Greenpeace Research Laboratories Analytical Results 2016-05

Analysis of a range of consumer products purchased in Korea and in the UK for the presence of plastic microbeads

September 2016

Introduction

A total of 25 consumer product samples purchased either in Korea or in the UK, including personal care products, cosmetics and household cleaning products, were received by the Greenpeace Research Laboratories in their original packaging between March and June 2016. A full list of the products analysed, along with the laboratory codes assigned to them, is given in Table 1 below. One sample, of wet-wipes (initially assigned laboratory code MB16023), was not taken further forwards for analysis.

Materials & methods

In all cases, a sample of known weight (between 0.5 and 28 grammes, depending on the nature of the product and its ease of filtration) was taken, mixed with 1 litre of deionised water heated to boiling point and filtered under vacuum through a pre-weighed Whatman Grade 4 filter paper (55 mm diameter), a method adapted from Napper *et al*. (2015). Additional hot water was used where necessary in order to flush the soluble components of the samples through the filter and retain only the non-water soluble components on the surface of the filter paper. In some cases, less than a gramme of sample could be filtered effectively in this way before the filter blocked; this inevitably limited the subsequent analysis of any solid residues by FTIR for those samples.

All filtered samples were placed in petri dishes and dried at room temperature over a period of several days to achieve a constant weight. They were then transported to **an independent research laboratory for separation and analysis of the solid residues on the filters (where they could be found) using Fourier-Transformed Infra-Red (FTIR) spectroscopy**, to determine which of the products contained plastic microbeads and, as far as possible, to confirm the identity of the plastics used.

Each sample was observed under a dissecting microscope with visual description taken for each product. Different colour forms were determined. Each colour form was then separated by hand using fine tweezers and analysed by FTIR spectroscopy using an Agilent Cary 630 FTIR spectrometer.

Greenpeace Research Laboratories
School of Biosciences
Innovation Centre Phase 2
Rennes Drive
University of Exeter
Exeter EX4 4RN, UK

This instrument detects absorbance and reflectance of wavelengths within the infra-red region of the light spectrum. Different material will absorb different wavelengths at different intensities. The resulting spectra were then compared against commercially available libraries of FTIR spectra for different plastics, other polymers and certain other types of common solid materials, using the Agilent MicroLab PC software, through a combination of automated library search algorithms and expert analysis and cross-checking of match qualities.

Results

Of the 24 consumer products received and prepared for analysis, 22 could be filtered in sufficient quantities to leave a detectable solid residue on the surface of the filter papers. Both the Gatsby branded hair wax (MB16021) and the Bullsone branded car compound (MB16022) were not sufficiently miscible with boiling water to enable effective filtration of sufficient quantities to obtain a solid residue on the filters within the time constraints of the study.

In the other 22 cases, sufficient solid material was retained by the filter paper to enable FTIR analysis. In some cases, only one homogenous material type could be distinguished. In others, two or more distinct types of solid could be distinguished and separated by hand to allow FTIR analysis of each material type.

Results are summarised in Table 1. In cases in which the identity of the material could be determined to a high level of confidence, the results indicate the closest material match from the library spectra, along with a percentage match score. For those cases for which the confidence in automated matching was lower, the spectra were compared manually against all possible library spectra in order to try to determine a likely identity for the material. In these cases, the possible identity of the material is recorded more tentatively, and with no percentage match quality recorded.

Of the 22 samples that could be analysed by FTIR:-

1. **the presence of polyethylene microbeads was confirmed in 6 samples, 3 from Korea**: MISSHA Essential Cream Scrub (MB16003), MISSHA Cacao & Cream Scrub (MB16004) and Eau Thermale Avene Gentle Purifying Scrub (MB16005) and **3 from the UK**: Arm & Hammer Truly Radiant toothpaste (MB16008), Clearasil Ultra Rapid Action scrub (MB16009) and Clean & Clear Exfoliating Daily Wash (MB16010).
2. **in a further 4 samples, results show the likely (high probability) presence of plastic particles:** polyethylene in the Himalaya neem purifying scrub (MB16006) and in The Real Shaving Company Multi-task Super 8 balm (MB16024), polypropylene in the Innisfree green tea pure body gel scrub (MB16013) and one of a number of other plastics (PET, PBT or PU) in the Domax kitchen ceramic cleaner (MB16018).
3. in addition, **three other samples showed evidence for the presence of plastic polymers or elastomers:** a paraffin wax or PE wax in the Face Shop Rice Water Bright all-in-one cleanser (MB16014), a bisphenol A-derived polymer in the Lovely me:ex Mango Seed lip scrub (MB16015) and polysulphide elastomer fibres in the Homestar Power scrub bathroom cleaner (MB16019).

No evidence for the presence of plastics could be found in the filtered samples for any of the other products, including the three other toothpastes analysed (MB16001, 2 & 7), three body washes (MB16011, 12 & 17), a hair shampoo (MB16016), a washing powder (MB16020) and a skin cream (MB16025).

The more detailed results provided by the independent analytical laboratory, including microscopic photographs of the filtered material, FTIR spectra for each of the distinct materials that could be separated and the results of the comparison with spectral libraries, are included in Appendix 1.

Note that this type of FTIR analysis is qualitative only, i.e. it can help identify the types of plastics or other materials present in the solids retained on the filters from each product, but cannot determine the absolute or relatively quantities of each material present in the original product. Such quantification would require quantitative and complete separation of all the different solid materials present, followed by separate determination of dry mass and/or comprehensive counts of each solid particle type. Such quantitative determinations were beyond the scope of the current study, which was designed simply to identify which of those products analysed contained plastic microbeads.

For more information please contact:

David Santillo or Kevin Brigden

References:

Napper, I.E., Bakir, A., Rowland, S.J. & Thompson, R.C. (2015) Characterisation, quantity and sorptive properties of microplastics extracted from cosmetics. *Marine Pollution Bulletin* 99: 178-185 http://dx.doi.org/10.1016/j.marpolbul.2015.07.029

Table 1: list of consumer products received and analysed, along with a summary of the qualitative (material identification) results obtained for each sample

Laboratory sample code	Product description	Summary of qualitative results	Plastic microbeads confirmed?
MB16001 (Korea)	LG HH - toothpaste - Perioe White Now (Red & white pack)	White caked sample with spherical particles (possibly silica) – no evidence of plastics	NO
MB16002 (Korea)	Aekyoung - toothpaste - 2080 New Shining White (white & blue pack)	White caked sample with no clearly identifiable particles – no evidence of plastics	NO
MB16003 (Korea)	MISSHA Essential Cream Scrub	Combination of white and transparent particles, along with yellow shards. Yellow and transparent material probably silica – **white particles confirmed as polyethylene, PE (96% match quality)**	YES
MB16004 (Korea)	MISSHA Cacao & Cream Scrub	Combination of white and black particles – **white particles confirmed as polyethylene, PE (80% match quality)** – black particles show characteristics similar to ethylene propylene plastic, but identify could not be confirmed	YES
MB16005 (Korea)	Eau Thermale Avene Gentle Purifying Scrub	Combination of fragile red spherical particles and smaller, harder white particles – **white particles confirmed as polyethylene, PE (96% match quality)** – red particles appear to be polyethylene also, but possibly with additional polymer components or additives	YES
MB16006 (Korea)	Himalaya - Neem scrub - purifying Scrub	Combination of irregular green-pigmented transparent particles and brown angular particles – **green-pigmented particles appear to be polyethylene, PE (79% match quality)** – brown particles identified as a natural organic material	LIKELY
MB16007 (UK)	Colgate Max White One toothpaste	White caked sample with blue irregular fragments – white material shows closest match to ethylcellulose (90% match quality) – blue fragments appears to be silica	NO

Table 1 (continued): list of consumer products received and analysed, along with a summary of the qualitative (material identification) results obtained for each sample

