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PolyCE

Post-Consumer High-tech Recycled Polymers for a Circular Economy

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Contributing Partners

MGG Polymers, UL. KU Leuven, Ecodom

Disclaimer

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Summary

At the current stage of Task 4.3 an industrially applicable method for the determination of the quality of a mixed plastic waste stream including relevant impurities has been developed by MGG Polymers and it has been applied to improve the production of a demonstrator (transparent and white polystyrene parts from fridges, mixed waste from washing machine drums and mixed waste from large household appliances).

A reference analytical method has been developed by KU Leuven to determine the size distribution (successfully tested), the colour composition and the plastic composition with FTIR that can also analyse black plastics.

The metadata definition has started and clarification of the data that can be potentially used starting from the collection on is evaluated by Ecodom.

UL provided a draft for test scheme to deliver quality throughout the vast processing chain from collection to reuse in structured phase gate process.

Content

1 Introduction

The requirements for testing recycled streams have to be studied in 2 different categories. One is the category "granulate" of the recycled polymer in form of granulate that shall be used to mould new articles. The other category "flake" is the quality of the polymer containing waste that is going to be recycled.
These 2 categories differ very much in the applied technologies for analysis and the requirements of quality assurance.
Granulate: The technologies to test polymers that are produced from post-consumer waste (PCR-polymers) are the same as those to test virgin polymers. Basic normed test exist in ISO and ASTM and advanced tests exist in the electronic and in the automotive industry.
For PCR-polymers the frequency of testing is increased, as described for example in the norms from Underwriter Laboratories for the yellow card listing of a non-traceable post-consumer recycled polymer.
Flake: The term "flake" is used here for all kinds of shredded plastic parts of any size and composition. Such shredded waste streams aren't normed and neither are the methods to analyse such a waste stream. Therefore there are also no norms defined on how to determine the quality of a waste that contains plastic and the quality of a plastic waste itself. Due to this lack of norms the shredded waste streams with plastic flakes vary extremely in their properties as for instance shape, composition, impurities.
This variation in waste properties is one of the key points to start to improve the circular supply chain. A reduced variation of waste properties allows to reduce efficiency losses that are caused by adjusting recycling operations to sudden big changes in qualities. The reduced variation of the waste properties also allows to target defined qualities and to improve the "clustering".
Cluster: A cluster is defined at the point of collection. Existing examples for clusters are the WEEE categories as for example large household appliances, small household appliances, IT and telecommunications equipment, etc. The definition of these clusters is based on risk assessments for the resulting waste streams. The cluster "Lightning" for example focuses on the handling of mercury in bulbs, whereas the cluster "large household appliances" focuses on fridges and the removal of ozone depleting coolants, etc. No cluster is defined with regards to polymer recycling. Therefore it is necessary to help waste treatment companies define new clusters to improve the supply chain for polymer recycling and to maintain the polymer value of such a cluster. Typical examples are "food compliance", "transparency", "pollutant free", etc.

1.1 Importance of testing

To maintain the quality of clusters and to bring sufficient material from the waste collector to the polymer recycler and finally to the original equipment manufacturer (OEM), it is necessary to provide quality tools at every stage of this supply chain to the operating waste treatment companies. The waste treatment companies are not specialists for polymers; furthermore the low value of the polymers on average makes them a less focused stream. Therefore it is most important to define quality testing methods that have highly reduced entrance barriers, are easily applied and have low complexity. Every test method shall therefore refer to the value of the stream and help to increase the value of the polymer-containing streams. Every generated information from a performed test shall give the waste treatment operator valuable information about his achievements and provide useful data to the down-stream operator to focus his operations in generating value.

1.2 Phases of testing

"Testing" can be very hands-on and very abstract. The supply chain consists of collection, first treatment and metal recovery before it reaches the polymer sorting.

Collection: Abstract testing is applied at the first stage, i.e. at the stage of WEEE collection. It is important to generate data about the quality of the collected cluster, the so called "Meta-data". There are statistical tests, which describe the quality of such meta-data. Such data could be the degree of "homogeneity" of, for instance, large domestic appliance collection, meaning that cars and washing machines are not collected together for shredding, or the degree of "non-scavenged" for PCs, meaning that at 100 % no mainboards have been removed, or the degree of "transparency visually inspected" and therefore 100 % shows that all transparent plastic parts in the collection bin were controlled visually. Information about depollution (batteries), removal of liquids (oils), packaging removal (wood pellets) can be generated as well at collection points. If such meta-data are generated by statistics, the tests will show highly robust confidence results, while estimated assumptions about the collection will generate results of low confidence.

Volume reduction and first treatment: After collection the usual first treatment step is volume reduction, either by manual dismantling (TVs, lamps, etc.), smashing and pressing (monitor back panels) or shredding (small domestic appliances). When meta-data informs that wood pellets and loud speakers have been removed, low or no content of wood can be expected and no test for wood impurities is required. If the meta-data shows limited confidence for the absence of wood then a test for wood content or wood impurities is required before the flake waste goes to the next process stage. At this stage iron is frequently removed with magnets to protect machines and permanent magnets are collected or lost, as they stick to the iron walls of the machines.

Metal recovery: After the volume reduction, that simultaneously releases the different parts of the electronics (if the degree of miniaturization and integration is not too high), follows the metal recovery. Some metal parts are handpicked (e.g. neodymium magnets) others are removed by density

gradients (aluminium), by sorting with X-rays (alloys), by colour sorting (PCBs), by magnets (iron), etc. Crucial for the production of a valuable waste stream with high amounts of recyclable polymers is the absence of inorganic impurities such as metals, ceramics and glasses.
Therefore the determination of the inorganic content in a flake waste is essential.

1.3 Parameters influences sorting process efficiencies

Particle size distribution
In general, particle sizes smaller than 3mm make the recycling of plastic impossible. Small particle sizes make even the best sorting processes inefficient due to the fact that all sorting processes rely on particle mass and impulse to move that particle mass. There is no difference whether gravitational force is used in a density bath, an air push in a NIR-sorter or the pull in the field of an electrostatic sorter: low masses, and therefore low particle sizes, cannot be controlled with the current state of the art sorting. A particle size range is a basic requirement to describe flakes.
Neutral properties: One of the unsolved issues in polymer sorting are the neutral properties that cannot be detected or determined with any of the existing methods. An example is the presence of phosphate based flame retardants in flakes. Phosphate based flame retardants do not change the physical properties of plastics, but significantly change their quality. Within this work package this issue cannot be addressed. Within PolyCE this point can be addressed in "Design-for-Recycling" and in "CIRCULARITY-APPROVED™".
Impurities: all sorting processes have one main goal, namely to enrich at least one material stream to a defined purity. However, all these processes fail to do one thing: purify. Some sorting steps even may concentrate impurities. For example, density separation with water will generate PP as well as wood. Especially in WEEE, non-plastic impurities are the main source of cross-contamination between the material groups that make up the composition. An important example is the surface contamination with microscopic metal, glass and ceramic splinters from optics or semiconductors. They can mask electrical or spectral properties. Therefore the main parameter that influences the result of a sorting process is the content of impurities. Therefore, the testing for impurities is essential for a well-performed separation.

1.4 Parameters influencing the final PCR quality

Impurities: The main factor determining the final quality of the PCR plastics are the impurities. Micro-particles of glass, ceramic and metal (precious metal nano-wires) are reducing the mechanical performance of PCR polymers, for instance yield and impact strength. Residues of char and tar from wood create unwanted predetermined breaking points in the polymer matrix. Rubber and silicon rubber particles destroy surfaces by forming holes or hills. Trace impurities of PVC reduce the polymer chain length of polyesters (such as PC, PC-ABS) and polyamides (PA) and is generally degrading at all typical processing temperatures of polypropylene (PP), polyethylene (PE), high impact polystyrene (HI-PS), Acrylonitril butadiene styrene (ABS); moreover it frequently contains banned substances as phthalates.

1.5 Parameters to be tested for policy compliance

All PCR-polymers have to comply with REACH when they are sold in the EU market and have to comply with RoHS when they are used again in electronics, as it is planned for the demonstrators in WP7. Therefore all PCR-polymers have to be tested for compliance with REACH and RoHS before they are placed the first time in the market. Additional schemes have to be considered, when the application is demanding additional compliance, as there is the case of applying, for instance, skin contact or food contact polymers.

1.6 Testing schemes adopted for ecolabeling

A general requirement for PCR-polymers is the recycling content. Some PCR-polymers consist of 100 % out of mined waste. Most PCR-materials are compounded with virgin additives or virgin plastics to fulfil the customer needs. Therefore, the PCR-content is one of the properties that has to be communicated. Some testing schemes as BlueAngel include metadata requiring high PCR plastics content.

2 State of the art testing of plastic flakes

This section provides an overview of existing testing techniques, datasheets and how the communication between different actors of the supply chain is done today.

2.1 Colour testing

Colour testing of bigger flakes (min. 15 x 15mm) can be done manually with colour measurements systems as BYK Spectro Guide. Very useful method has been developed by KU Leuven, where every particle is analysed for its composition, size and colour.

2.2 Size testing

The typical method for the determination of the size distribution is a sieve analysis. Alternatively the method from KU Leuven offers a convenient solution for automatic size distribution determination.

2.3 Presence of impurities testing (wood, glass, etc.)

The relevant non-plastic impurities are: fines, rubber, metals, printed circuit boards, glass, concrete, ceramic, wood, foam, glass fibre filled plastics. Some can be detected automatically, like wood, for others no commercial automatic system is available yet. PVC and POM are plastics that are regarded as impurities in a plastic waste.

