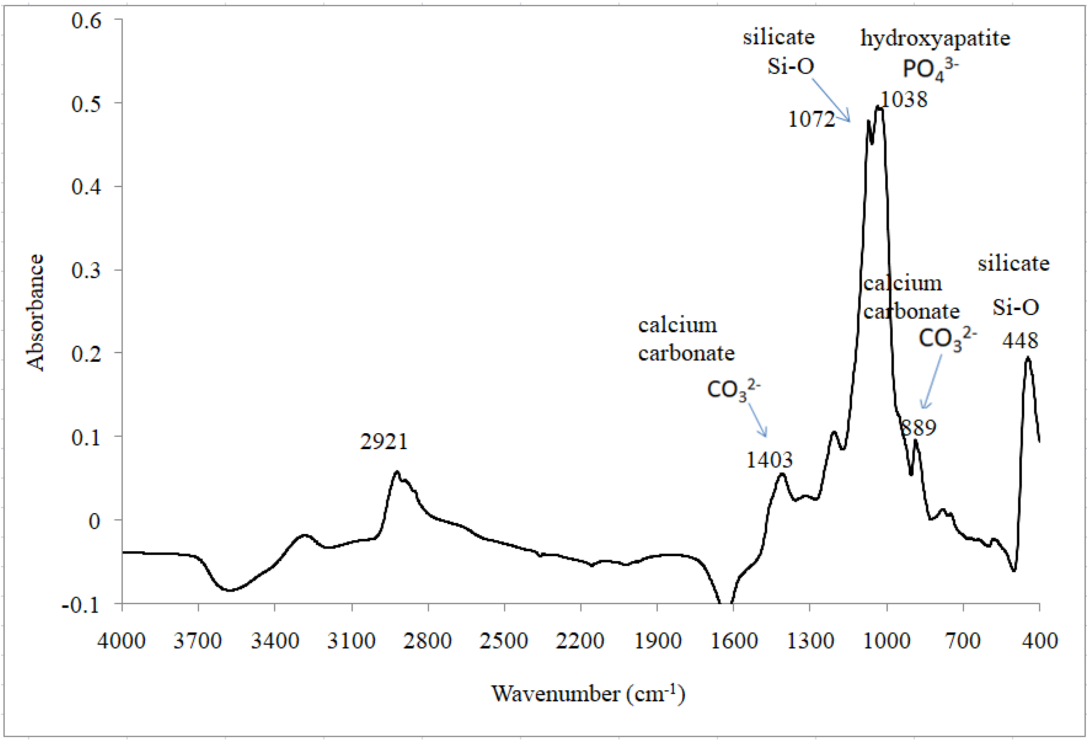


Fig. 11. The ATR-FTIR spectrum of a toothpaste sample.

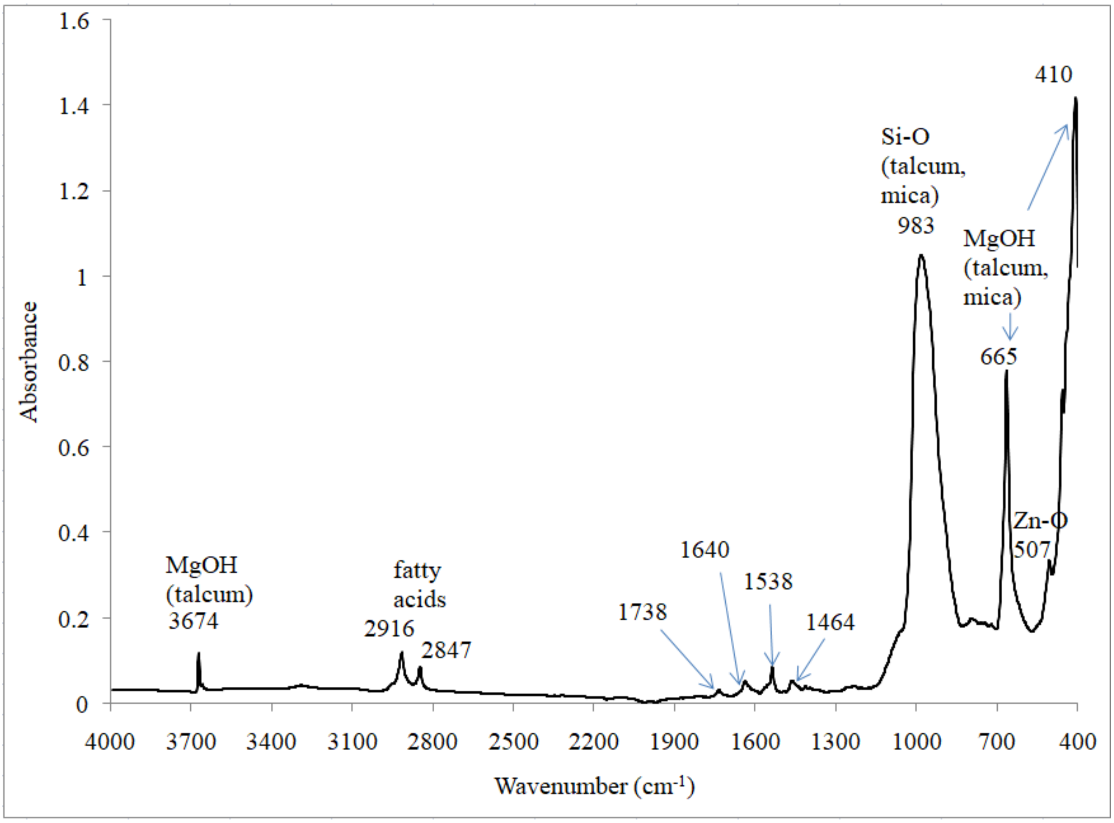


Fig. 12. The ATR-FTIR spectrum of a blush sample

Generally, the blush consists of talc, stearic acid, a natural fatty acid, mineral pigments such as mica, zinc oxide and titanium dioxide. The presence of these components [28,29] in blush is shown on the ATR-FTIR spectrum of blush (Figure 12). Regarding the presence of plastics in the blush, we identified three (out of four) very low intensity IR absorption peaks (2916, 2847 and 1464 cm^{-1}) which are characteristic to PE plastic. This fact does not surely confirm the presence of PE because these peaks also appear in the spectrum of fatty acids which exist in the blush composition.