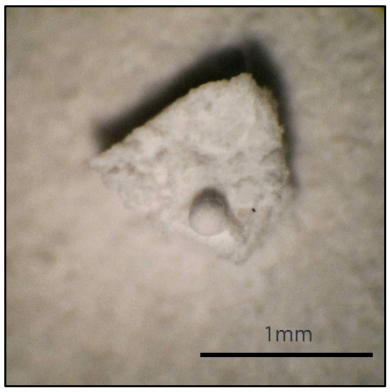
MB16008 (UK)	Arm & Hammer Truly Radiant toothpaste	White caked sample with fragile blue fragments – white material possibly fluorosilicone or ethyl cellulose (identity unclear) – **blue fragments confirmed as polyethylene, PE (96% match quality)**	YES
MB16009 (UK)	Clearasil Ultra Rapid Action scrub	Small transparent particles with larger blue spheres – **white particles confirmed as polyethylene, PE (95% match quality) – blue spheres also confirmed as polyethylene, PE (92%)**	YES
MB16010 (UK)	Clean & Clear Exfoliating Daily Wash	White particles – **confirmed as polyethylene, PE (84% match quality)**	YES
MB16011 (UK)	Simple Kind to Skin Smoothing Facial Scrub	Smaller white fragment particles with larger off white particles – white particles possibly silica – off-white particles identified as wood, but probably another natural organic material	NO
MB16012 (UK)	The Body Shop Tea Tree Squeaky Clean Scrub	White and transparent fragmented particles – white particles confirmed as cellulose – transparent particles appear to be silica	NO
MB16013 (Korea)	Innisfree green tea pure body gel scrub	Combination of irregular white particles and larger purple fragments – white particles identified as cellulose triacetate (88% math quality) – **high probability that purple fragments are synthetic plastic, possibly polypropylene (PP) or similar**	LIKELY
MB16014 (Korea)	The Face Shop Rice Water Bright all-in-one cleanser	Soft brown spherical particles of widely varying sizes – **possibly a mix of paraffin wax with polyvinyl acetate (88% match quality), though also showing many characteristics of polyethylene, PE**	POSSIBLY
MB16015 (Korea)	Lovely me:ex Mango Seed lip scrub	Large, sticky sand-grain sized light brown particles – **possible identification as bisphenol A-derived polymer along with polypropylene glycol additive**	POSSIBLY

Table 1 (continued): list of consumer products received and analysed, along with a summary of the qualitative (material identification) results obtained for each sample

MB16016 (Korea)	Amorepacific - Ryo - Hair shampoo, scalp deep cleansing shampoo (acrylate copolymer)	Relatively large, soft, spherical green-pigmented particles – possibly cellulose or cellulose-based material (95% match quality)	NO
MB16017 (Korea)	Unilever - St. Ives - exfoliating body wash, smoothing apricot (acrylate copolymer)	Combination of transparent, light brown and black irregular particles – possibly silica/sand	NO
MB16018 (Korea)	Global cosmed internation - Domax - kitchen ceramic cleaner ('an abrasive' included)	Irregular transparent fragments – **identified as plastic, but possible identities include polyethylene terephthalate (PET), polybutylene terephthalate (PBT) or polyurethane (PU) (all matched at 76-78%)**	LIKELY
MB16019 (Korea)	LG HH - Bathroom cleaner - Homestar Power scrub for bathroom cleaning ('an abrasive' included)	White caked sample with embedded fibres – **best match for fibres is polysulphide elastomer, but not confirmed**	POSSIBLY
MB16020 (Korea)	CJ Lion - washing powder - BEAT – (green or blue beads)	White caked sample – identified as aluminosilicate	NO
MB16021 (Korea)	*Gatsby - Hair wax - Styling wax, ultra hard type - Polyethylene , polysorbate60*	*Small white fragments – insufficient material to conduct FTIR analysis*	*UNKNOWN*
MB16022 (Korea)	*Bullsone - Swirl remover, Car compound*	*No identifiable solids on filter paper – unable to conduct FTIR analysis*	*UNKNOWN*
MB16024 (UK)	The Real Shaving Company Multi-task Super 8 balm	Small number of small white particles – automated searches give low match quality **but appears to be polyethylene, PE (with some additional peaks that could be related to additives)**	LIKELY
MB16025 (UK)	L'Oreal Lancombe Hydra zen BB cream	Brown clay like caked material, with no hard microbeads/fragments found – spectral matching inconclusive	UNKNOWN

Appendix 1: detailed results by sample

MB16001:



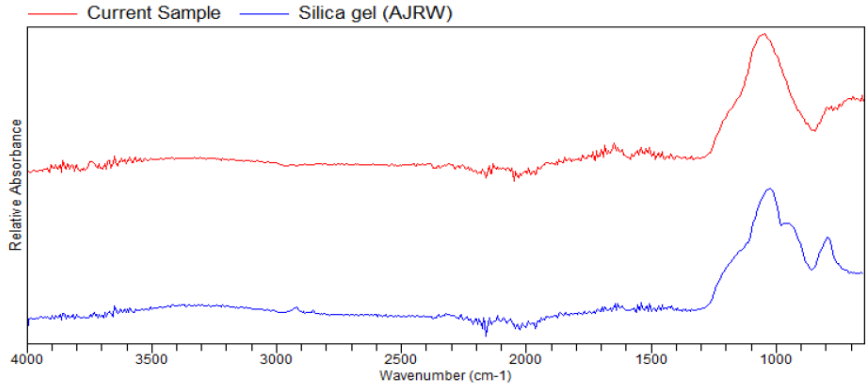
LG HH - toothpaste - Perioe White Now (Red and white package)

Description:

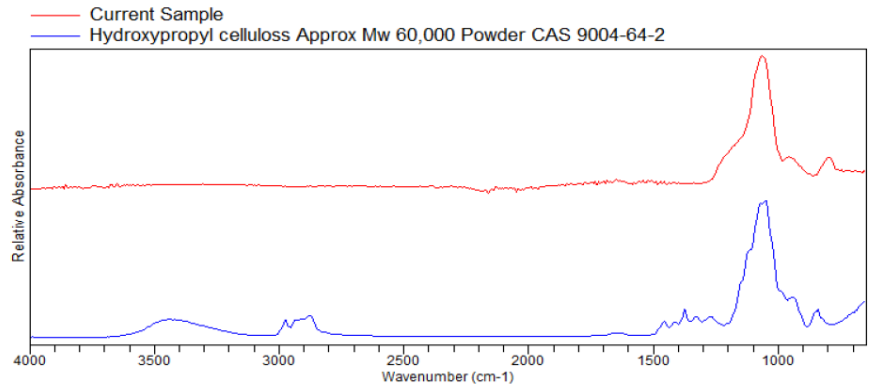
White caked sample with spherical particles

FTIR:

White Balls: Silica (94.47%)



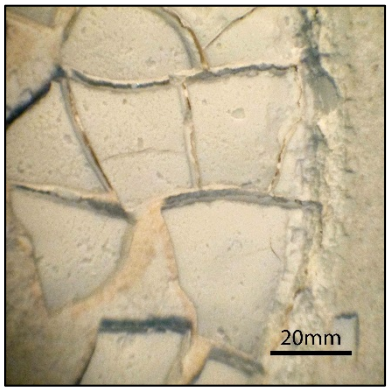
White cake: Possible Cellulose derivative Hydroxypropyl cellulose (89.72%) an emulsion stabiliser



Confidence in result

No evidence of plastic present

MB16002:



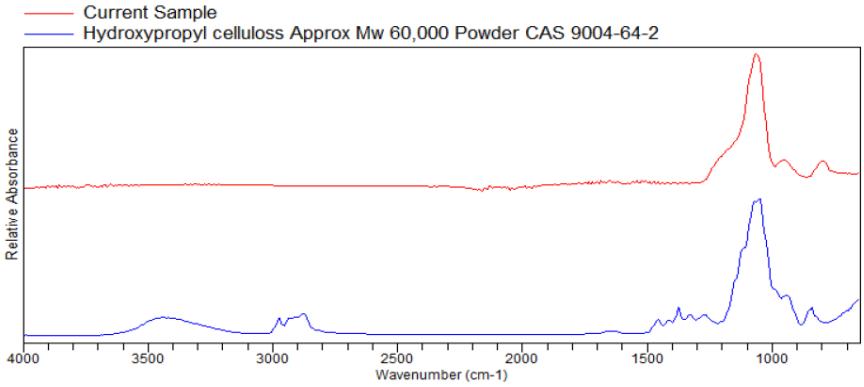
Aekyoung - toothpaste - 2080 New Shining White (white and blue package)

Description:

White caked sample with no clear particles

FTIR:

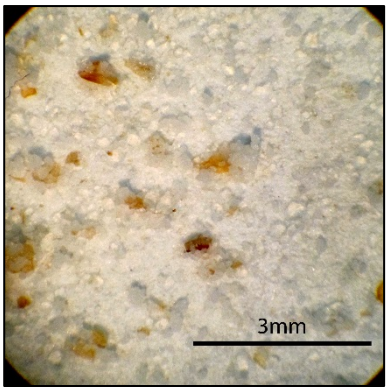
White 'cake': Possible Cellulose derivative Hydroxypropyl cellulose (88.67%) an emulsion stabiliser