2.4 Presence of additives testing (such as flame retardants)

There are no commercial methods available to test the presence of all potential additives in plastic flakes in a representative manner. Some additives, like brominated flame retardants, can be tested with ATR, FTIR and Raman spectroscopy at single flakes, but not on an automated representative scale. Pigments, stabilizers and lubricants based on heavy metals can be roughly estimated in a half-quantitative manner over the heavy metal presence with XRF methods. Such methods are not available in automated form to test representative samples and quantities. The only validate method that is able to cover the whole range of potential additives are the HPLC-MS (high pressure liquid chromatography – mass spectrometer) methods. These methods are laboratory intense and not representative due to very small sample sizes. They can only be applied for homogenous samples like polymer granulates.
This lack of analytical methodologies has serious consequences on the control of legal compliance, which is further investigated in the phase-gate process, see chapter 5.

2.5 Food grade testing

Food grade compliance is a process of good manufacturing practice, where a produced formulation is kept clean to avoid cross-contamination. Due to the definition of food grade by law, there is no test procedure that allows to determine the property “food grade”. The testing for the absence of substances that are forbidden for food grade use does not qualify a material as a food grade.

2.6 Plastic quality testing (rubber content, etc.)

Plastic quality testing of flakes is typically done by using a commercially available ATR-spectrometer. Impurities like wood, foams and polybutadiene rubbers can be determined. The development of automatic methods is necessary. The determination of contaminations with metals and other inorganic material need either visual inspection or chemical analysis.

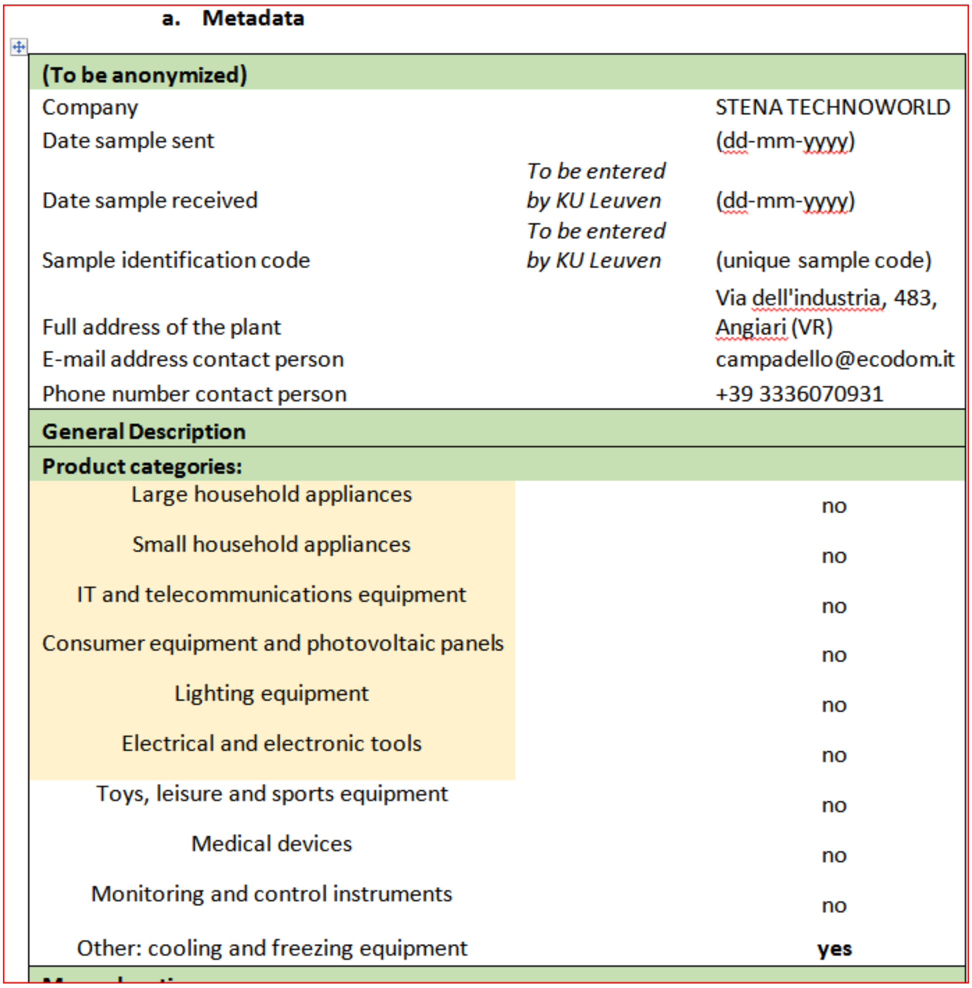
3 Use of metadata to avoid testing

Metadata that are collected along the supply chain are valuable to increase the confidence that the high value of a waste stream is maintained. Very important is quality information about the controlled and well managed processing of the waste clusters. The used processes themselves are not of relevance. Collecting information about technical processes would violate and infringe intellectual property rights of the involved companies. How well collection and waste treatment operations are managed are of much higher value and it is more likely that such information are provided in the down-stream of the value chain.
Of relevance is information about established quality systems as ISO 9001 and 14001, EuCertPlast certifications and collection system audits that extend to the presence and use of cleaning, storage and maintenance procedures, measures against cross contamination etc. For the final granulate it is relevant that the polymers are extruded and moulded at the proper processing conditions and that the polymer has been developed for these processing conditions (s. CIRCULARITY-APPROVED® [(2)]).

3.1 Source of material

The most important information is created at the beginning, at the point of generating a waste stream cluster through collection from the consumer: how well and clean has the cluster been collected? Can consumers throw everything uncontrolled in a bin, or are they advised and instructed what kind of waste has to be placed in what bin or container and has this collection been controlled and corrected? The more reliable a source is, the more likely this material will be fully processed and recovered without tremendously increasing efforts to analyse the quality of the down-stream.
Same down-stream information is relevant: is the value of a cluster maintained? Is a high quality controlled cluster processed without contamination of other waste streams like, for instance, agricultural packaging or automotive waste? Is the storage kept clean and organized and are the materials stored separately? Is the waste treatment site ISO 9001 and ISO 14001 certified?
Such metadata are generated during audits from different types of organisations as EuCertPlast, Ecodom, Lloyd, UL, etc.
The basic information is about the collected WEEE cluster which is defined by the WEEE directive.

Example:



a. Metadata

(To be anonymized)		
Company		STENA TECHNOWORLD
Date sample sent		(dd-mm-yyyy)
Date sample received	*To be entered by KU Leuven*	(dd-mm-yyyy)
Sample identification code	*To be entered by KU Leuven*	(unique sample code)
Full address of the plant		Via dell'industria, 483, Angiari (VR)
E-mail address contact person		campadello@ecodom.it
Phone number contact person		+39 3336070931
General Description		
Product categories:		
Large household appliances		no
Small household appliances		no
IT and telecommunications equipment		no
Consumer equipment and photovoltaic panels		no
Lighting equipment		no
Electrical and electronic tools		no
Toys, leisure and sports equipment		no
Medical devices		no
Monitoring and control instruments		no
Other: cooling and freezing equipment		**yes**

Figure 1: Example of a metadata sheet

3.2 Adopted (recycling) processes

Metadata with high reliability and confidence level allow adopted plastic recycling processes. They increase the confidence that an investment into mining a special cluster pays back. When for instance in the cluster "Lightning equipment" the glass is reliably removed then the resulting transparent lampshades are of much higher value then the glass and allow a simplified process to produce a highly valuable translucent polymer. The same accounts for "food grade". When metadata can prove that the collection is kept clean to avoid any cross-contamination and they repeatedly give the information that a stream is food grade compliant treated, then it is possible to treat this special stream with special operations.

4 Minimum requirement of plastic flakes

The minimum requirements for plastic flakes are defined by the definition of B3010 (s. Federal Waste Management Plan of Ministry of Environment of

Austria[1]) and these requirements are reflected by the analysis report from MGG Polymers as enclosed in the Appendix (p. 36).

5 Innovative testing techniques

5.1 Evaluating the size and colour distribution of mixed plastic recyclates using computer vision

5.1.1 General

Better information about plastic recyclates originating from waste of electrical and electronic equipment (WEEE) will lead to improved closing of the material loops (see work package 3. Task 3.4). Therefore the goal of this WP is to develop an innovative method for automated identification of the size and colour of plastic flakes and to create an information sheet. The method relies on colour images that are assessed using computer vision. Computer vision algorithms to calibrate, filter the background, achieve contour recognition, and determine the pixel colours inside the contour flakes are used for this purpose. Several validation batches have been analysed to evaluate the developed method. The size characterization has been validated by comparing the results of the developed program with sieving tests, using different sieve sizes. The results show that the developed program effectively allows determining the size and colour of plastic flakes and can provide valuable insights for recycling companies to evaluate the value of mixed plastic recyclates.

5.1.2 Setup

5.1.2.1 Photo booth setup

The used method is based on images taken from plastic flakes. To take high quality images it is first of all important that lighting conditions remain constant and repeatable. Therefore, external light on the image is avoided to make sure the results are not influenced by, for example sunlight. Hence a closed photo booth is required. The photo booth is equipped with its own diffused even spread led light source. In the photo booth a uniformly coloured background plate (blue) is placed on which the plastic flakes are spread. The background plate will simplify the process of determining the different plastic flakes present on the pictures. In the photo boot a high quality area camera CMOS sensor of 24 megapixels is used (Canon D750). To analyse a representative sample see as planned in work package 4.4 the background plate will be replaced by a conveyor system with a loading conveyor and a vibration plate at the input to spread the flakes over the belt. To make images of the flakes during the transport over the belt a line scan camera Teledyne Dalsa 2K has been configured. The first tests with the line scan camera are already performed during this task mounted on a robot arm. Results of this test demonstrated significant improvement in the lighting of the flakes, and accordingly in the robustness of the colour analysis.

