



D3.1 DATABASE OF STUDIED WASTE FRACTIONS, THEIR ANALYTICAL RESULTS AND PROCESS CASCADES THEY WERE SUBJECTED TO

WORK PACKAGE 3

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Characterisation and qualification of waste plastics

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PUBLISHABLE EXECUTIVE SUMMARY

As part of the CIRCULAR FoodPack project, the polymer composition of flexible packaging waste fractions from sorting sites in three European countries, France, Belgium and Germany, was analysed. This current work was carried out from June 2021 on, as part of Task 3.1, Work Package 3 - Purification and Recycling of Flexible Packaging, and will still be ongoing until the end of the project (November 2024, Month 42). In the course of the project, the characterization will also be extended to flexible packaging waste fractions, which will be sorted using improved sensor-based and tracer-based sorting methods that are being developed and optimized within the project. The aim of these trials is to support the development of these sorting technologies by providing information on the sorted waste streams, and to help in the selection of suitable processes for the recycling of the respective waste fractions.

So far, three polyethylene (PE) rich packaging waste fractions have been investigated, each divided into food and non-food packaging: flexible packaging from France and Belgium, and LDPE-rich films from the German sorting fraction 310. With the help of specific solvents from the CreaSolv® Process developed at the Fraunhofer IVV, the single plastic types within the fractions are consecutively dissolved and a mass balance is calculated.

It has been shown that the non-food packaging has a significantly higher PE content of 80-90% than the food packaging (60-70%). In a European comparison of the overall flexible packaging waste composition the French sorting site had the highest PE share (91%), followed by the German (89%) and the Belgium sorting site (83%). Other polymers present in the fractions could be identified and quantified as Polypropylene (PP), Polyamide (PA) and Polyethylene terephthalate (PET). The enclosed foreign polymers (other than PE), with the main component of PP, may affect recyclability or lead to a reduction in the recyclate quality. Mass balancing was supported by analyses for polymer type and purity of the separated polymer fractions using Fourier-Transform Infrared Spectroscopy (FTIR) and Differential Scanning Calorimetry (DSC).

With the results gathered in this study, a database was set up to compile the information on the waste fractions considered in this project. The database will support the development of sorting technologies within the project and help to organize and streamline the recycling of these fractions with suitable recycling methods.



ABBREVIATIONS

Abbreviation	
DoA	Description of Actions
DSC	Differential Scanning Calorimetry
EVOH	Ethylene Vinyl Alcohol
FTIR	Fourier Transform Infrared Spectroscopy
LDPE	Low-density polyethylene
LLDPE	Linear low-density polyethylene
NIR	Near Infrared Spectroscopy
PA	Polyamide
PE	Polyethylene
PET	Polyethylene terephthalate
PO	Polyolefine
PP	Polypropylene
PS	Polystyrene
PVC	Polyvinylchloride
WP	Work package



1. INTRODUCTION

Work package (WP) 3 deals with the recycling of flexible packaging waste. The aim is to develop and apply recycling processes that enable the recyclates to be used in novel food packaging. For this purpose, deodorisation technologies are developed to actively remove odours from the recyclate. Furthermore, delamination and deinking procedures are investigated to remove a high amount of printing inks. In order to obtain pure recyclates from multilayer films or composites, the CreaSolv® Process is used and further developed, which can produce high-purity recyclates using specific solvents. The aim is to obtain the purest recyclate possible by combining these processes in cascades.

According to the Description of Actions (DoA), as a first step, Task 3.1 will characterise the polymer composition of various waste streams of flexible packaging, which is related to this deliverable. Firstly, the current status of flexible packaging sorting fractions provided by SUEZ from three different European countries, France, Belgium and Germany, will be mapped. Previous scientific work has mainly used Near Infrared Spectroscopy (NIR) to analyse the polymer composition of larger quantities of shredded flakes from film sorting fractions of European countries for example the Netherlands [1]. Multi-material composite packaging, however, is attributed to a single polymer in this case. McKinlay et al. have shown that these make up a significant proportion in the sorting fractions (around 20% in the flexible packaging waste of the UK) [2]. Detailed studies of the polymer composition of individual selected packages, including monolayer and multilayer films, have already been carried out by Roosen et al. [3]. The aim of CIRCULAR FoodPack project is to investigate the polymer composition not only of individual packaging items but on a sample basis of the film sorting fractions of three countries in Europe. Selective dissolution is used to enable also the separation of multi-material composite packages into their individual polymer fractions and determine their mass fraction. The trials serve as an exemplary study to determine the PE and Polyolefine (PO) content on the one hand and to understand the type and extent of impurities in order to estimate their influence on mechanical and solvent-based recycling, on the other hand. For solvent-based recycling, the yield of the target polymer can thus also be estimated in advance.

Furthermore, the project will investigate and further develop different sorting technologies, such as sensor- or tracer-based sorting. The resulting sorting fractions are processed by the project partners, analysed in terms of their composition during the course of the project and compared with the current state of sorting. Fraunhofer IVV uses specific solvents to dissolve the various enclosed polymers one after the other, and calculates mass balances accordingly. The results are supported by analyses for the purity of the individual fractions. Together with information on the waste fractions gathered in WP2, the collected data is fed into a database, which is stored on EMDESK and continuously updated by the WP3 partners.



2. MATERIALS

In the course of the project, different samples, which differ essentially by the type of sorting but also by their origin, are being analysed for composition and other specific properties like odour components. Flexible packaging waste from households waste (sample A) collected and sorted in three different European countries, France, Germany and Belgium, were characterized in Task 2.1 by SUEZ to determine the composition in terms of packaging categories. The PE rich sorting fractions were sorted into two categories, food and non-food packaging, which are used as input for Task 3.1. The samples do not only differ in their origin, but also in the sorting that was performed in the respective sorting site. All samples were passed through ballistic separation, wind sifting, NIR sorting and final manual sorting. For the French waste one NIR separation step focussing on PE was performed, the Belgian sorting site uses two NIR sorting steps focussing on PE. From the German sorting site three different sorting fractions for PE and polyolefins are characterized, which are sorted by three consecutive NIR sorting steps. The first fraction contains mainly Low-Density Polyethylene (LDPE) films and is being referred to as number 310. The second fraction, number 323-2, is sorted for flexible polyolefin packaging and the third fraction, number 352, contains flexible packaging mixed with other lightweight plastic materials.