PEGs are polyethylene glycols widely used in the personal-care products and cosmetics as emulsifiers, humectants, surfactants, cleaning agents and skin conditioners [30]. The PEG-45M, which contains 45000 units of ethylene glycol, is a synthetic ingredient of shampoos used as a stabilizer and thickener [31].

The ATR-FTIR spectra of all the shampoo samples (obtained by subtracting the water spectrum) are presented in the figure 13. We can notice that the spectra have a high grade of similarity as regards the position of IR absorption bands, but their intensity is different. Therefore, we can state that their composition is very similar, but the components are included in different percentages.

By overlapping the spectrum of PE plastic with the spectra of the shampoo samples, it is observed that the characteristic IR absorption bands of PE are present in the spectra of shampoos (2923, 2854, 1463 and 717 cm^{-1}). Consequently, our shampoo samples contain PE plastics. Also, the presence of IR absorption band around 3466 cm^{-1} is attributed to the PEG polymer [29].

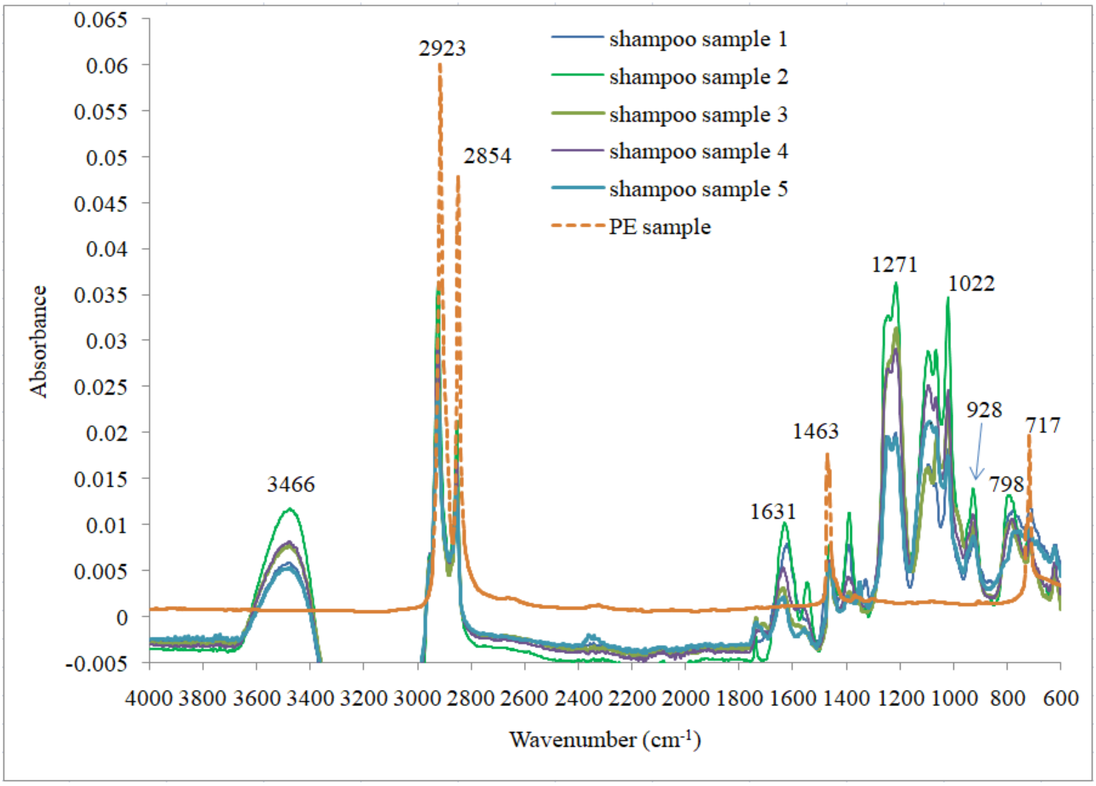


Fig. 13. ATR-FTIR spectra of shampoo samples and PE plastic consumer goods.

4. CONCLUSIONS

Due to the widespread presence of plastics fragments released in the environment and their associated pollution hazard, it is very important to employ appropriate investigation techniques for their identification in products destined to consumers' use and assessment of the health risk due to human microplastic exposure.

In the present paper we investigated the presence of plastics in personal-care products and cosmetics using the ATR-FTIR technique. First, we performed research on ATR-FTIR spectra of some synthetic polymers (PET, PP, PS, PE, HDPE and LDPE) in order to identify the characteristic IR absorption peaks of each type of polymer, as well as their vibrational assignments. Based on this information, we have further identified the presence of synthetic polymers in ATR-FTIR spectra of the biodegradable bag, personal-care products and cosmetic samples. Thus, in the lipstick sample, the sunscreen emulsion for adults and shampoo samples, we found PE plastics. In addition, we identified the presence of PEG polymer in the shampoo samples and the PS polymer in the case of the sunscreen emulsion for adults.

ATR-FTIR spectroscopic technique proved to be a useful tool for the identification of the polymer constituents in cosmetics and personal-care products. This research will be continued by applying other adequate techniques such as micro-FTIR and Scanning Electron Microscopy (SEM) which may add supplementary information on this work.

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