5.1.2.2 Validation sieves

Nowadays, the separation efficiency of sieving processes for plastic flakes is commonly evaluated by performing small scale manual tests with woven-wire or round hole perforated steel plate sieves. Therefore, sieves are used to validate the program outcome. The sieves are made of plexiglass and cut by a laser. Two of the sieves used have round holes with respectively 10mm and 20mm diameter. To mimic the woven-wire sieve two test sieves are made with square openings with sides of 10 and 20mm respectively.

5.1.3 Method

5.1.3.1 Calibration

The area camera and the lens of the setup need a onetime calibration to determine the intrinsic and extrinsic camera parameters. The intrinsic parameters are used to compensate the distortion caused by the lens. The extrinsic parameters are used to transform the pixel coordinates to world coordinates. This transformation consists of a translation and rotation and correction of the skewing of a pixel. In case that a line camera is used a calibration plate is only used to determine the x and y scale of the image to relate pixel coordinates to world coordinates. The obtained calibration parameters are stored and used every time an image is analysed. Practically the parameters are determined for both cameras by using a circle grid plate.

5.1.3.2 Identification of the different flakes

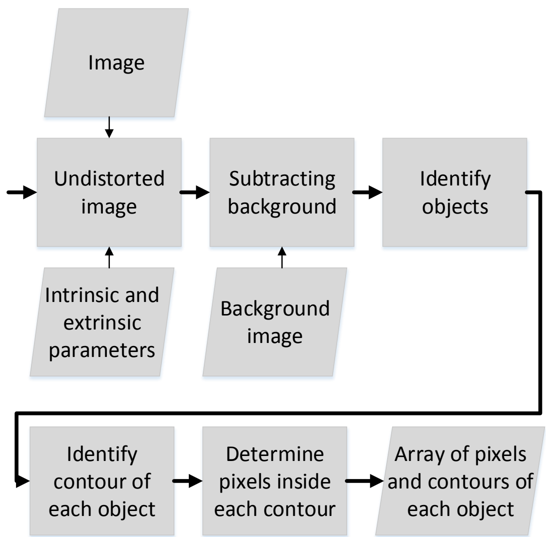


Figure 2. Steps to identify the different flakes

An image is loaded into the program as a matrix where each pixel is an element of the matrix. A plastic flake in the image consists of several pixels. The first step after undistorting the image, as shown in Figure 3, is to determine which pixels belong to which flake.

Figure 3. Undistorted image

For this it is important to remove the background. The use of a smooth coloured background allows the removal of the background colour from the image and retain only the plastic flakes in the image. Since the performance of a simple threshold value is heavily dependent on uniquely lighting conditions, it was decided to subtract an image with only the background from the image with flakes. After the subtracting an image with a black background and coloured flakes is left. The colour of the flakes is heavily influenced, as shown in Figure 4, but this image is only used to find the contour and not the colour.

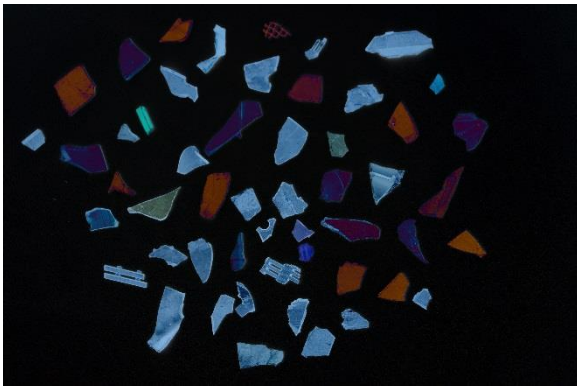


Figure 4. Image after subtraction of the background

The contours are subsequently detected using a Canny edge detection algorithm after noise reduction using a 5x5 Gaussian filter. The result is a binary image, as shown in Figure 5.

Figure 5. Result after Canny edge detection

As shown in Figure 5, there are still gaps in the contours and still there is noise in the image. Consequently, the contours that define the flakes are still not completely closed and defined. Therefore, the subsequent step makes use of the morphological image processing operation erosion and dilates to close the contours. By applying a dilate operation on the image, an extra layer of pixels is added to the contours, see Figure 6.

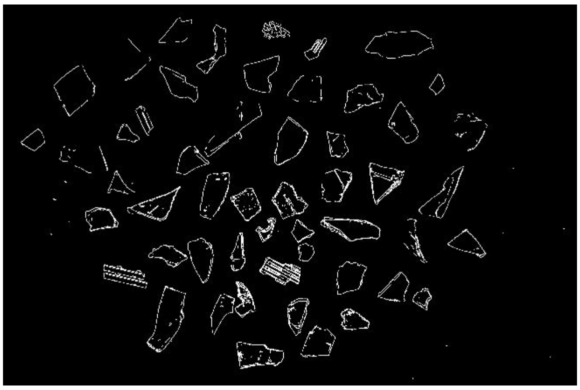


Figure 6. Result after dilate operation

Because the size may not be affected by the dilate operation, the erode operation is applied on the image. The erode operation will strip out the outermost layer of pixels in the contours. The result of this operation is shown in Figure 7.

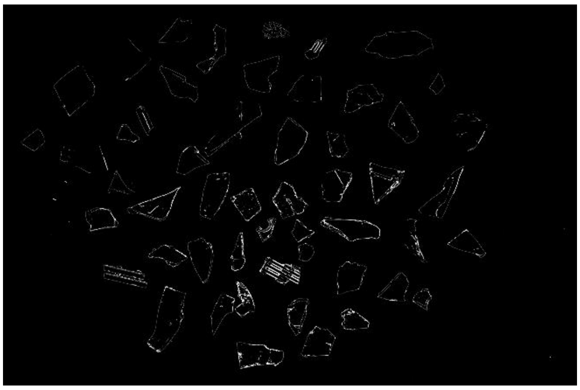


Figure 7. Result after erode operation

Finally, all contours are drawn around the almost closed flakes. The better the preparation of the image is performed the more the contours will approach the real shape of the flakes.

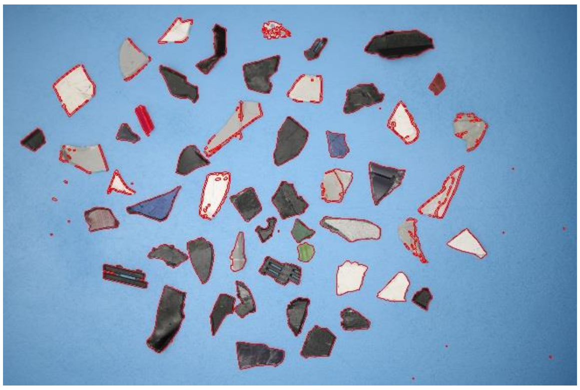


Figure 8. Contours around flakes

5.1.3.3 Determining size

To determine the smallest dimension a minimum area rectangle is calculated around each contour, and the width of that rectangle defines the smallest dimension of the contour that represents a flake, see Figure 9.

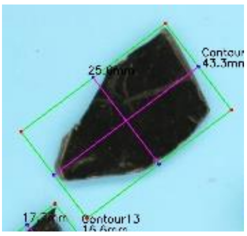


Figure 9. Bounding boxes around flake

5.1.3.4 Determining mass

Since recycling companies are not interested in the number of flakes that can be recycled by their recycling processes, but in the mass based recycling efficiency it is crucial to estimate the mass of the flakes. Therefore, the area of the flakes is determined based on the analysed contour and by counting the number of pixels inside this contour. To optimize computing time, the minimum area rectangle is isolated from the image for this calculation. The area of one pixel can be calculated by using the extrinsic parameters (1).
Once the area of the flake is determined, an estimation of the volume is calculated (2). The thickness of all flakes is obviously not the same, but the thickness of WEEE plastics was in first instance assumed to be independent of the shape or colour of the plastic. Hence, an average thickness was first determined from random measured plastic flakes from WEEE. Based on the density and the volume, the mass is then estimated (3). The density used for these calculations is based on the density of the average composition of plastic from WEEE.
To measure the thickness of the flake instead of using an average thickness, a line triangulation camera LMI 2340 has been installed and configured for further testing of the benefits of improved thickness measurements in task 4.4, as it is expected that thickness information will increase the accuracy of the mass determination.

$$Area_{1pixel} * NumberOfPixels_{1object} = Area_{object} \ (1)$$
$$Area_{object} * t_{avg} = Volume_{object} \ (2)$$
$$Volume_{object} * Density_{avg} = Mass_{object} \ (3)$$

5.1.3.5 Determining colour

While scanning all the pixels to determine the area, the RGB pixel value of the original image of each pixel inside the contour is stored. The RGB values are useful because they can be matched with industrial RAL values. However, the analysed RGB colour is strongly influenced by the camera and light settings. Therefore, a library of the definition of the RAL values in RGB values has been defined for the used setup. Beside the colour an indication of the lightness is calculate out of the RGB value. For each base colour (RGB) the lightness percentage is calculated. To determine the lightness the average is taken from the minimum and maximum value of the Red, Green and Blue value in %. Depending on the lightness it is first determined whether the plastic can be categorised as white, light grey, coloured, dark grey or dark plastic, as shown in Figure 10.