At a later stage in the project, also samples prepared by the project partners, marked and sorted by tracer based sorting (i.e. sample B), and benchmark samples from outside the consortium, marked and sorted by Digital Sorting Technology, e.g. HolyGrail project (i.e. sample E) will be analysed. Additionally, WP4 will provide samples fortified with organic contaminants (i.e. sample D) to determine the purification potential of single processes (challenge test).

All samples and its analytical data will be fed into the database displayed in this deliverable.



3. CHARACTERIZATION OF WASTE COMPOSITION

The collected and sorted flexible packaging waste (Figure 1) is washed and shredded by SUEZ, as described in D2.2, and sent to Fraunhofer IVV for characterisation of the contained polymer types. With the specific solvents from the CreaSolv® process, the contained polymer types are consecutively dissolved (Figure 2). For this purpose, 200 g of the respective waste fraction is used as input and treated with the solvents in a beaker with a volume of 5 L under defined laboratory conditions, which cannot be specified in more detail due to confidentiality reasons. The waste is fed into the respective solvent and the targeted polymer is dissolved. The dissolved polymer (Figure 3) is separated from the residue (Figure 4) after each step by filtration with a 25 µm fine filter. For polymer types that occur in higher proportions, the residue is re-extracted again under the same dissolution conditions to completely dissolve and separate adhering residues of the dissolved polymer and any residual undissolved portions of the targeted polymer. The filtrated polymer solution is weighted and the dry weight of the polymer is determined with an infrared moisture analyser MA35 from Sartorius AG, Göttingen. The following polymer types are separately dissolved during the tests: PE, PP, PA, Polyvinylchloride (PVC), Polystyrene (PS), PET. The composition of the waste fraction is calculated by mass balancing. The dissolved polymer fractions are dried in a vacuum oven to recover the polymers for further analysis.

At this point in the project, only single determinations of the mass balance have been carried out. In the further course of the project, all tests will be carried out a second time in order to show the influence of the heterogeneity of the shredded input material and to include it in the results.



Figure 1: Flexible food packaging waste (FR) Figure 2: Dissolution of PE



Figure 3: Dissolved PE



Figure 4: Residue after PE extraction



Figure 5: Residue after consecutive extraction of all polymers

Table 1 shows the composition of the PE rich flexible packaging waste fractions from France, Belgium and Germany (310), whereas Table 2 and Table 3 display the composition divided into food and non-food packaging. With ~90% PE share, the sorting fractions from the French and German sorting site have a higher PE content than the Belgian sorting fraction (~83% PE). However, it should be noted that the Belgian and German sorting fractions show certain losses of 3-6 % in the mass balance. In further trials these losses, which are mainly due to unavoidable residues on laboratory equipment, will be analysed for polymer type and added to the respective fractions. The collected non-food packaging has a significantly higher PE content than the food packaging in all three cases, which is due to their high shares of secondary packaging and bags. The collected food packaging contains 30-40 % foreign polymers, which hamper the recyclability of these fractions. Especially the Belgian food fraction with ~22 % contains a high share of PP. The results are within the range of known studies on the polymer composition of flexible packaging waste, although the PE fraction is about 7-14% higher than in Brouwer et al. [1]. However, a comparison of the different studies is only possible to a limited extent due to the different sample selection and analysis methods used.

For PS and PVC, which are extracted in the first two dissolution steps, a mass share of 0,1-3 % is measured. However, in FTIR analysis the dried recovered polymers could not be unequivocally identified as PS or PVC. The measured mass fraction also contains easily soluble barrier polymers, such as ethylene vinyl alcohol (EVOH), adhesives and printing inks. The different components

cannot be clearly distinguished with the available analytical methods and are therefore added to the residue.

The residue after all dissolution steps contains mainly paper and aluminium, but also metal wires were found (Figure 5). Also other insoluble components such as adhesives also remain in the residue.

Table 1: Composition of sorted flexible packaging waste fractions from French, Belgian and German sorting site

Country	PE [w%]	PP [w%]	PA [w%]	PET [w%]	Residue [w%]
France	91	3,5	1,5	1	1,7
Belgium	83	6,2	1,1	1,1	5
Germany (310)	89	2,3	2	0,9	2,7

*Table 2: Composition of sorted flexible **food-packaging** waste fractions from French, Belgian and German sorting site*

Country	PE [w%]	PP [w%]	PA [w%]	PET [w%]	Residue [w%]
France	70	11	8,7	5,8	3,2
Belgium	58	22	5,1	5,1	3,9
Germany (310)	73	6,7	8,9	4,5	3,3

*Table 3: Composition of sorted flexible **non-food-packaging** waste fractions from French, Belgian and German sorting site*

Country	PE [w%]	PP [w%]	PA [w%]	PET [w%]	Residue [w%]
France	95	2,1	0,2	0,2	1,5
Belgium	88	2,6	0,2	0,3	5,2
Germany (310)	93	1,3	0,4	0,1	2,6

4. ANALYSIS

The separated dried polymer fractions are analysed for purity using Fourier Transform Infrared Spectroscopy (FTIR) and Differential Scanning Calorimetry (DSC) measurements.

4.1 INFRARED SPECTROSCOPY

The dried polymer fractions are analyzed by means of FTIR measurements. This allows the polymers to be clearly identified on the basis of specific spectra and possible contamination by foreign plastics to be detected. Infrared spectroscopy is a physical analysis method that works with infrared light. When a material is irradiated with electromagnetic waves, certain frequency ranges are absorbed, causing the bonds to vibrate. These are visible in the form of deflections of the transmission in the measured spectrum. The frequency required for this is characteristic of the respective bond, which means that materials can be identified with the aid of databases.