Confidence in result

No evidence of plastic present

MB16003:



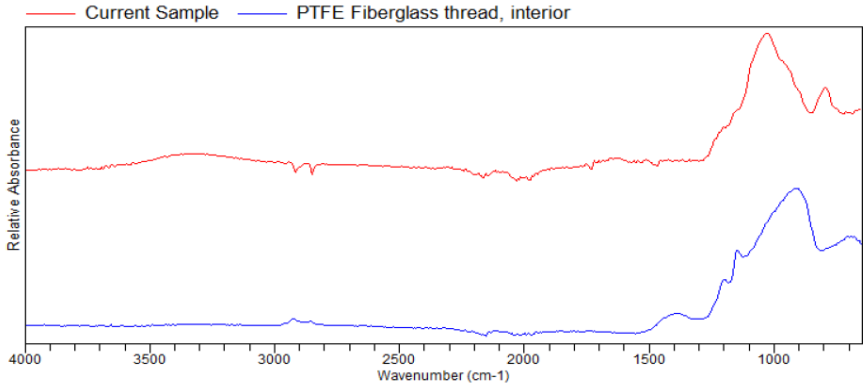
MISSHA Essential Cream Scrub

Description:

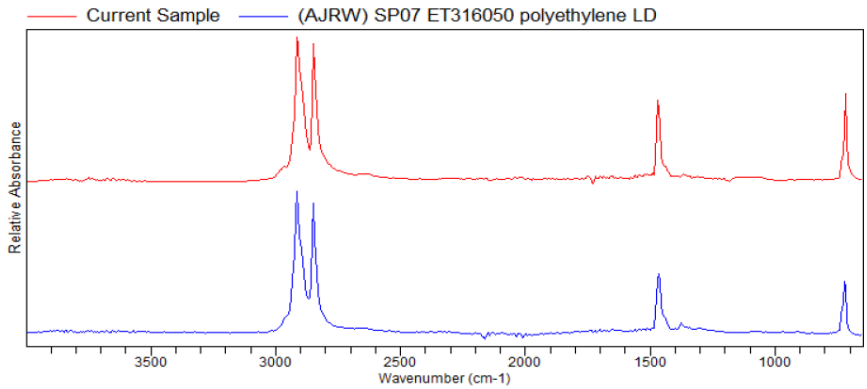
White and transparent particles with yellow stripped shards

FTIR:

Yellow shards: probably silica (recorded as fibre glass as closest quality match (86.59%)



White: Polyethylene (96.44%)

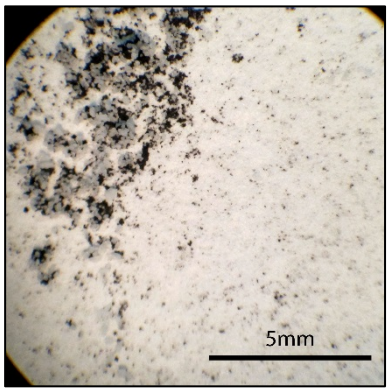


Clear: fibre glass (90%) probably silica

Confidence in result

Confident this is a correct detection

MB16004:



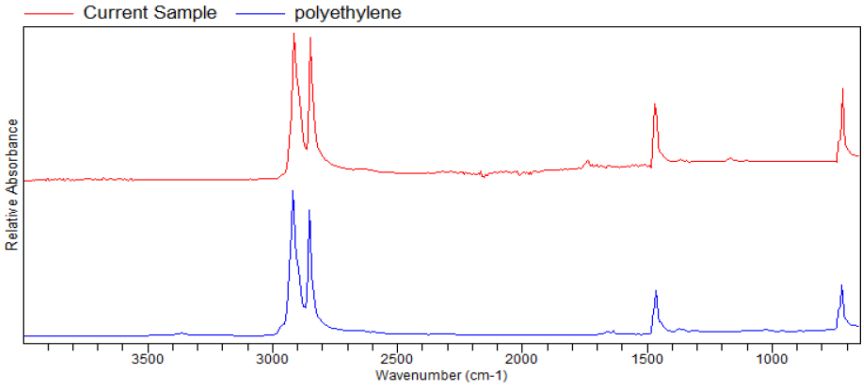
MISSHA Cacao & Cream Scrub

Description:

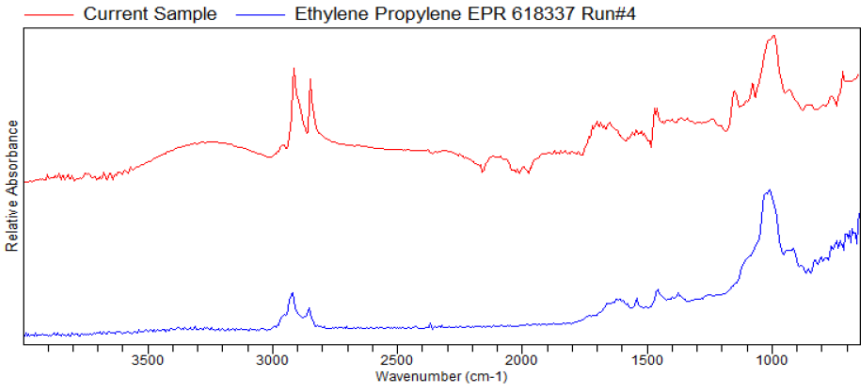
White and black particles. Black particles are sticky and therefore stuck to the filter paper and clumped together

FTIR:

White: Polyethylene (80.29%)



Black: Ethylene propylene (81.88%)

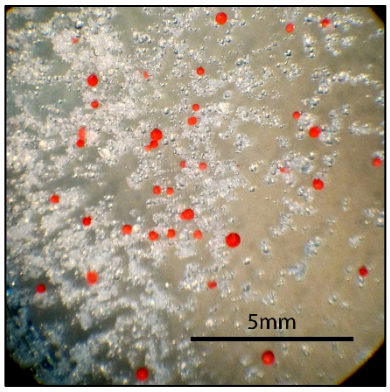


Confidence in result

White Particles are definitely Polyethylene

EPM physical characteristics do however fit the visual appearance in this structure (a rubbery stable polymer). The smaller peaks are quite different between the sample and the result but overall match quality is high.

MB16005:



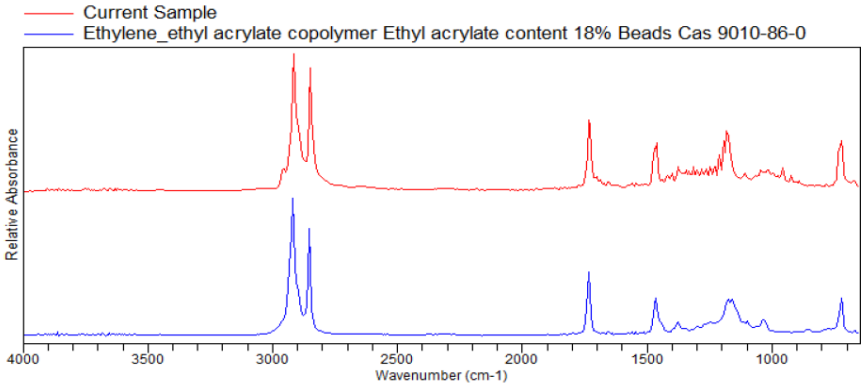
Eau Thermale Avene Gentle Purifying Scrub

Description:

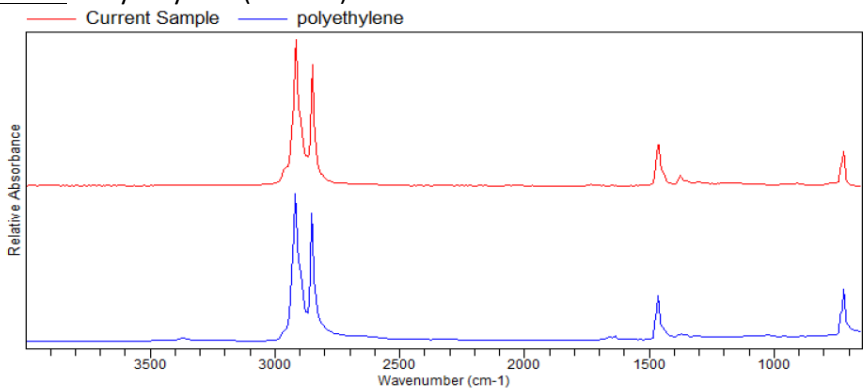
Red and White Spherical Particles, red more spherical than white and red particles breaks on under pressure of the forceps.