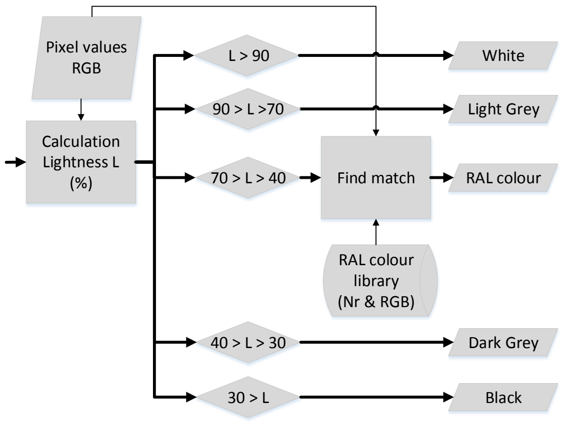


Figure 10. Flowchart to determine colour and lightness

5.1.4 Results & discussion

5.1.4.1 Variation in function of the position of the plastic flake

For high accuracy analysis the result of the computer vision should be the same independently of the position of the flake under the lens. To investigate the external influences of the position and the non-uniformly distributed light a reference flake with a specific dimension and RAL colour is moved according to the red lines depicted in a quarter image shown in Figure 11. The average variations in measured size compared to the size measured in the center are plotted next to the lines. Based on this analysis it can be concluded that the dimensional variation is small and the size is determined accurately independent of the position under the lens. Analyzing the lightness has more influence but can easily be solved by applying a nice uniform illumination.
Because in a later stage a line scan camera is used instead of area camera, a collimated line light decreases the lightness variation on the images.

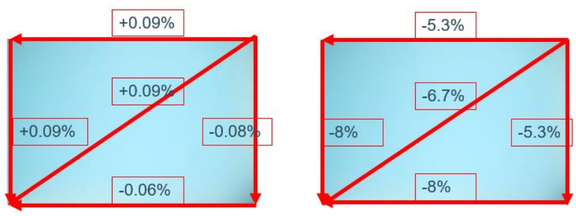


Figure 11. Dimensional Variation (Left) and lightness variation (right)

5.1.4.2 Accuracy of the mass fraction

To validate the calculated mass fraction, 135 flakes are weighed manually and analyzed by the program. The result of this analysis, shown in Figure 12, indicates that the calculated weight is slightly deviating. Nevertheless the error remains below 1wt%.

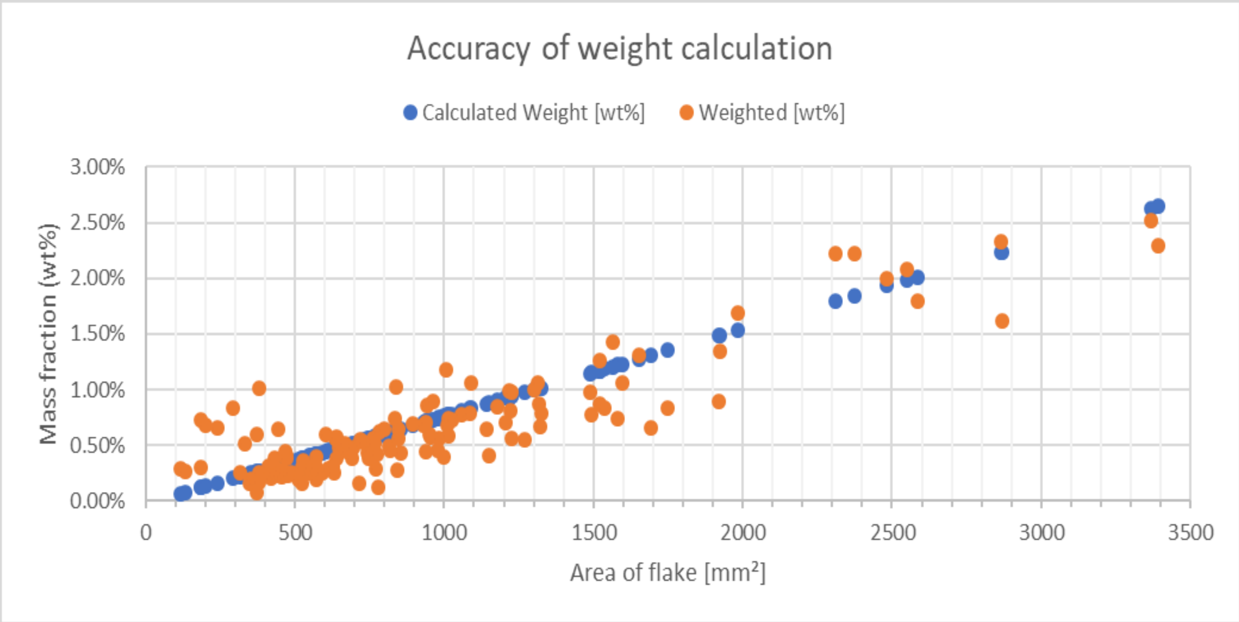


Figure 12: Accuracy of weight calculation

5.1.4.3 Validating the size distribution analysis by the use of sieves

A representative sample of flakes is first analysed by the program. Afterwards, the computer vision result is compared with sorting results. For this the sample is first manually sieved during a period of 6 minutes with the largest sieve. After manually counting and verifying both output fractions, the smallest fraction is sieved once more with the smallest sieve. Again the throughput is counted and verified. This test is both performed with the round holes and the square holes. Based on the results of these tests it can be concluded that the program and the sieving tests had a slightly different outcome, which was to be expected, as plastic flakes with a size close to the hole size most of the time didn't go through the sieve. Accordingly, when using the results of the by computer vision analysed size distribution for making projections on the sorting efficiency, this should be considered by using a slightly smaller size for the calculations than the actual sieve size.

5.1.4.4 Conclusion

The presented results demonstrate that the developed method allows to determine the size and the colour of manual plastic flakes spread out on a blue plate in a photo booth with diffused and uniform lighting.
To communicate the calculated size and colour distribution of the mixed plastic flakes the use of graphs with a curve showing the cumulative mass percentage and stacked bar charts presenting the mass percentage per colour per size category is proposed. An example of these graphs is displayed in

Figure 13 and Figure 14. This way of displaying the result makes it easy to read the expected outcome of using a specific sorting method, as the sieve size can be read from the x-axis and by drawing a line towards the curve and back to the y-axis the expected separation efficiency can easily be calculated for a broad variety of sieve sizes. By using the information and curves on the information sheet, the shredder machine outcome can be evaluated, compared and optimized and the most suitable sorting technique can be selected. The colour distribution curve can be used to estimate the colour outcome and/or the required type and amount of additives to achieve the desired colour after compounding. The testing procedure is ready to determine the colour and size distribution on a larger scale after building a small conveyor system. A line camera has been configured and tested and the images captured are analyzed by the program to generate automatically the reports. At this stage the impurities are still determined manually. The reason is that for the determination of the concentration of impurities no classic computer vision algorithms can be used due to the large variation in the shape, colour and texture of these impurities. The only option considered valuable for the detection of presence of these impurities is the use of artificial intelligent algorithm, such as neural networks. Unfortunately the number of images captured during the development phase and the data collected is too little to be able to properly train a neural network. Therefore, substantial experience with training neural networks was already acquired within this task and the required software has been prepared for training such neural networks, more specific a convolutional neural network named YOLO (you only look once). When there is sufficient data available from the tests that will be performed as part of task 4.4, test will be performed to analyze the impurities automatically by such-algorithms and the performance in terms of speed of detection and accuracy will be evaluated.