The spectra of the measured polymer fractions are shown in Figures 6-17. It can be seen that the spectra of the measured samples match the spectra of the respective plastics stored in databases with a high degree of accuracy. This indicates that the polymers can be recovered in high purity. The measurements of the Belgian non-food PA fraction did not give clear results, therefore PA cannot be clearly detected in this fraction. The measured weight share of the PA fraction (0,2%) is therefore added to the residue.

With the aid of DSC measurements, these observations can be verified, since even minor impurities from foreign polymers can be detected.

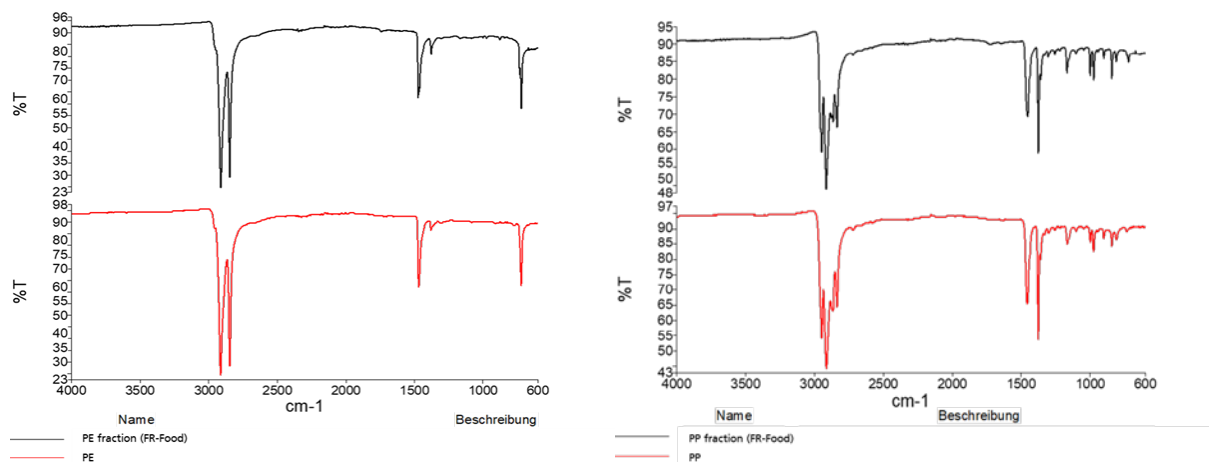


Figure 6: FTIR pattern of PE (left) and PP (right) fraction - French sorting site (food)

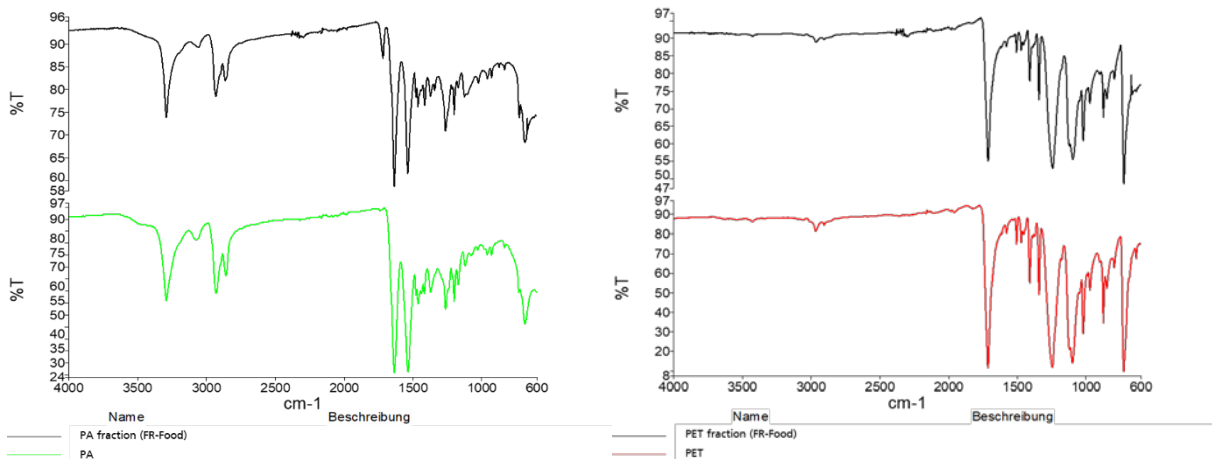


Figure 7: FTIR pattern of PA (left) and PET (right) fraction - French sorting site (food)

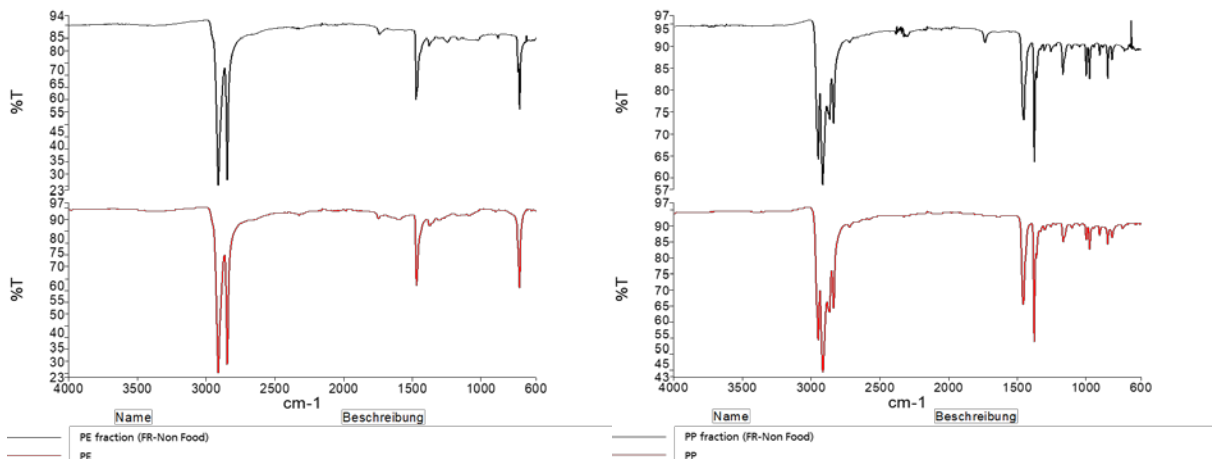


Figure 8: FTIR pattern of PE (left) and PP (right) fraction - French sorting site (non-food)