FTIR:

Red: either Ethylene- ethyl acrylate (77%) or Polyethylene. FTIR spectra of PE but with extra peaks



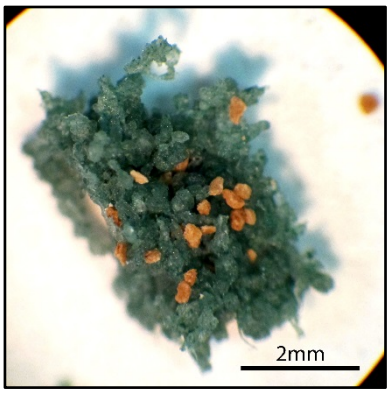
White: Polyethylene (96.43%)



Confidence in result

Confident of plastic present. Both particles are ethyl based. Red samples are most probably also polyethylene with additional constituents present.

MB16006:



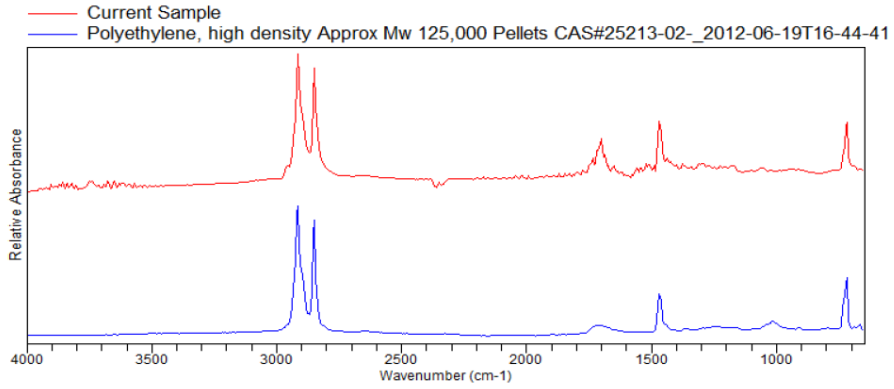
Himalaya - Neem scrub - purifying Scrub

Description:

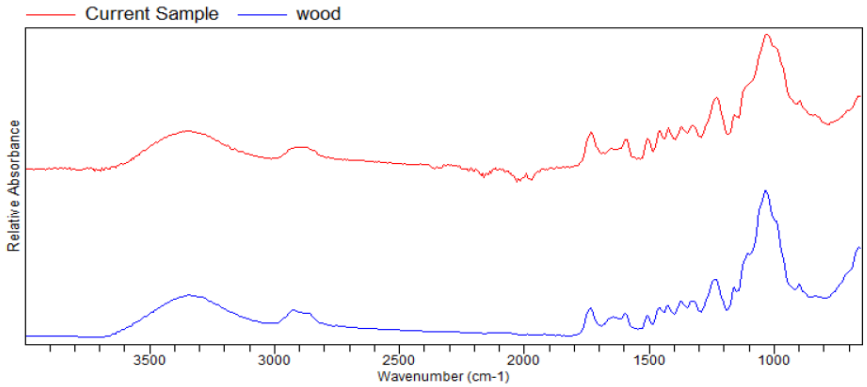
Clear particles covered in green pigment- irregularly shaped - along with brown angular particles

FTIR:

Green/clear: Polyethylene (79.74%)

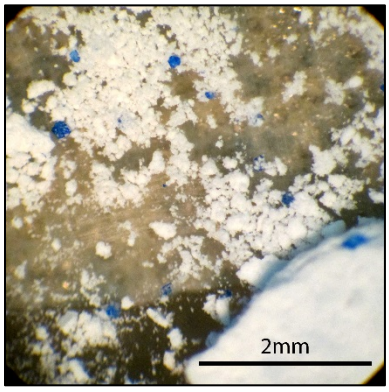


Brown: wood (95.28%)



Confidence in result

Confident that the green particle is plastic and likely to be polyethylene with additional constituents. Brown particles were identified as wood, but were most likely other similar organic matter, such as apricot shells.



MB16007:

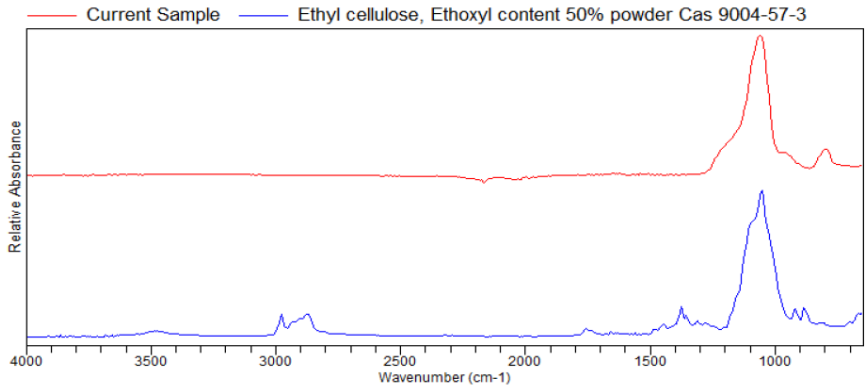
Colgate Max White One Toothpaste

Description:

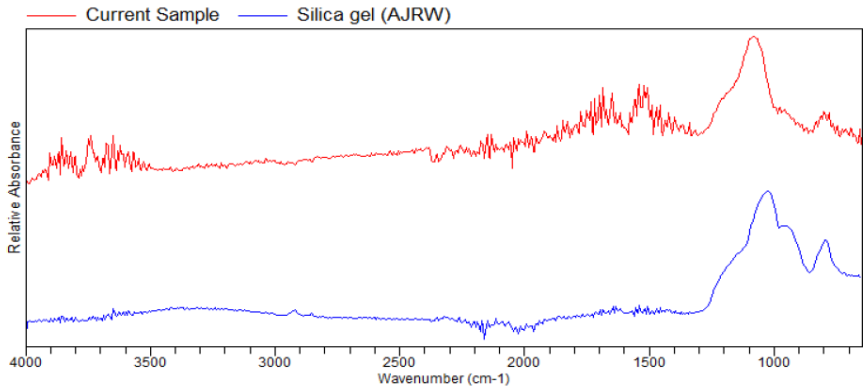
White Caked sample with blue specks- irregular shapes

FTIR:

White: Ethyl cellulose (90.09%)



Blue: low signal therefore noisy signal possible silica

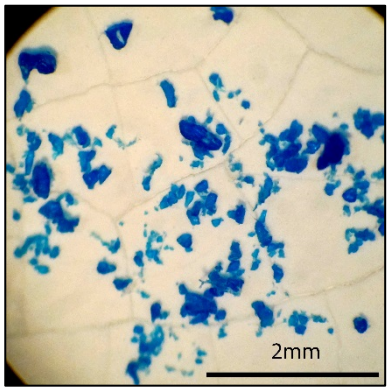


Confidence in result

No evidence of plastic.

Blue speckles break under pressure like silica, the FTIR spectra indicates a high probability that this is silica.

MB16008:



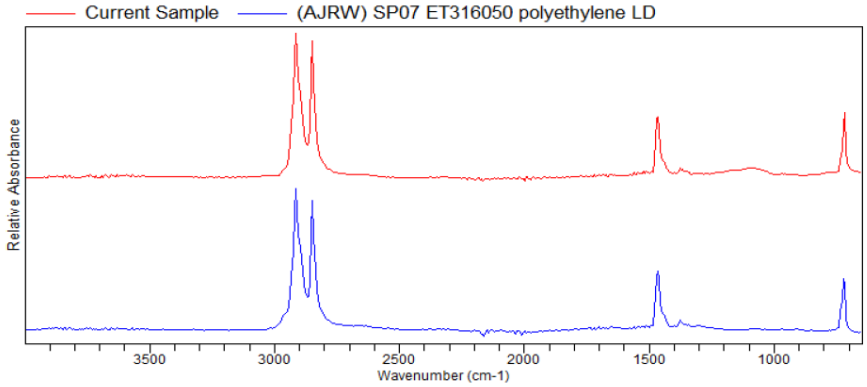
Arm & Hammer Truly Radiant toothpaste

Description:

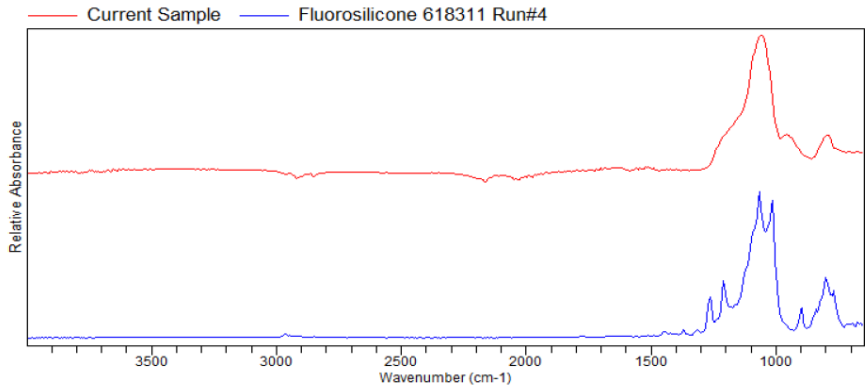
White caked sample with blue specks that fragment easily

FTIR:

Blue: Polyethylene (96.98%)



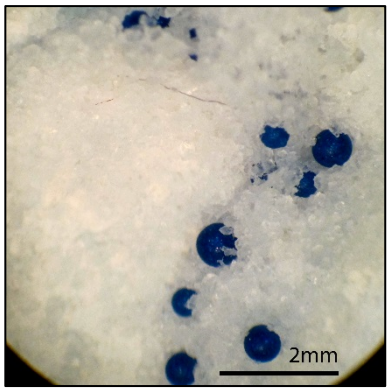
White: Possible Fluro silicone 618311 (89.82%)



Confidence in result

Confident that the blue particles are polyethylene, white substance could be the same as sample 7 white material (ethyl cellulose).

MB16009:



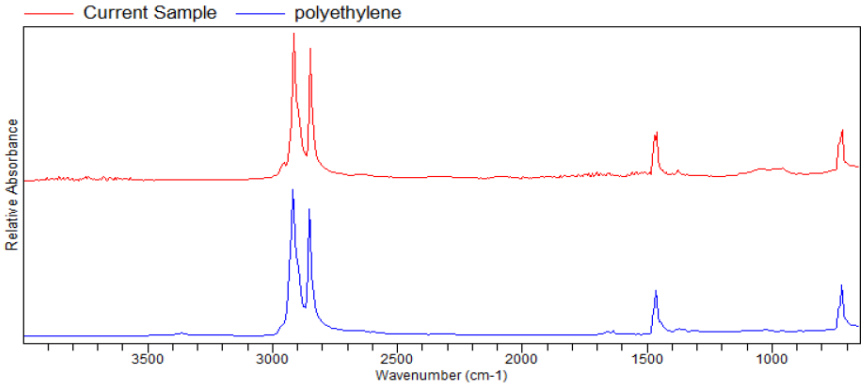
Clearasil Ultra Rapid Action scrub

Description:

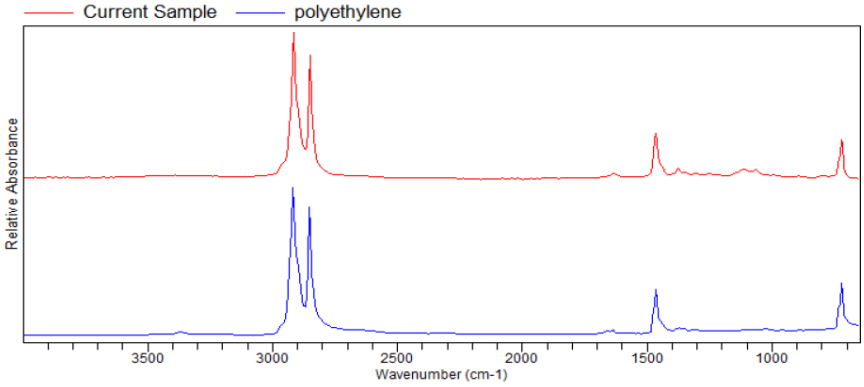
Small clear particles with larger blue spheres

FTIR:

Blue: Polyethylene (92.39%)

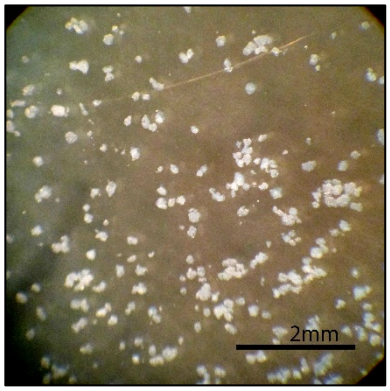


White: Polyethylene (95.90%)



Confidence in result

Both the white and blue particles are polyethylene



MB16010:

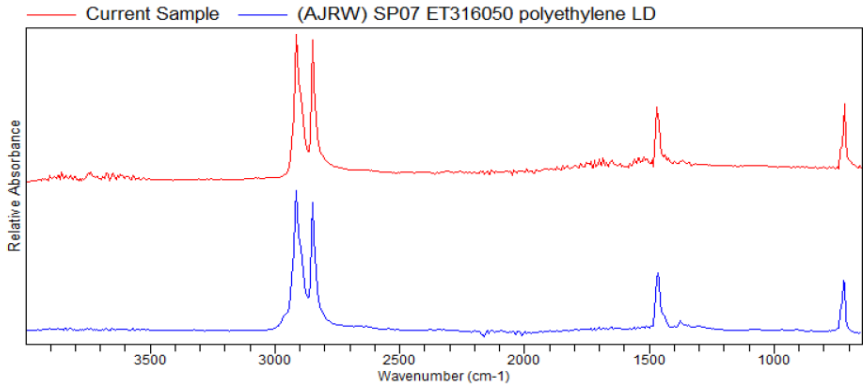
Clean & Clear Exfoliating Daily Wash

Description:

White particles

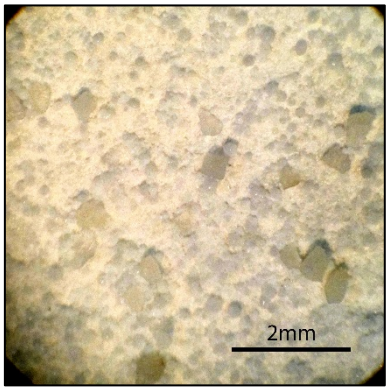
FTIR:

White: Polyethylene (84.89%)



Confidence in result

Theses are polyethylene particles



MB16011:

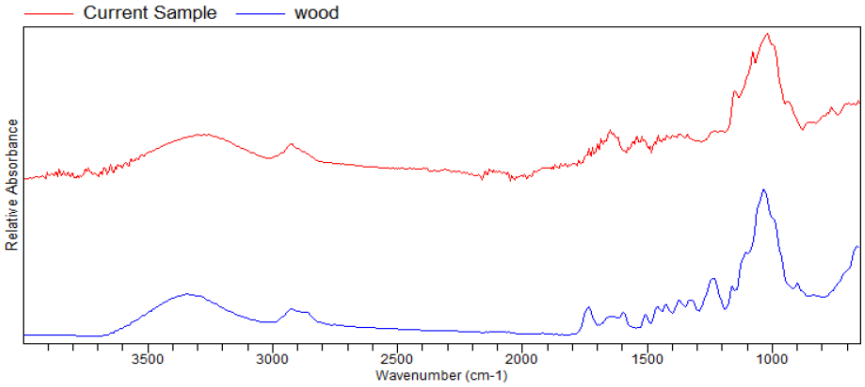
Simple Kind to Skin Smoothing Facial Scrub

Description:

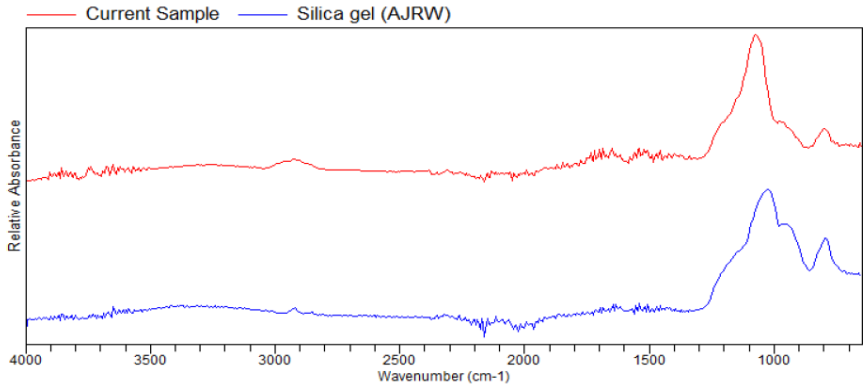
Smaller white fragment particles with larger off white particles

FTIR:

Off white: Wood (92.24%)



White: Possible silica (text find)