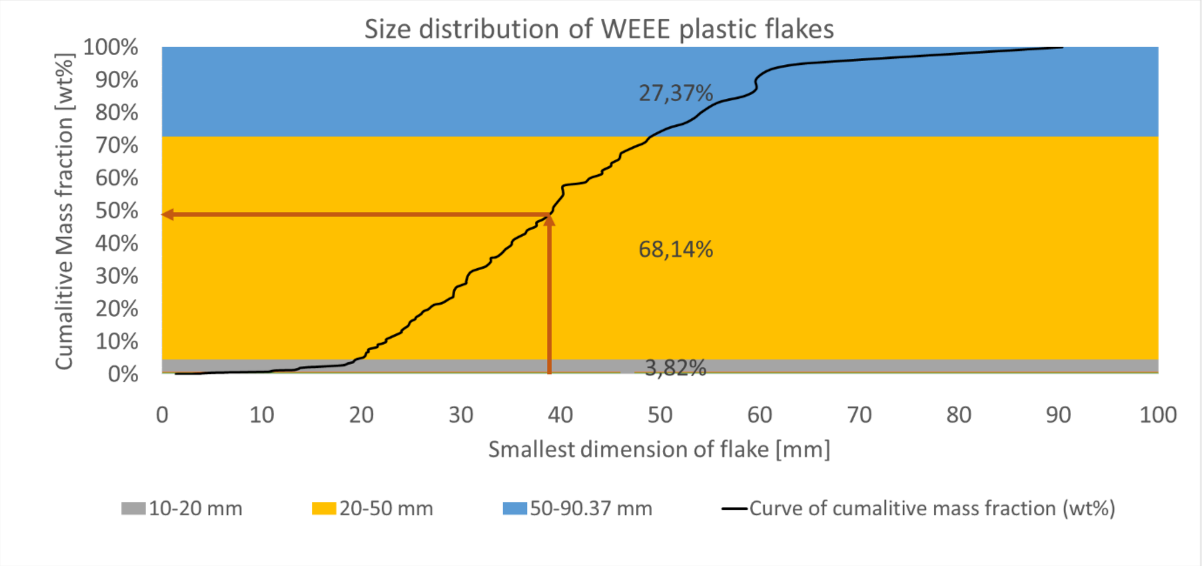


Figure 13: Size distribution example

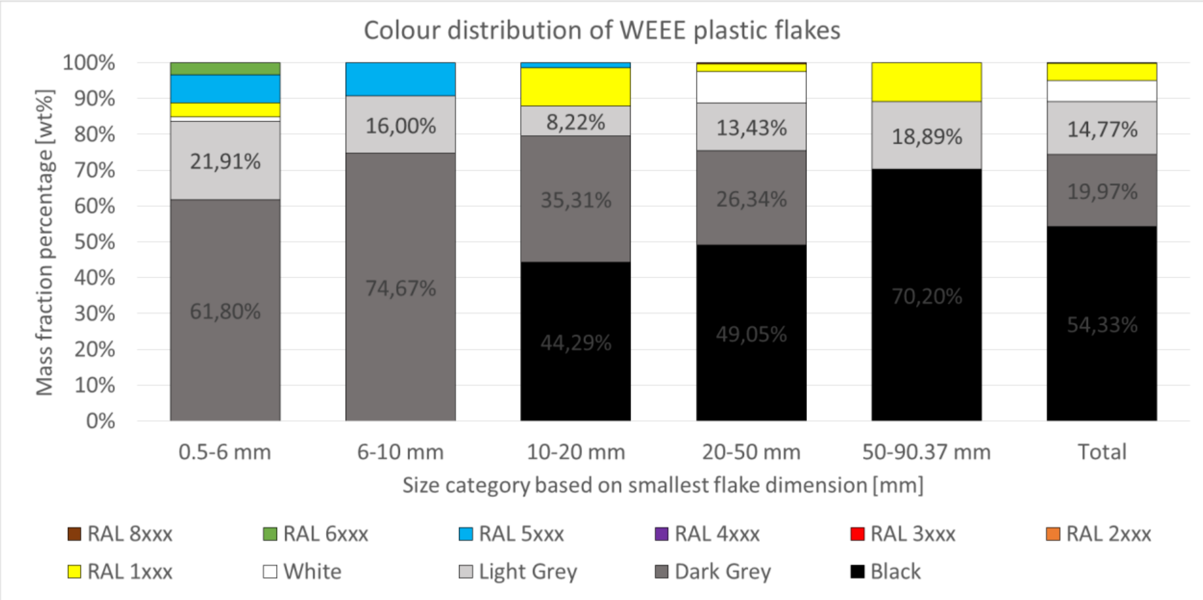


Figure 14: Colour distribution example

5.2 Testing scheme for the plastic composition and quality

Main influences on the quality of recycled plastics are the purity or presence of impurities, degradation that may occur during the lifetime of a product or processing as well as the variation of many plastic grades with different properties and present additives that are being processed together. Quality assessment techniques for plastic flake testing need to capture main characteristics in a rapid and economical manner. Several spectroscopic techniques are available that can determine the plastics type and the presence of brominated or phosphorous flame retardants:

- Fourier Transform Infra-Red (FTIR) spectroscopy measures the plastics response to infrared light. The incident light causes characteristic vibrations based on the chemical structure that are visible due to the absorption or transmission of the incident light. In contrast to NIR, the mid infrared range is used which allows to identify also black plastics. This technique is commonly used for quality management as it allows a reliable identification of the plastic type. On industrial scale for post-shredder sorting, this technique is not used as it is considered too slow.
- Raman spectroscopy works with the principle of the Raman light scattering effect and delivers spectra similar to FTIR. The technique is seen as complimentary to FTIR as it is more sensitive to the detection of e.g. C-C, C=C and C ≡C bonds where FTIR is more sensitive to the detection of OH bonds. According to Florestan et al. (3) the technique can also deliver information about mineral fillers in plastics under the right conditions. This technique is in specific cases also used for industrial scale post-shredder plastic sorting.

- X-Ray Transmission (XRT) analyses the extent to which X-rays are absorbed by a material and determine the atomic density. XRT is capable of differentiating between FR and non-FR plastics, however, due to overlapping densities no distinction between Br and P based FRs can be made. This technique is only in exceptional cases used for plastic sorting due to its limited applicability.
- X-Ray Fluorescence (XRF) on the other hand uses X-rays to excite the atoms of a material in order to create a characteristic fluorescence light response to identify the different elements that are present. This technique allows to identify Br, however the quantification is known to be difficult and P cannot be detected with sufficient accuracy in a polymer matrix. Hence, this technique is commonly used for metal sorting and only in specific cases to sort out brominated flame retardant plastics.
- Sliding Spark Spectroscopy (SSSP) uses a high voltage spark to vaporize a small amount of the plastic surface. The emitted radiation can be measured to differentiate Br and P based FRs. The determination of the plastic type is also possible, but a testing series performed by the authors in 2014 delivered unreliable results for the distinction between ABS and HIPS. This technique is rather slow and requires a contact measurement and can therefore only be used for manual sorting.
- Laser Induced Breakdown Spectroscopy (LIBS) uses a high energetic pulse laser beam to form plasma and vaporize a small amount of the material surface. Excited atoms emit light and elements present in the surface can be identified. The technique allows the identification of several plastic types and the detection of Br and P FRs. However, an excellent spectral library as well as nitrogen conditions is necessary to identify certain polymer types such as PC/ABS. This technique is significantly more costly compared to FTIR and is less stable. Since it also requires a contact measurement and is rather slow (multiple seconds) it is not used for the post-shredder sorting of plastics on an industrial scale.

Most of the discussed spectroscopic techniques are available as table top systems or robust handheld scanners that are suitable for on-site measurements. These technologies are designed to be robust in order to withstand constant transport and the use in manufacturing environments. The applicability of the described plastic and FR analysis techniques is summarized in Table 1 with selected plastic and flame retardant types commonly found in WEEE.

Table 1: Plastic and FR types can be differentiated with the different plastic analysis techniques (Y: can be identified, N: cannot be identified, *Excellent library required)

	PC/ ABS	HIPS	ABS	PFR	BrFR
FTIR	Y	Y	Y	N/Y*	N/Y*
SSSP	N	N	N	Y	Y
LIBS	N/Y*	Y	Y	Y	Y
XRF	N	N	N	N	Y
XRT	N	N	N	Only FR or non-FR	
NIR	Black=N / coloured=Y			N	N

5.2.1 Fourier Transform Infrared Spectroscopy

FTIR spectroscopy is considered a highly reliable technique and is suggested as the preferable technique for the determination of the plastic type of unknown samples. The plastic composition of WEEE plastics that are commonly found in black and grey shades can be measured with this technique. Portable, handheld options are available on the market, they are easy to use and can deliver good measurement results. However, the options in the top of the table are more commonly used in laboratories for quality management, as they are considered to have a better quality and reliability in the measurement results. To obtain the spectra a sample preparation is needed in order to make sure the sample makes good contact with the measurement crystal. In Figure 15, small shredded plastic flakes are shown that can be used for composition analysis in FTIR.

Figure 15: Shredded plastic pieces for plastic composition analysis.

Shredded fractions from WEEE contain a broad mix of different materials such as metals, plastics, glass, rubbers, wood, cables and many more. Metals and glass for instance cannot be sufficiently identified with this technique and the full characterization of a WEEE fraction composition therefore requires further analysis techniques. In Figure 16 the composition analysis of a highly complex shredded WEEE samples is shown. The material composition given in the table (see Figure 16) is based on manual analysis. The list contains the most important materials worth to be analysed based on the experience of MGG Polymers. Future tests will also contain wood as an important impurity (which was not present in the analysed fraction). The plastic composition is shown as a pie chart based on polymer types. For a reliable analysis result a representative sample is crucial, which can be achieved by dedicated sample taking techniques. In addition, large amounts of measurements are needed. While one measurement takes only a few seconds, the sample preparation and placement on the measurement crystal requires 5-10 min per analysis, so it is labour intensive if large amounts of measurements are required. Therefore, automated FTIR has been developed, is further fine-tuned and adapted to fit the requirements of shredded WEEE samples.

Sample Composition	[%]
Metals	10,68
Plastics	27,04
Glass	0,00
Rubbers	1,05
Cables	33,52
Others	27,71

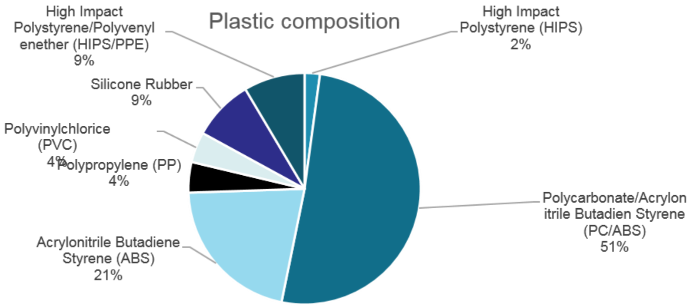


Figure 16. Plastic composition graph based on the analysis of a mixed plastic flakes sample.

5.2.2 Advanced FTIR analysis

FTIR is a powerful technique that is capable of generating valuable information about the chemical structure of a polymer that goes beyond the pure identification of a plastic type. Future research will investigate the possible advanced characterisation options that are possible with FTIR and evaluate the suitability and value of this information in the context of plastics recycling. Some important influences on the quality of a recycled plastic have been summarized:

- Impurities

Some mayor impurities such as other plastic types or wood can be measured with FTIR and give a good indication at flake level. The table below shows an overview of different impurities and the possibilities and boundaries of FTIR to detect them.

Table 2. Overview of impurities and the their detectability with FTIR

Impurities that influence the quality of recycled plastics	
Can be detected by FTIR	**Cannot be detected by FTIR**
Other plastics	Metals
Wood	Glass
high concentrations of organic pollutants	Trace impurities
High concentrations of flame retardants	RoHS and REACH levels of flame retardants

- Degraded content

FTIR allows to measure the chemical degradation in the plastics that may occur during the lifetime of the plastic or the processing. Oxidation groups as well as rubber content in engineering plastics can be characterized and give an indication of the quality of the plastic flakes. Potential highly degraded sources can be identified and avoided in order to increase the quality of recycled plastics. Studies show that oxidation of ABS and a decrease of the polybutadiene content are significant chemical ageing processes that may occur in this material and can strongly affect the mechanical properties. Figure 17 shows the results of the analysis of oxidative groups identification of polystyrene samples before and after Corona treatment. In this case the oxidative groups were created by Corona treatment to increase the adhesive strength of a plastic surface.