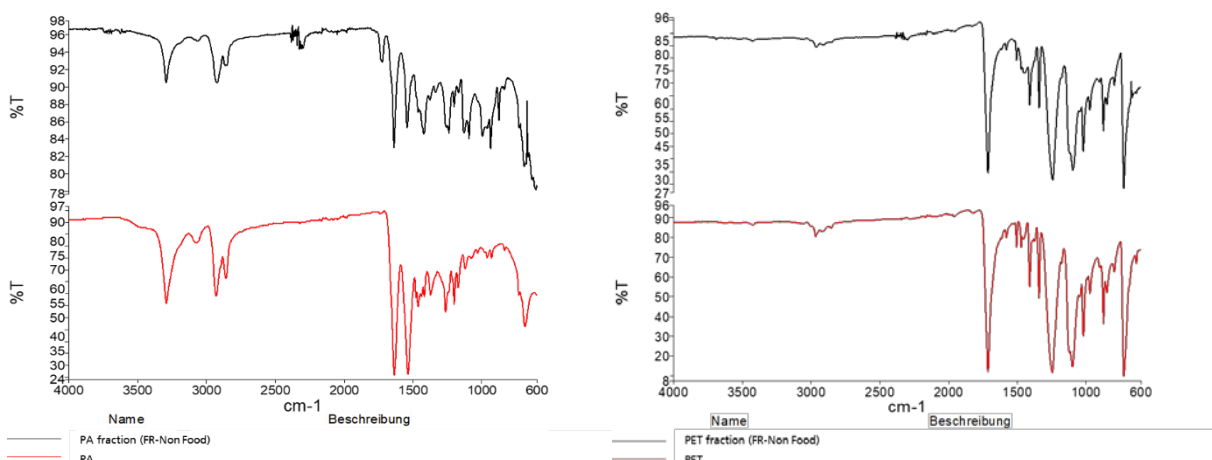


Figure 9: FTIR pattern of PA (left) and PET (right) fraction - French sorting site (non-food)

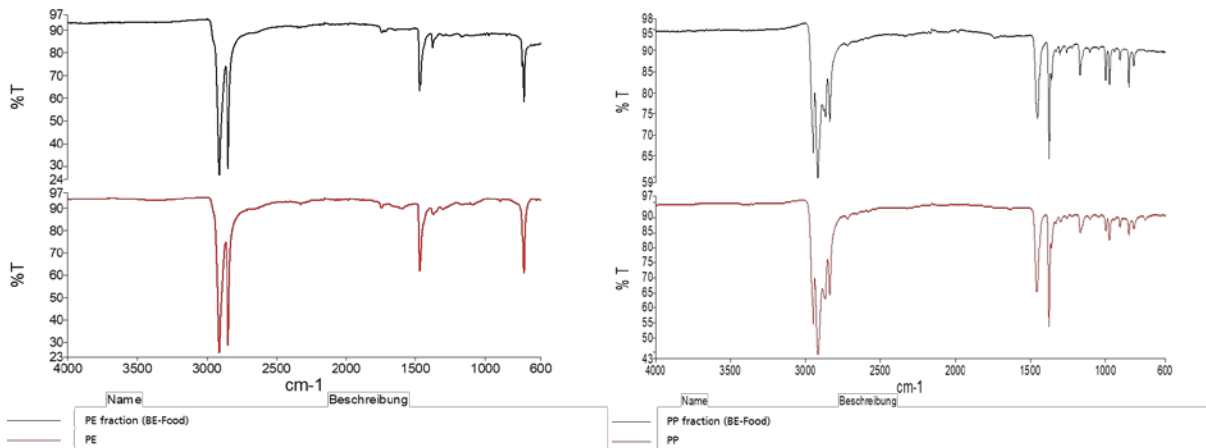


Figure 10: FTIR pattern of PE (left) and PP (right) fraction - Belgian sorting site (food)

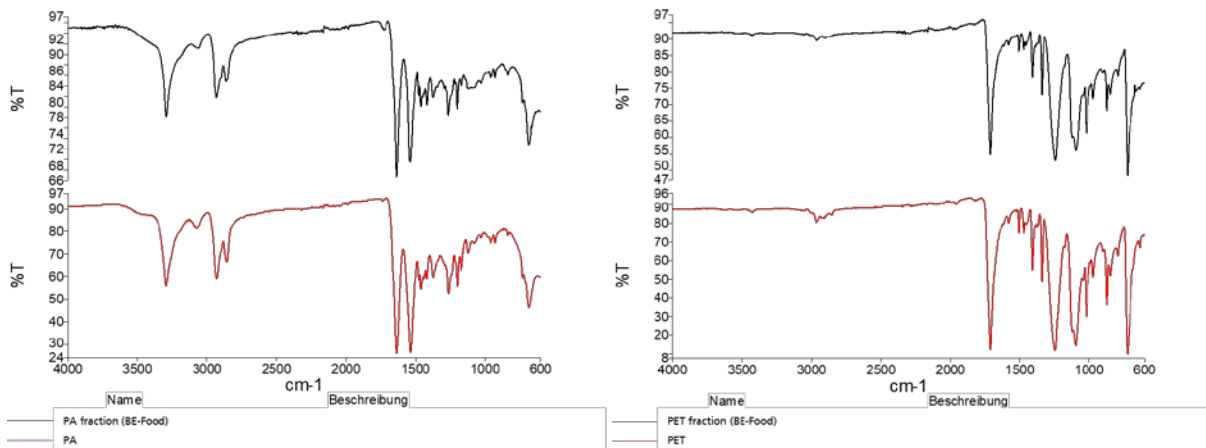


Figure 11: FTIR pattern of PA (left) and PET (right) fraction - Belgian sorting site (food)