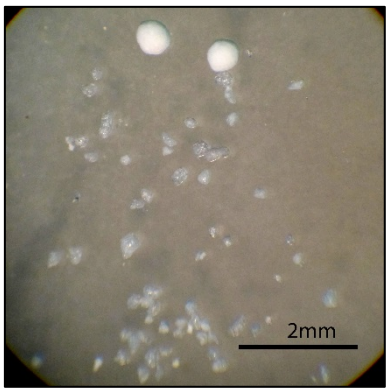


Confidence in result

Confident that there is no plastic in this sample

Off white sample was indicated as wood (similar to sample 6). White particles were unidentified however resemble silica in their spectra and physical appearance

MB16012:



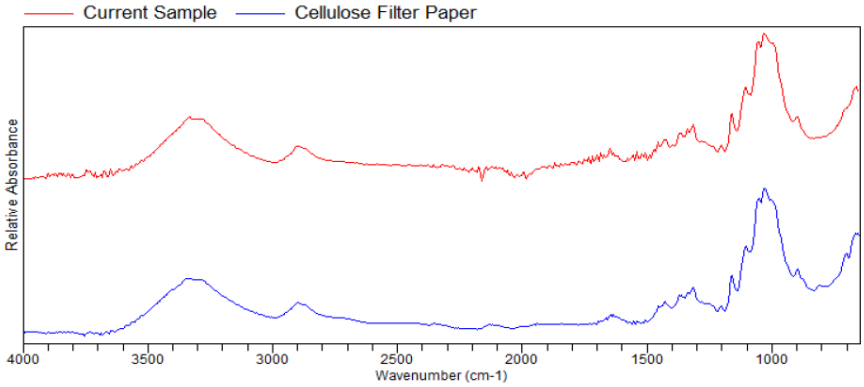
The Body Shop Tea Tree Squeaky Clean Scrub

Description:

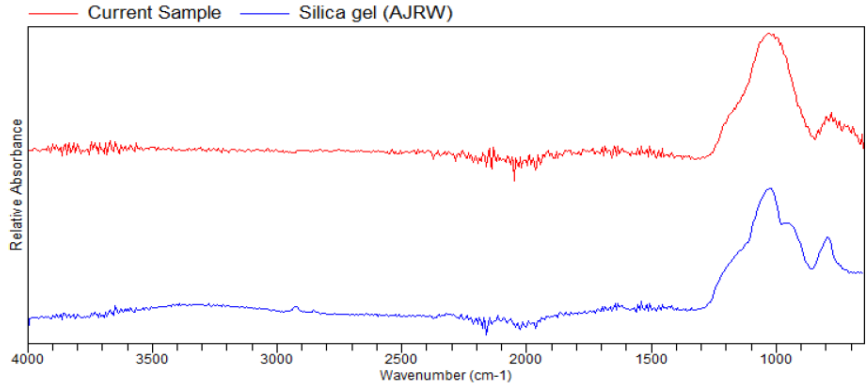
White and transparent fragmented particles

FTIR:

White: cellulose (98.16%)



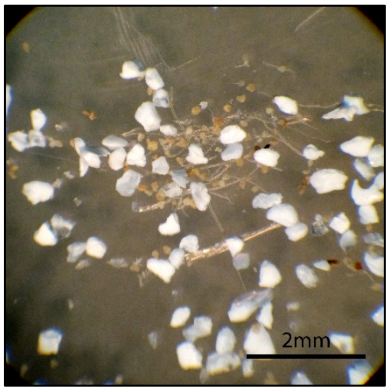
Clear: Silica (82.22%)



Confidence in result

No evidence of plastic

MB16013:



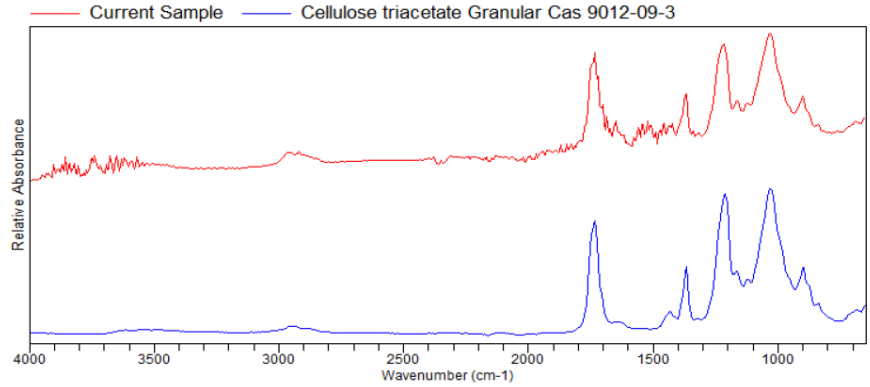
Innisfree green tea pure body gel scrub

Description:

White particles, smaller sand coloured particles, larger purple larger fragments

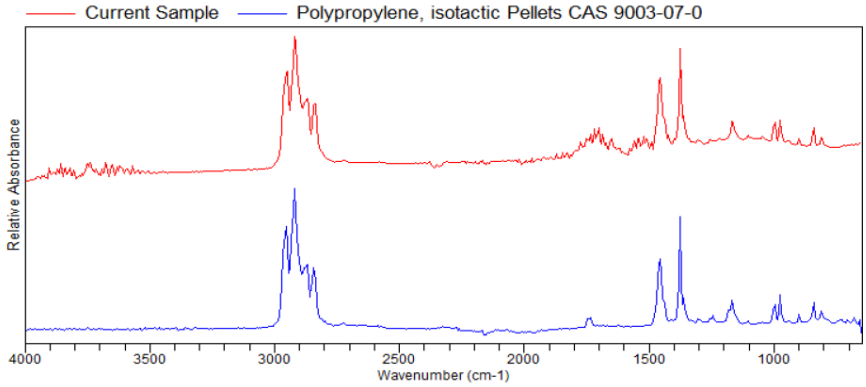
FTIR:

White: Cellulose triacetate 9012-09-3 (88.73%)



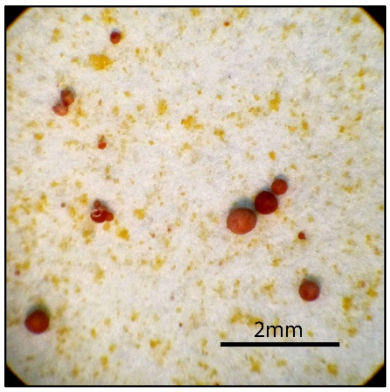
Sand coloured: insufficient sample for analysis

Purple: Polypropylene (text find)



Confidence in result

High probability that the purple sample is plastic, the spectra have a strong match with polypropylene. Sand coloured particles could not be collected in large enough numbers to get an accurate reading. White particle likely to be derived from cellulose.



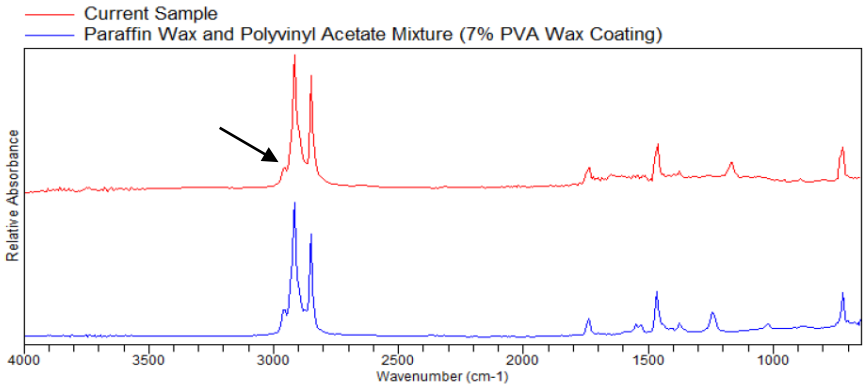
MB16014:

The Face Shop rice water bright all-in-one cleanser

Description: Multi-sized very spherical beads light to dark brown. Soft, will break under pressure of forceps.

FTIR:

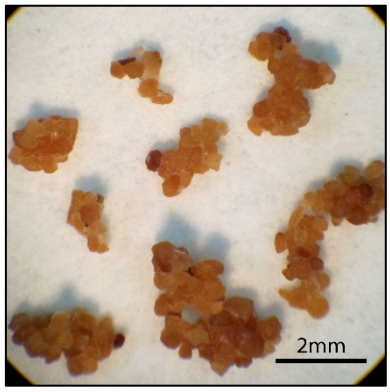
Brown: Possible paraffin wax with polyvinyl Acetate (88.78%) or Polyethylene with a few missing peaks (88.47%)



Confidence in result

This material is soft and will break with very little pressure of the forceps. It could therefore be paraffin wax (88.78%). The spectrum is, however, also very similar to polyethylene (88.47%). The small peak (indicated by the arrow) is present in the paraffin wax standard but not the polyethylene.