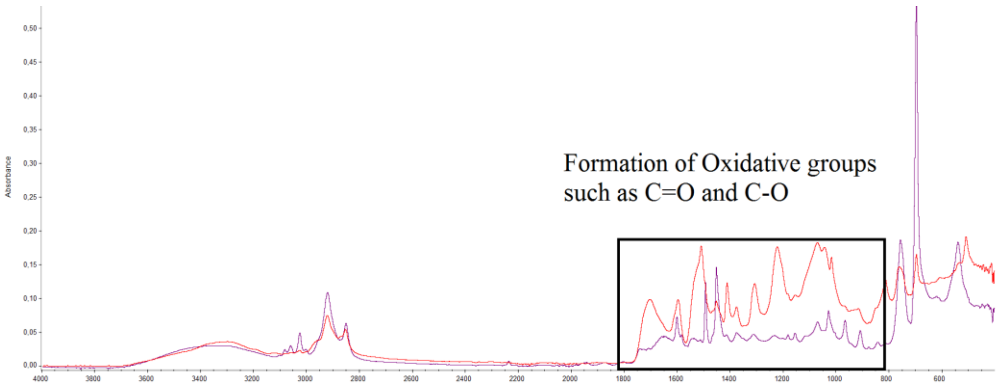


Figure 17: FTIR measurement of oxidative groups in plastic flakes.

- Plastic grade composition

Many different plastic grades are produced for every plastic type such as ABS. The properties of the material can be adjusted to the needs of the component manufacturer by the modification of the chemical structure or the integration of additives in the polymer matrix, as shown in Figure 18. As a result there are many different grades with various properties present in electronic products.

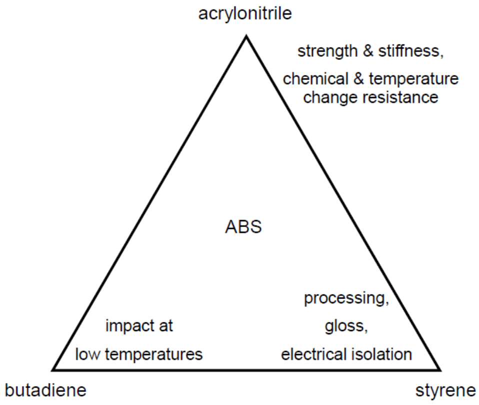


Figure 18. ABS copolymer composition triangle (presentation Close WEEE project, Dr Arne Rüdiger, Sitraplas).

FTIR allows the determination of a plastic composition by the identification of certain chemical groups that can be quantified. In Figure 19 a FTIR spectrum of high impact polystyrene is shown. The peak associated to the rubber phase can be identified and quantified. While the quantification is subject to limitations it could give an indication of composition differences, which can result in property variations of the final recycled material output if not buffered accordingly.

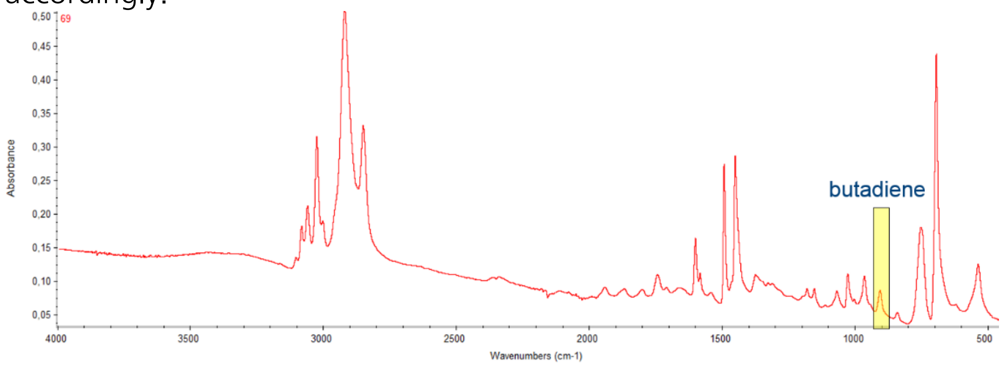


Figure 19. FTIR spectrum of polystyrene with a butadiene phase for impact strength improvement.

5.3 Testing scheme for plastic flake testing with computer vision and FTIR spectroscopy

A systematic analysis scheme for plastic flakes was developed that includes the use of metadata, sieving, sample taking, computer vision analysis of colour and size distribution and the plastic composition analysis with FTIR. The metadata incorporates valuable information of upstream processing steps, processes and companies involved.

For representative sample taking the work of Pierre Gy (4-9) gives a good overview of many different sampling options dedicated to different situations, which are adopted in the developed testing scheme.

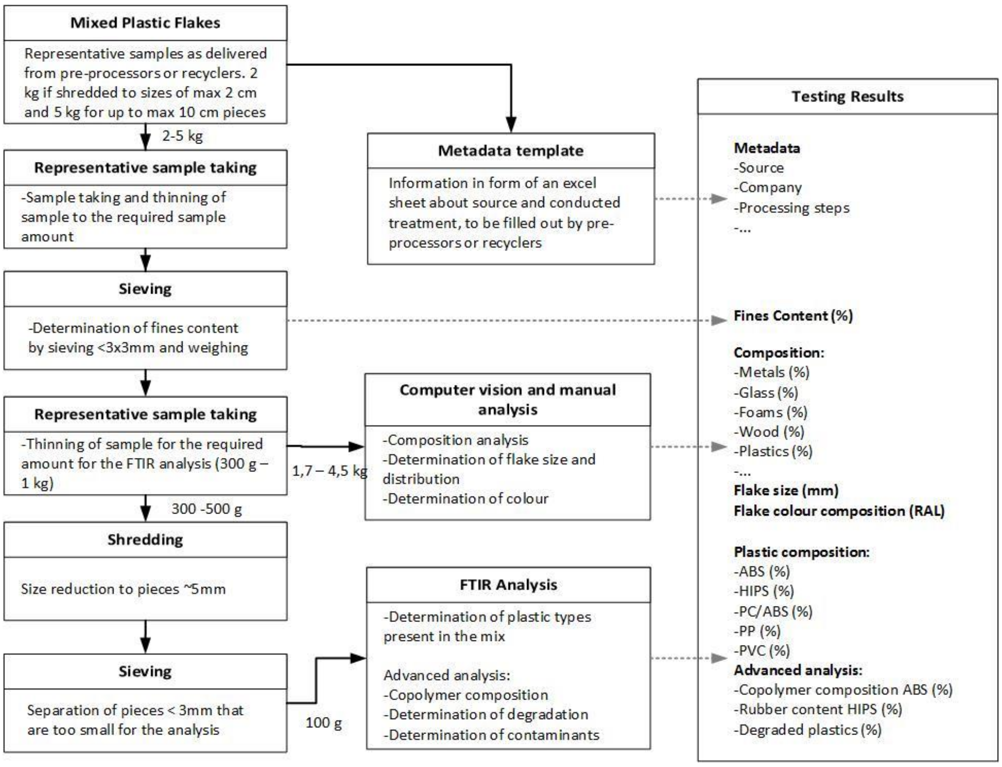


Figure 20. Testing scheme for plastic flakes.

The achieved testing results from the metadata questionnaire, the composition analysis by manual sampling, size and colour results from computer vision analysis and the plastic composition from FTIR spectroscopy is summarized in a graphical datasheet. A proposal for the communication of relevant information obtained from the proposed analysis scheme for plastic flakes is shown in Figure 21. The test reports created for other fractions analysed to evaluate the applicability of the developed testing scheme can be found in the Appendix.

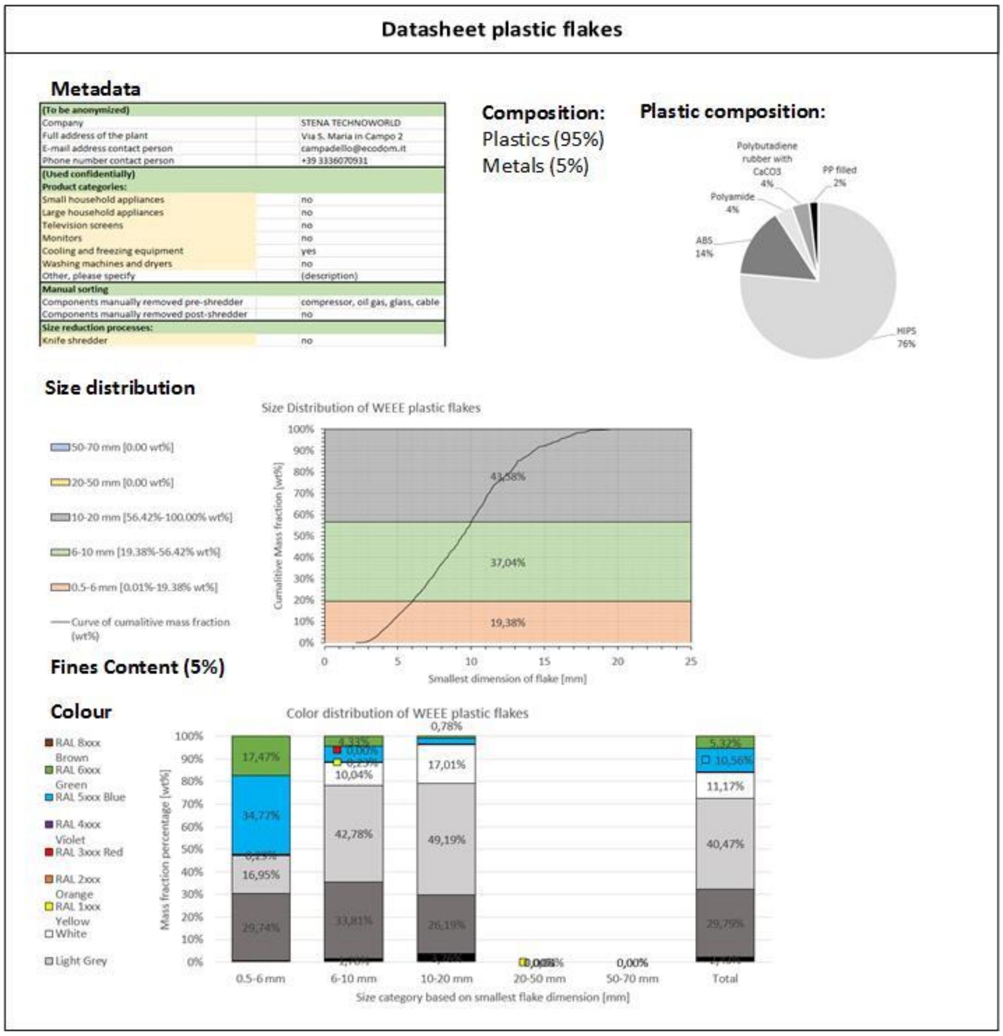


Figure 21: Graphical datasheet of the results from plastic flakes testing.