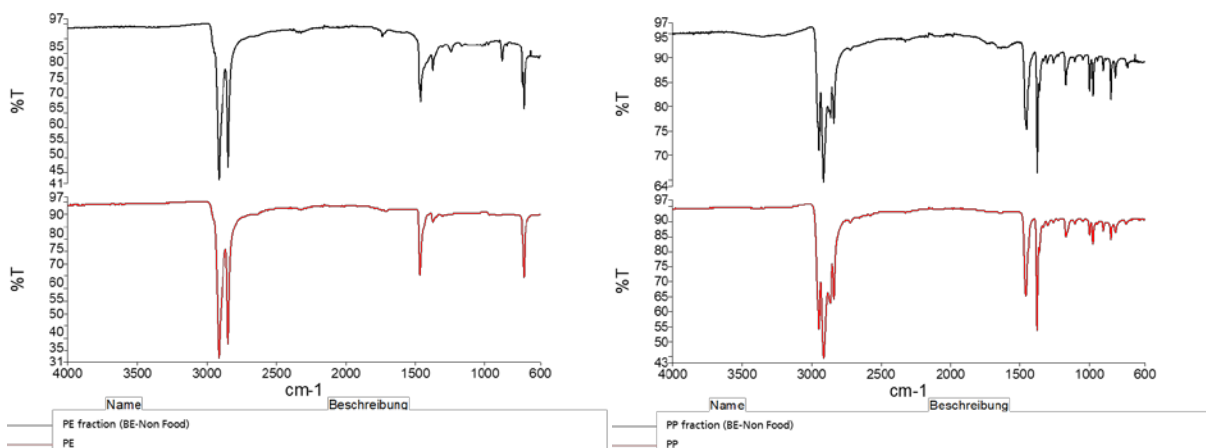


Figure 12: FTIR pattern of PE (left) and PP (right) fraction - Belgian sorting site (non-food)

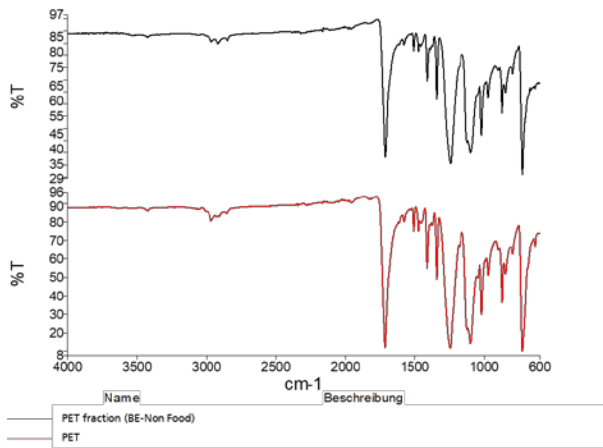


Figure 13: FTIR pattern of PET fraction - Belgian sorting site (non-food)

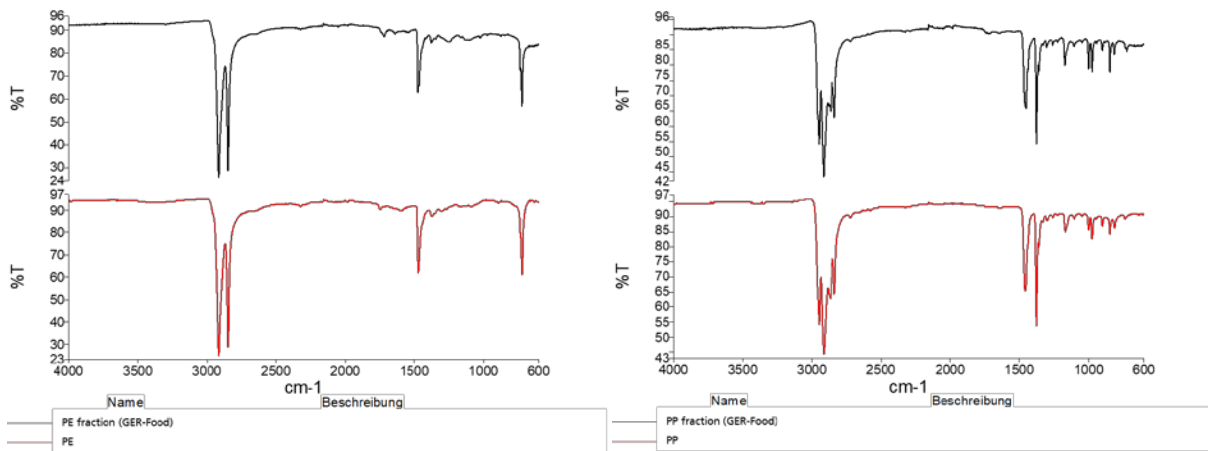


Figure 14: FTIR pattern of PE (left) and PP (right) fraction - German sorting site (food)

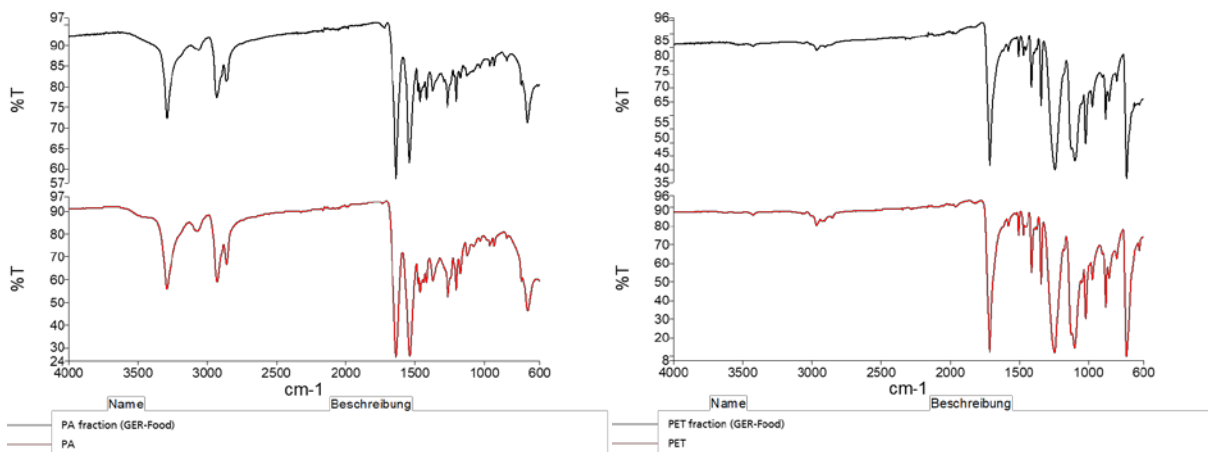


Figure 15: FTIR pattern of PA (left) and PET (right) fraction - German sorting site (food)