MB16015:

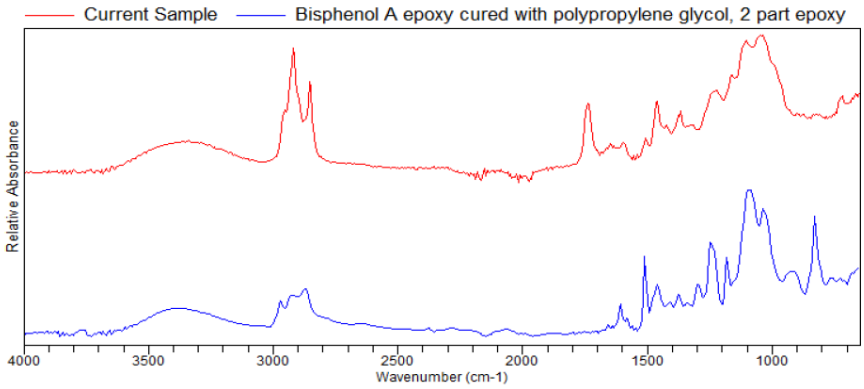


Lovely me:ex Mango Seed lip scrub

Description:

Large sand grained sized and coloured particles which amalgamate together

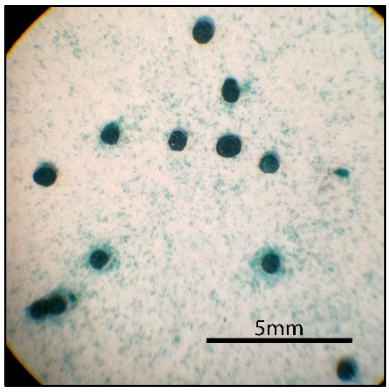
FTIR: Bisphenol A epoxy cured with polypropylene glycol, 2 part epoxy (81.75%)



Confidence in result

There was no clear identity for this sample. Two of the library spectra matches related to the polycarbonate monomer bisphenol A (BPA), which might indicate the presence of BPA in the particle matrix. However, this would need to be confirmed using mass spectrometry for full chemical analysis.

MB16016:



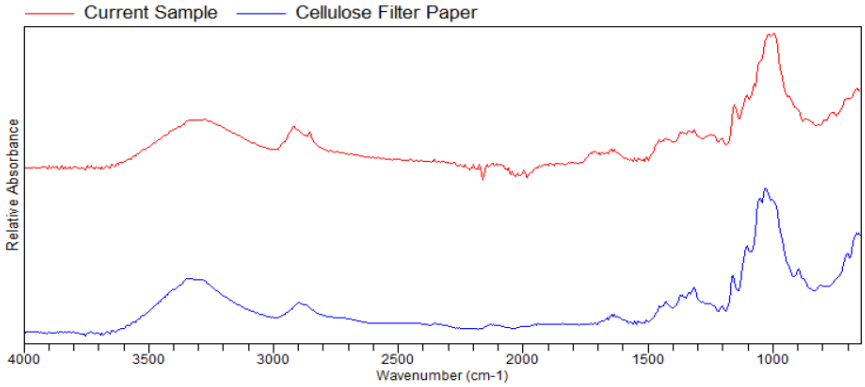
Amorepacific - Ryo - Hair shampoo, scalp deep cleansing shampoo - acrylate copolymer

Description:

Large slightly spherical green pigmented particle - pigment leaching readily to filter. Soft particles that can be squashed under physical pressure of FTIR analysis

FTIR:

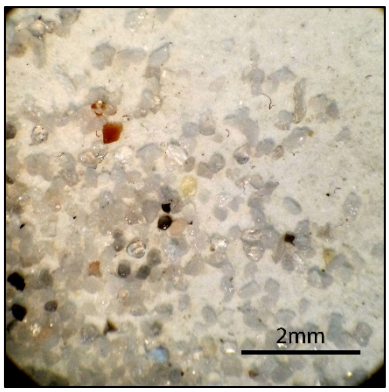
Green: Cellulose (95.93%)



Confidence in result

No evidence of plastic present

MB16017:



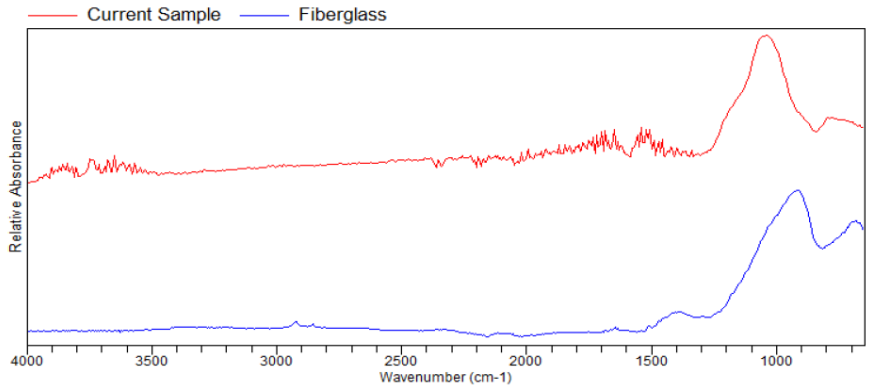
Unilever - St.ives - exfoliating body wash, smoothing apricot - acrylate copolymer

Description:

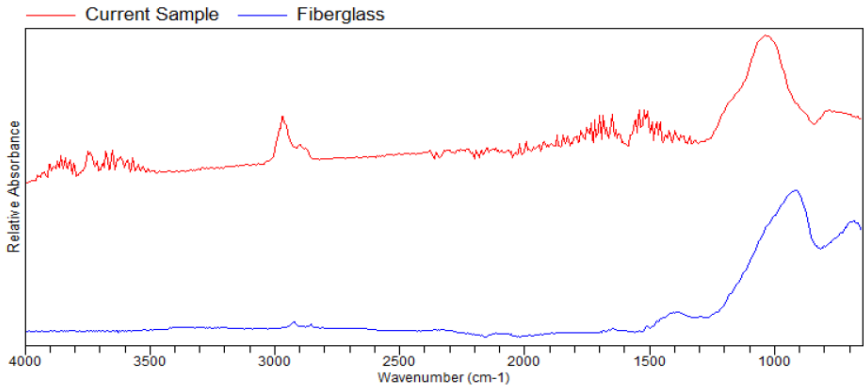
Transparent, sand coloured and black fragment particles - very hard material – difficult to get reliable contact and signal on FTIR

FTIR:

White: Silica – recorded as fibre glass as closest quality match



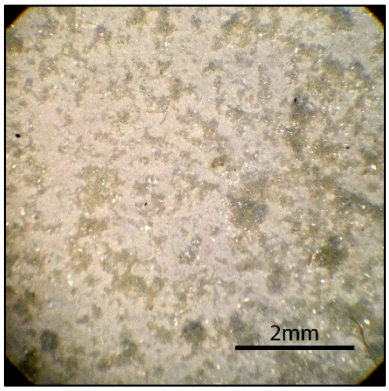
Black: Silica- recorded as fibre glass as closest quality match but with another peak



Confidence in result

No evidence of plastic in this product

MB16018:

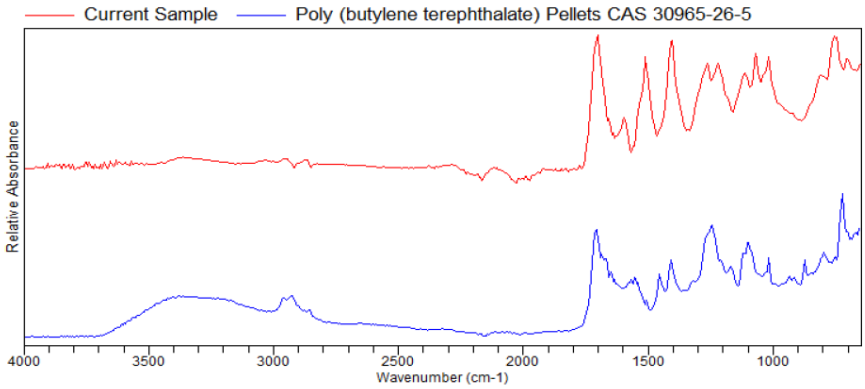


Global cosmed internation - Domax - kitchen seramic cleaner - 'an abrasive' included

Description:

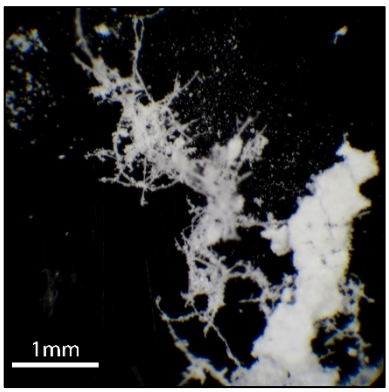
Transparent fragmented particles

FTIR:
White: either poly butylene terapthalate (78%), Polyurethane (76%) or PET (76.56%)



Confidence in result
Confident that this is plastic, but not confident which polymer.