In the developed testing scheme the detection of flame retardants is not possible. Based on the performed literature research and experiments, the detection in a fast and economically viable manner with sufficient accuracy to confirm RoHS and REACH compliance is found to be not technically feasible. Therefore, a phase-gate strategy has been developed, that allows to estimate risks related to the presence of additives and impurities based on available data at the different actors of the plastic value chain. In addition, future research will investigate the feasibility of integration of an XRF measurement system in the automated setup that has been constructed to perform FTIR analysis, to amongst others allow measurements of bromine content and to provide valuable information on the feasibility of achieving RoHS and REACH compliance.

6 Phase-Gate quality management scheme

The Phase-Gate approach is patterned off an approach to managing the steps in product design. Before accepting materials for the next phase in processing the material has to meet certain properties or characteristics which are determined by the final use. The goal is to increase confidence in the quality of the material, assure compliance of the material with end requirements and to do so with a minimum of additional cost. For example an early gate might require the absence of brominated flame retardants. Once a BFR free batch of material is created and if such material is kept separate in further processing there is no need for further BFR testing. Requirements are passed from the users of the material up the supply chain and the gate requirement is set between the phases where meeting the user specification is the most effective and introduces the least cost. Information about properties of that material travels with batches and is sent down the supply chain to create a folio of test/gate results for the final material.

Figure 22 depicts the phases of plastic recycling in the supply chain as agreed to by PolyCE members. Each phase represents operations performed on the plastic while the gate is the set of requirements which the plastic has to meet before being accepted in the following phase. In practice the gates are between the phases and not passing a gate could disqualify the batch of material for the intended purpose.

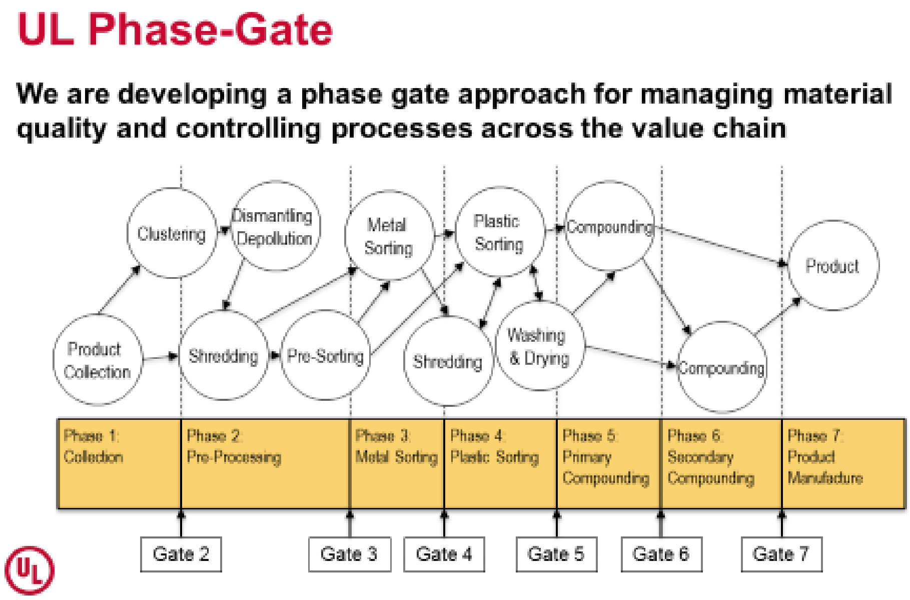


Figure 22. Phases of plastic recycling in the supply chain

6.1 Opportunities for simplification of certification for PCR

Currently PolyCE is focusing on two broad end use requirements, RoHS/REACH compliant and food grade plastics. Each of these has a set of requirements for the final material, some of which cannot be determined by a test of the end product.

Today testing for RoHS or REACH compliance may take place at multiple points in the supply chain with the potential for little or no communication of the testing status among players in the supply chain. As a result testing may be duplicated, increasing cost to the supply chain.

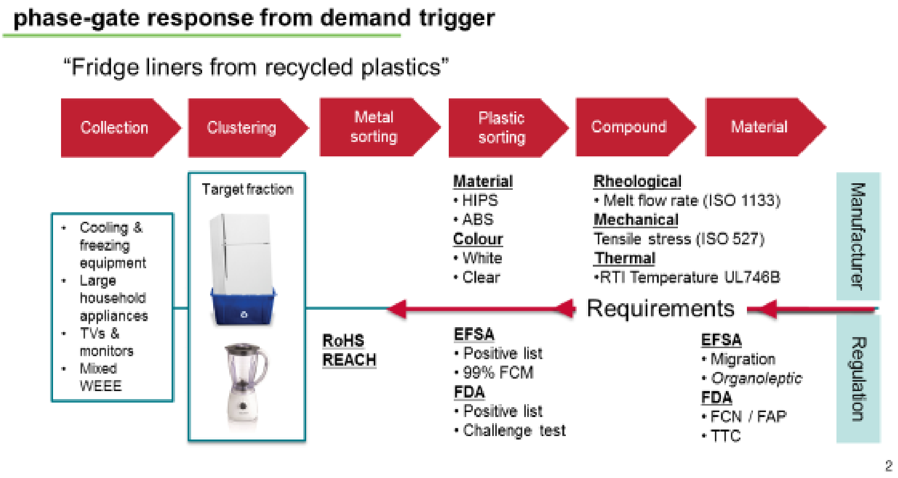


Figure 23. Phase-gate response from demand trigger with fridge liners as example

6.2 Proposed phase-gate scheme

At this point in the project UL has begun to apply the requirements for polymer granulates gathered in WP4, Task 4.1 and apply those at the appropriate place in the supply chain. Ideally formulations and substances that are not REACH and RoHS compliant should be separated out as early as possible in the supply chain. The premises of the WEEE directive are a good minimum requirement for the gates 2 and 3, i.e. to remove mercury bulbs, cadmium batteries, capacitors, lead glass, etc. The earlier absence of impurities and REACH compliance are achieved the more valuable a waste stream is. It has to be made clear that to achieve this goal, special design requirements must be met, as for example concentrating toxic parts in one easily separable module (s. Task 8.1 Eco-design). Until Gate 6 a recycled plastic must be REACH and RoHS compliant in form of granulates for the use in electronic articles.
Once waste stream has met requirements no further testing is required before use in the demonstrators if the material is kept separate in the supply chain. Requirements from manufacturers flow upstream to the source of materials and are used to define the gate requirements. Information about processing quality and testing passes downstream to the product such that when finally used in the product the material has a complete folio of quality and testing history. The appropriate application of testing at the appropriate phases helps to assure and document compliance with the manufacturers' requirements

(including REACH and RoHS). Details of the parameters and gate requirements are in the appendix.

Table 3. Test requirements for polymer granulates

		Gate						
		1	2	3	4	5	6	7
Test Name	Test Standard	Collection	Processing	Metal Sorting	Sorting	Compoundin g	Compoundin g	Manufacturin g
Rheological								
Melt Flow Rate	ISO 1133							
Shrinkage at Production	ISO 294-4							
Mechanical								
Tensile stress at Break	ISO 527-2/50							
Tensile stress at yield	ISO 527-2							
Tensile Strain at Yield	ISO 527-2/50							
Tensile Strain at Break	ISO 527-2/50							
Flexural Modulus (23°C)	ISO 178							
Tensile Modulus (23°C)	ISO 527-2/1							
Tensile Strength (23°C)	ISO 527-2/50							
Charpy Unnotched (23°C)	ISO 179-1eU							
Charpy Notched (23°C)	ISO 179-1eA							
Thermal								
Heat Deflection Temperature	ISO 75-2B							
RTI temperature	UL746B							
Vicat Softening Point	ISO 306/A50							
Flammability Rating	UL94							
Electrical								
Other								
Recycled Content	EN 15343							
Colour	ISO 11664							
Gloss	ISO 2813,							
Density	ISO 1183							
Filler content	ISO 3541-4							

Residual Humidity	EN 12099							
REACH Compliance								
RoHS Compliant	201165EU							
Target Fraction								
Purity/Fraction concentrations								
UL registered	Yellow Card							
Food contact approval	EC10/2011							
Management System	ISO 9000							
QC	QAS EC 2023/2006							
Inventory Control/Shipping								
Folio/Dossier Prepared								
visual inspection for contamination	Sorting Effectivenes s							
	Chemical/St ain							
Chemicals Used during processing	Food Safe or removed							
Degradation during processing	max Temp							
Recycling Process(es)	EN15343							
leaching test with simulants -	regulation No10/2011							
Challenge Test History (Control Charts)								

Legend: Green requirements proposed, orange TBD.