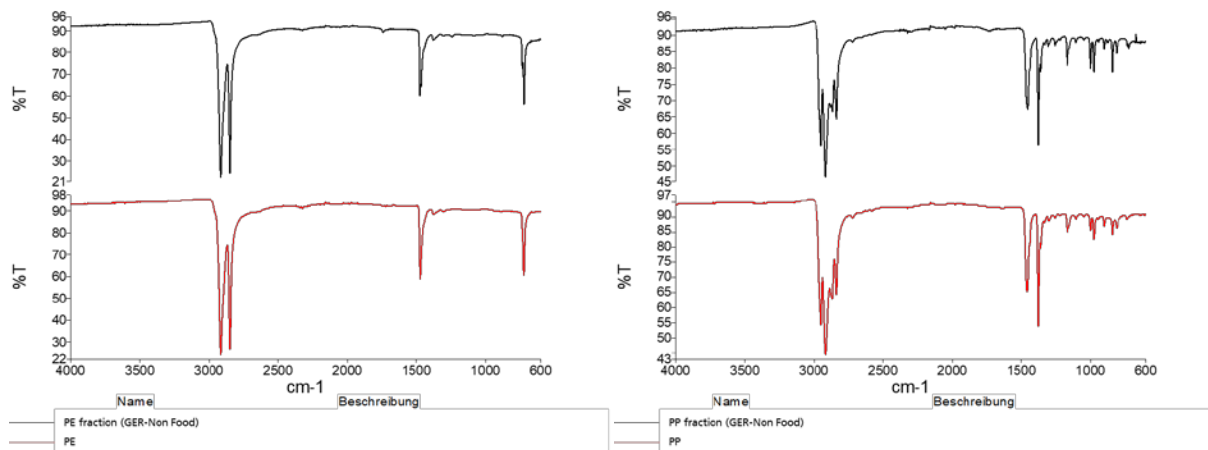


Figure 16: FTIR pattern of PE (left) and PP (right) fraction - German sorting site (non-food)

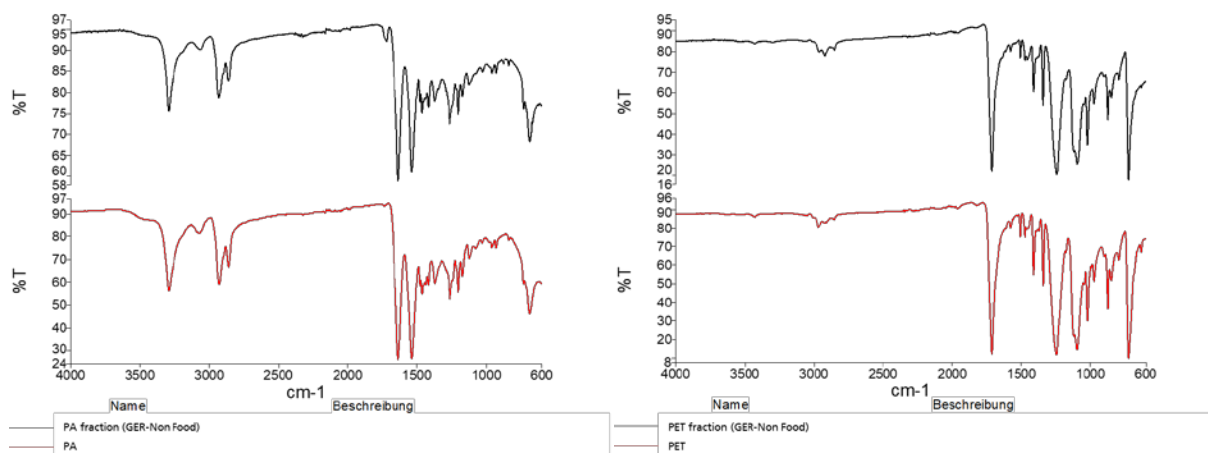


Figure 17: FTIR pattern of PA (left) and PET (right) fraction - German sorting site (non-food)

4.2 DIFFERENTIAL SCANNING CALORIMETRY

With DSC the thermal properties e.g. glass transition temperature or melt temperature of polymers can be determined. This can be used to identify the polymer type of a sample from its typical thermal properties. DSC measures the amount of heat required to increase the temperature of a sample and a reference. During physical or chemical transformations of the sample, the required amount of heat differs from the sample depending on the heat consumed or released during the process. The difference between sample and reference is plotted as a function of temperature. Typical transitions of polymers like glass transition or melting are visualised as peaks.

Here the samples are investigated for their melting temperature to identify the polymer types in the separated polymer fractions. The DSC curves are shown in the annex. The first heating run (black curve) is used to eliminate the thermal prehistory of the material, before it is cooled at a fixed rate and heated for a second time. Therefore, conclusions about the thermal properties of the materials are usually made on the basis of the second heating run (red curve).

The samples of the PE fractions from all three countries are dominated by two melting peaks at 105-115 °C and 120-125 °C which corresponds to LDPE and Linear Low-Density Polyethylene (LLDPE) [4]. These PE types are usually used for packaging applications. Typical melting temperatures of all polymers considered are given in Table 4. The samples from the Belgian and German non-food PP fractions are showing a small LLDPE peak besides a larger PP peak, indicating a < 5% share of PE. This can probably be traced to PE that sticks at the residue after PE extraction due to the high PE content. So the PE shares of the Belgian and German non-food fractions might be slightly underestimated for the benefit of the reported PP shares.