MB16019:



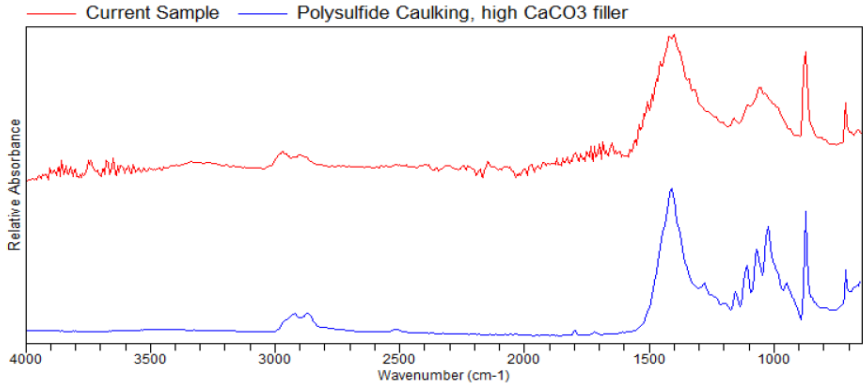
LG HH - Bathroom cleaner - Homestar Power scrub for bathroom cleaning - 'an abrasive' included

Description:

White caked sample with fibres embedded throughout the sample. Fibres removed and washed with ethanol to remove white substance. Fibres then dried at 60°C for 10 minute.

FTIR:

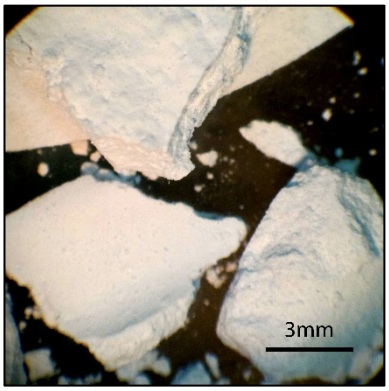
White fibres: Unknown possibly a polysulfide polymer



Confidence in result

These fibres were confirmed not to have been derived from the filter paper. Identity of these fibres is not known, but appear to be either either derived from, or coated in, polysulfide caulking agent (an elastomer).

MB16020:



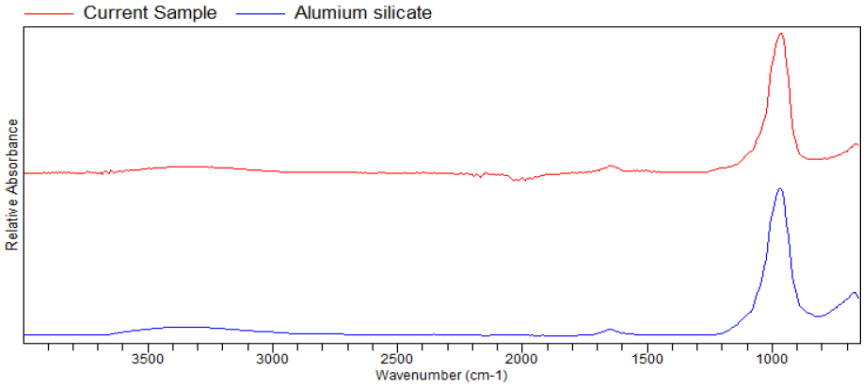
CJ Lion - washing powder - BEAT - green or blue beads in it

Description:

White caked sample with no visible green or blue beads

FTIR:

White: Aluminium silicate (98.30%)



Confidence in result

The green and blue beads mentioned in the sample description could not be found in the filtered samples as delivered for FTIR analysis.

MB16021: Gatsby - Hair wax - Styling wax, ultra-hard type - Polyethylene, polysorbate60

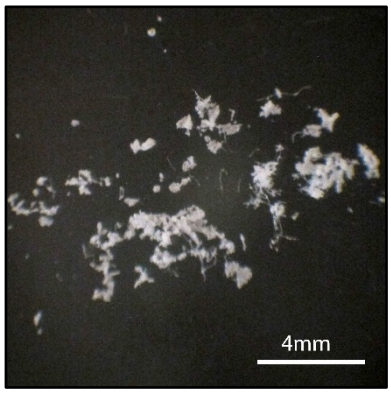
Description: small white fragmented particles

FTIR:
White: insufficient sample for FTIR analysis

MB16022: Bullsone - Swirl remover, Car compound

Description: no solid material could be found on the filter to subject to FTIR analysis

MB16024:



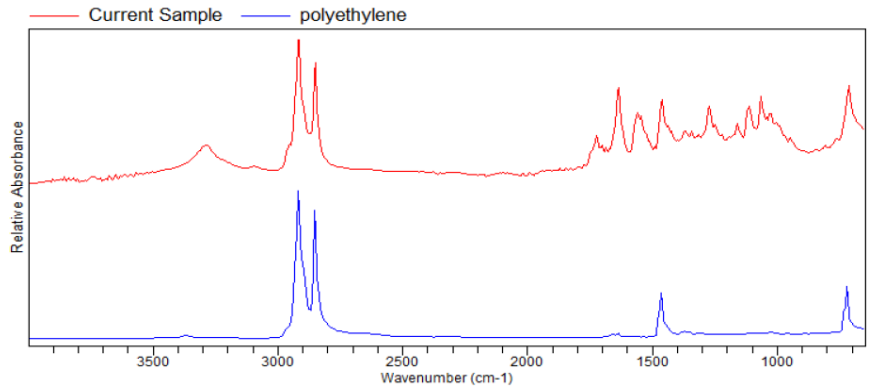
The Real Shaving Company Multi-task Super 8 balm

Description

A few white particles, no caking

FTIR

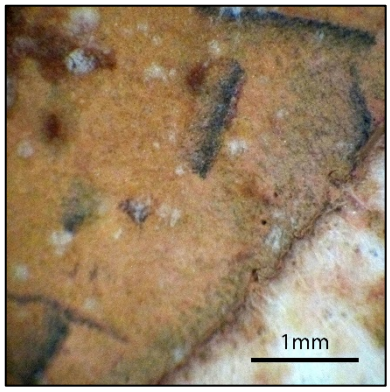
White: No results suggested by data base search. Polyethylene possible but with extra peaks (Text Find)



Confidence in result

Relatively confident that this is polyethylene but with additional constituents. Physical appearance is similar to other polyethylene microplastics found in cosmetics.

MB16025:



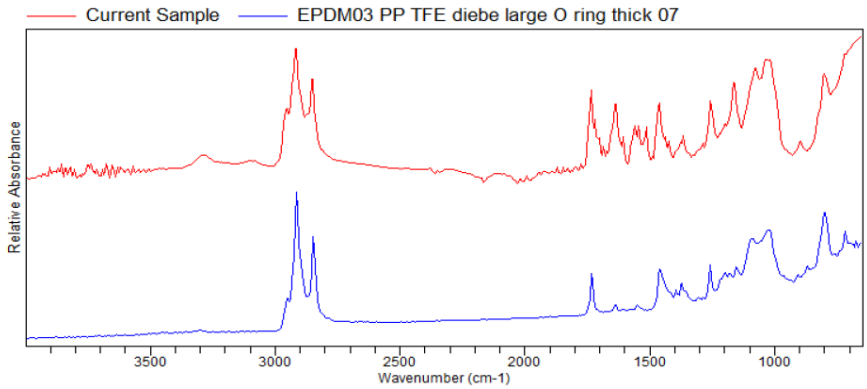
L'Oreal Lancombe Hydra zen BB cream

Description

Brown clay like caking, no hard microbeads/fragments present

FTIR

Brown: Double peak around the 2700-2800 cm-1 wavelengths suggest a synthetic polymer – however, not likely to be plastic.



Confidence in result

There were no fragments seen in this sample - sample was the brown paste. No evidence of plastics found