Table 4: Melting temperatures of different polymer types [4]

Polymer type	Melting Temperature [°C]
LDPE	105-118
LLDPE	115-125
PP	135-168
PA6	220-225
PET	250-260



5. DATABASE

The mass balances and analytical results gained in Task 3.1 and also reported from Task 2.1 are fed into a database based on Microsoft Excel. In it, all the collected information is gathered to specify the considered waste streams. In the course of the project, the database will be extended by further waste streams. In addition, the treatment of the waste streams in the recycling process cascades will be tracked and streamlined. The database is stored on EMDESK and made accessible and writable for all partners. Its current status on the example of France is showed in Figure 18-20.

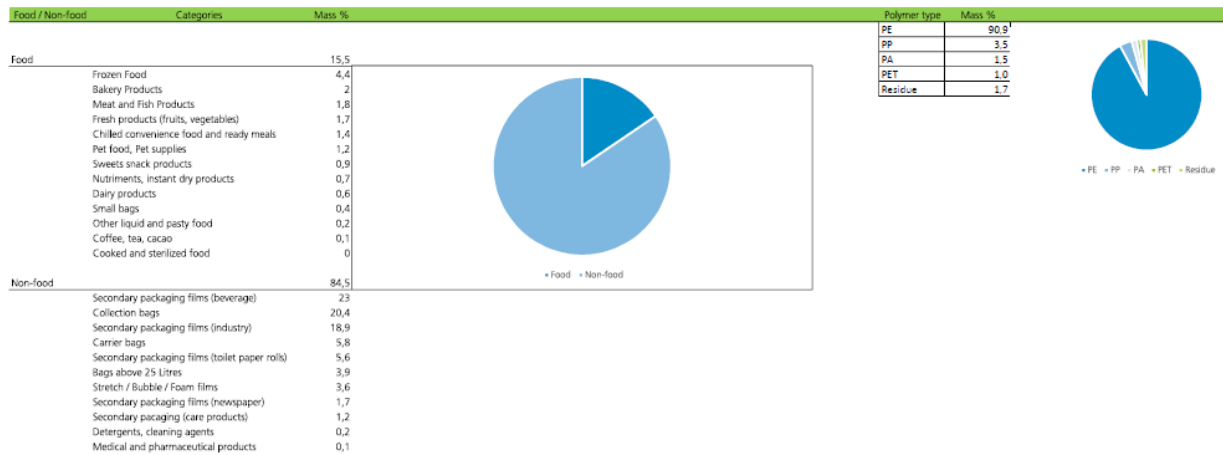


Figure 18: Database (in EMDESK) - Composition of the French flexible packaging

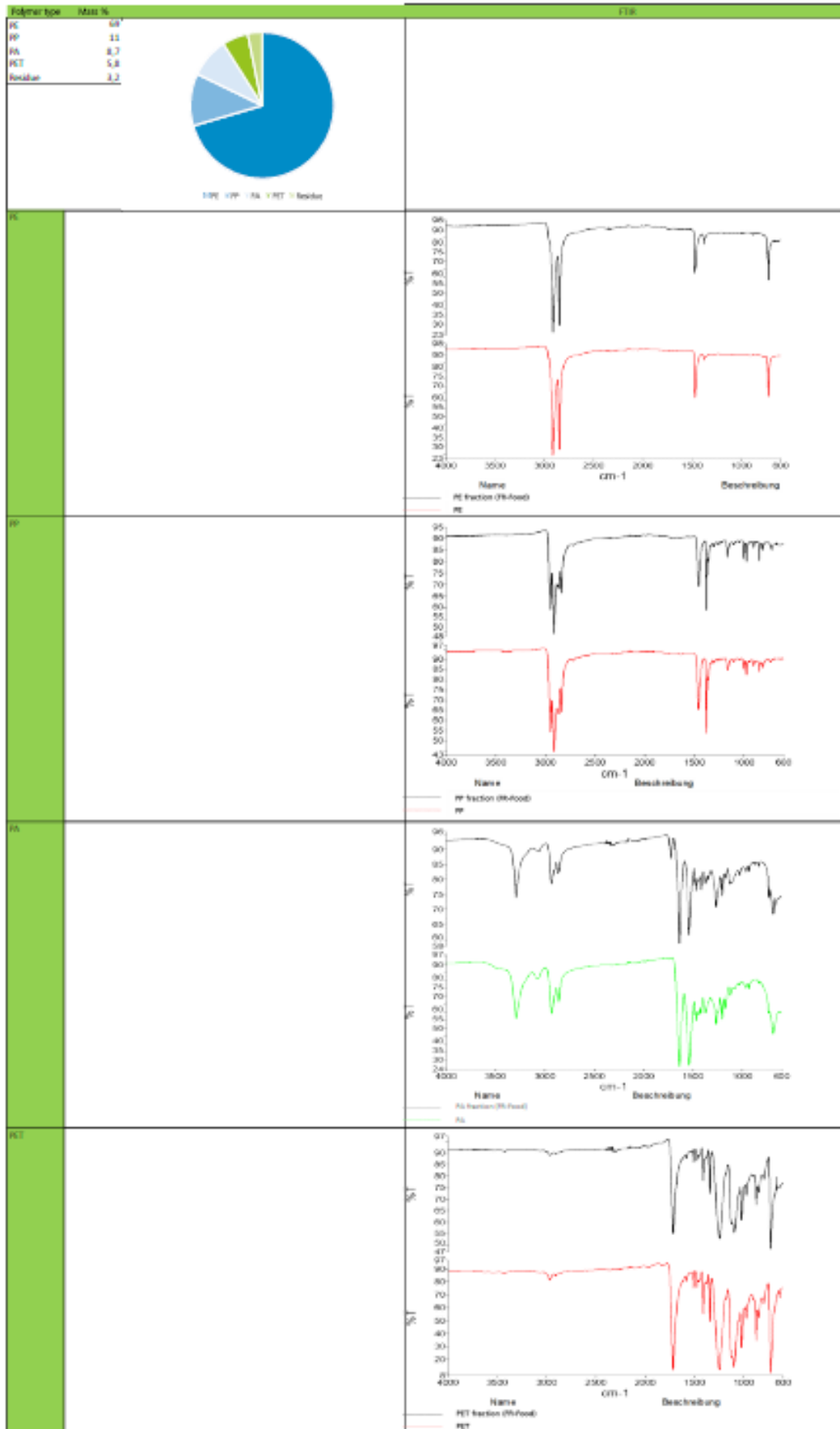


Figure 19: Database (in EMDESK) - Composition of the French Food waste and analysis of the polymer fractions



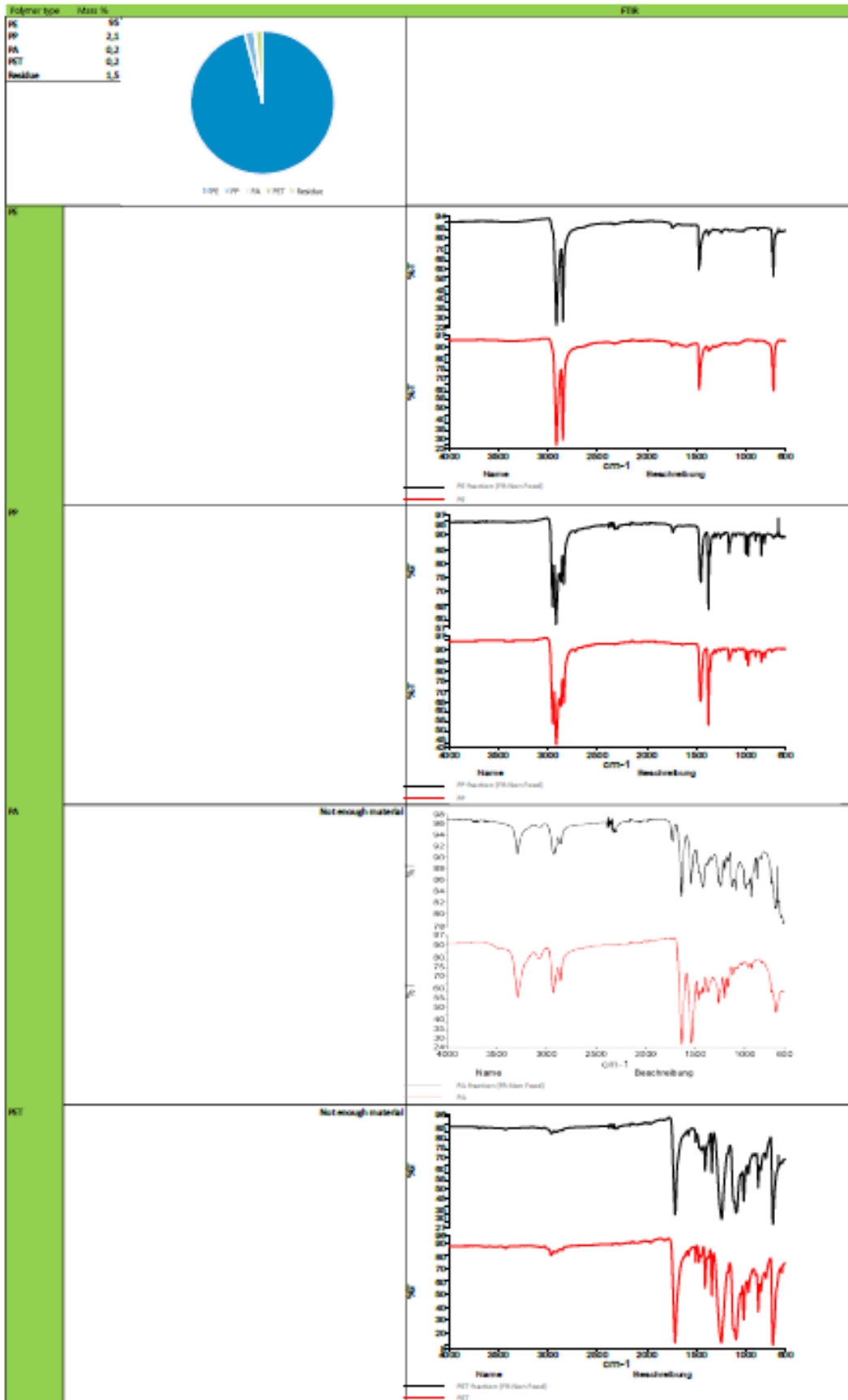


Figure 20: Database (in EMDESK) - Composition of the French Non-food waste and analysis of the polymer fractions

6. CONCLUSION

In Task 3.1 of CIRCULAR FoodPack the polymer composition of PE rich flexible packaging waste from three European countries, France, Belgium and Germany was investigated. The waste fractions were collected and sorted by SUEZ at sorting sites in the three countries and categorized into food and non-food packaging. With specific solvents from the CreaSolv® Process, the polymer types contained in the waste fractions were consecutively dissolved and mass balanced.

It was shown that the PE shares in the sorted fractions differ in a European comparison. The French (91%) and German (89 %) sorting fractions have a higher PE content than the fraction from Belgium (83 %).

Also, the packaging application influences the shares of the contained polymers. Non-food packaging (80-90 %) has a significantly higher PE share than food packaging (60-70 %). This was expected due to higher functionality of food packaging, which currently requires different types of materials.

FTIR and DSC analysis of the separated polymers were used to identify the polymer types and analyse them for purity. It was seen that the polymers could be separated in a high purity. An exception is made by the French and German non-food fractions, where the DSC analysis shows PE residues in the PP fraction.

The results of this Task were fed into a Database available at EMDESK that will be extended in the course of the project with data from other packaging waste fractions provided by the project partners as described in Task 3.1 of WP3. It will support the development of sensor-based and tracer-based sorting technologies and help to organize the recycling of the waste fractions with suitable recycling processes.



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