7 Conclusions and future work

- Pre-Processing analysis:

Up to date MGG Polymers developed an analytical report that focuses on the target plastic content, which ranges from 1 to 5 plastics, and the relevant impurities that reduce the value of a raw material.

This report can be used to provide immediate information to the supplier (up-stream), so the pre-processors have continuous information to improve their quality.

- Reference Analysis

The reference analysis from KU Leuven shows already very useful results for the size distribution. The colour distribution is useful but has to be related to the size distribution.
The automated FTIR analysis has very strong potentials and the robustness of this method has to be proved. The automated determination of impurities has to be developed.

- Meta-data

The metadata need to be set up and verified in relation to audits and self-assessments related to operational quality and reliability.
Meta-data needs to be collected for each processing procedure and input material (e.g. mechanical treatment of fridge drawers). It will constitute the information to be passed to the subsequent operator together with the confirmation that the processing procedures follows the ones used when the meta-data were collected. Further investigation needs to be performed about the minimum information of the meta-data that can be considered reliable among time (e.g. colour of the flakes can vary during time considering it depends on the input material).

- Phase-gate Quality Scheme

The phase-gate quality scheme has to be tested together with all participants, including pre-processors (from Ecodom) to evaluate the acceptance, the robustness and the applicability.
A very important point for the acceptance is the costs of the quality scheme.
Here the creativity and effectiveness of PolyCE team will be challenged as we continue to develop the scheme, in particular it will be defined the way in which the information will be transferred from one operator to another (e.g. by a declaration, by an excel sheet, by a paper copy attached to the delivery note,…).

8 References

(1) Federal waste management plan 2017 part 2, p.204 – 210
(2) Circularity-approved® is a trademark registered by MGG Polymers and developed by MGG Polymers under Horizon 2020, Grant 730308, PolyCE
(3) J. Florestan, A. Lachambre, N. Mermilliod, J. C. Boulou, and C. Marfisi, "Recycling of plastics: Automatic identification of polymers by spectroscopic methods," Resour. Conserv. Recycl., vol. 10, no. 1, pp. 67–74, Apr. 1994.
(4) P. Gy, "Sampling of discrete materials—a new introduction to the theory of sampling: I. Qualitative approach," Chemom. Intell. Lab. Syst., vol. 74, no. 1, pp. 7–24, Nov. 2004.
(5) P. Gy, "Sampling of discrete materials: II. Quantitative approach—sampling of zero-dimensional objects," Chemom. Intell. Lab. Syst., vol. 74, no. 1, pp. 25–38, Nov. 2004.
(6) P. Gy, "Sampling of discrete materials: III. Quantitative approach—sampling of one-dimensional objects," Chemom. Intell. Lab. Syst., vol. 74, no. 1, pp. 39–47, Nov. 2004.
(7) P. Gy, "Part IV: 50 years of sampling theory—a personal history," Chemom. Intell. Lab. Syst., vol. 74, no. 1, pp. 49–60, Nov. 2004.
(8) P. Gy, "Part V: Annotated literature compilation of Pierre Gy," Chemom. Intell. Lab. Syst., vol. 74, no. 1, pp. 61–70, Nov. 2004.

Appendix

1. Table of Phase Gates and Tests with preliminary requirements for refrigerator liners

				Gate						
				1	2	3	4	5	6	7
Test Name	Test Condition	Unit	Test Standard	Collection	Pre-Processing	Metal Sorting	Plastic Sorting	Primary Compounding	Secondary Compounding	Product Manufacturing
Rheological										
Melt Flow Rate	200C/5.0 kg	cm^3/10 min	ISO 1133							2.8g
Shrinkage at Production		%	ISO 294-4							0.5 - 0.8
Mechanical										
Tensile stress at Break		MPa	ISO 527-2/50							24
Tensile stress at yield		MPa	ISO 527-2							16
Tensile Strain at Yield		%	ISO 527-2/50							
Tensile Strain at Break		%	ISO 527-2/50							60

Flexural Modulus (23°C)		MPa	ISO 178							1650
Tensile Modulus (23°C)		MPa	ISO 527-2/1							1650
Tensile Strength (23°C)		MPa	ISO 527-2/50							
Charpy Unnotched (23°C)		kJ/m^2	ISO 179-1eU							
Charpy Notched (23°C)		kJ/m^2	ISO 179-1eA							65, 38
Thermal										
Heat Deflection Temperature	0.45 Mpa	°C	ISO 75-2B							80
RTI temperature		°C	UL746B							
Vicat Softening Point		°C	ISO 306/A50							89
Flammability Rating		Classification	UL94							HB
Electrical										
Other										
Recycled		%	EN		100			Project	Project	Project

Content			15343					Requirements	Requirements	Requirements
Colour			ISO 11664		White			Project Requirements	Project Requirements	Project Requirements
Gloss			ISO 2813,					Project Requirements	Project Requirements	Project Requirements
Density		kg/m^3	ISO 1183							1.05
Filler content		%	ISO 3541-4							
Residual Humidity		%	EN 12099							
REACH Compliance						Yes				
RoHS Compliant			201165 EU			Pass				
Target Fraction					Refrigerators with white Liners	Refrigerator Liners	HIPS			
Purity/Fraction concentrations					100%	100%				
UL registered			Yellow Card							

Food contact approval			EC10/2011		From food contact application			Pass/Yes	Pass/Yes	Pass/Yes
Management System			ISO 9000		Yes	Yes	Yes	Yes	Yes	Yes
QC			QAS EC 2023/2006		Yes	Yes	Yes	Yes	Yes	Yes
Inventory Control/Shipping					Yes	Yes	Yes	Yes	Yes	Yes
Folio/Dossier Prepared					Full History	Full History	Full History	Full History	Full History	Full History
visual inspection for contamination		%	Sorting Effectiveness		100%					
			Chemical/Stain		No Visible Staining					
Chemicals Used during processing			Food Safe or removed		Compliant	Compliant	Compliant	Compliant	Compliant	Compliant
Degradation during		°C	max Temp							

proccesing										
Recycling Process(es)			EN15343		Table 1	Table 1	Table 1	Table 1	Table 1	Table 1
leaching test with simulants -			regulation No10/2011		Pass					
Challenge Test History (Control Charts?)					Work in progress	Work in progress	Work in progress			

2. MGG test reports:

Date:	19.12.18	
Inspector:	DP	
Sample:	PolyCE WP 7.1/REF:14.12.2018	

Analysis	Ecodom PS fridge clear	MGG Reference
HIPS (%)	96,9%	100%
PVC (%)	0,0%	-
Fines (%)	0,0%	-
Rubber (%)	0,0%	-
Metals (%)	0,0%	-
Glas, Concrete, Ceramic (%)	0,0%	-
Wood (%)	0,0%	-
Foam (%)	0,0%	-
other plastics (%)	3,1%	-
Br (mg/kg):	delayed	n.d.
Cd (mg/kg):	delayed	n.d.
Pb (mg/kg):	delayed	n.d.
Sb (mg/kg):	delayed	n.d.
Ba (mg/kg):	delayed	n.d.

Date:	19.12.18	
Inspector:	DP	
Sample:	PolyCE WP 7.1/REF:14.12.2018	

Analysis	Ecodom PS fridge white	MGG Reference
HIPS (%)	81,1%	100%
PVC (%)	0,0%	-
Fines (%)	0,0%	-
Rubber (%)	0,0%	-
Metals (%)	0,0%	-
Glas, Concrete, Ceramic (%)	0,0%	-
Wood (%)	0,0%	-
Foam (%)	0,0%	-
other plastics (%)	18,9%	-
Br (mg/kg):	delayed	n.d.
Cd (mg/kg):	delayed	n.d.
Pb (mg/kg):	delayed	n.d.
Sb (mg/kg):	delayed	n.d.
Ba (mg/kg):	delayed	n.d.

Date:	13.10.18	
Inspector:	som/ch	
Sample:	PolyCE WP 7.1/REF:10.10.2018	

Analysis	Ecodom PS fridge baseline	MGG Reference
HIPS (%)	86,7	86,8
PVC (%)	4,4	1,2
Fines (%)	3,9	0,1
Rubber (%)	1,9	1,5
Metals (%)	0,1	0,1
Glas, Concrete, Ceramic (%)	0,0	0,0
Wood (%)	0,1	0,3
Foam (%)	0,0	0,4
other plastics (%)	2,9	9,6
Br (mg/kg):	delayed	<50
Cd (mg/kg):	delayed	<20
Pb (mg/kg):	delayed	<5
Sb (mg/kg):	delayed	<5
Ba (mg/kg):	delayed	<500

3. Test reports from KU Leuven (~~Content~~ to be changed from process information to quality information)

PolyCE

Post-Consumer High-tech Recycled Polymers for a Circular Economy

Subject: Analysis of plastic flakes from Ecodom samples

Report for project internal communication

Author: KUL

Date:22.01.2019

Report content

The report includes the following samples:

Samples	Pre-Processing Datasheet (Metadata)	Fines content by sieving	Size & Shape Analysis	Colour Analysis	Composition with FTIR
1_Ecodom -white flakes fridge drawers warm up	yes	yes	yes	Yes*	yes
2_Ecodom-transparent flakes fridge drawers warm up	yes	yes	yes	Yes*	yes
3_Ecodom-plastics from wahsing machines warm up	yes	yes	yes	yes	No
4_Ecodom-plastics from washing machine drums warm up	yes	yes	yes	yes	No

*further development of testing techniques needed

The samples 1 and 2 are samples from warm up trials conducted by Ecodom for the recycling of food grade quality PS for demonstrator 7.1.2.

The samples 3 and 4 are samples from warm up trials conducted by Ecodom for the PP improved recuyclngg for demonstrator 7.7.2.

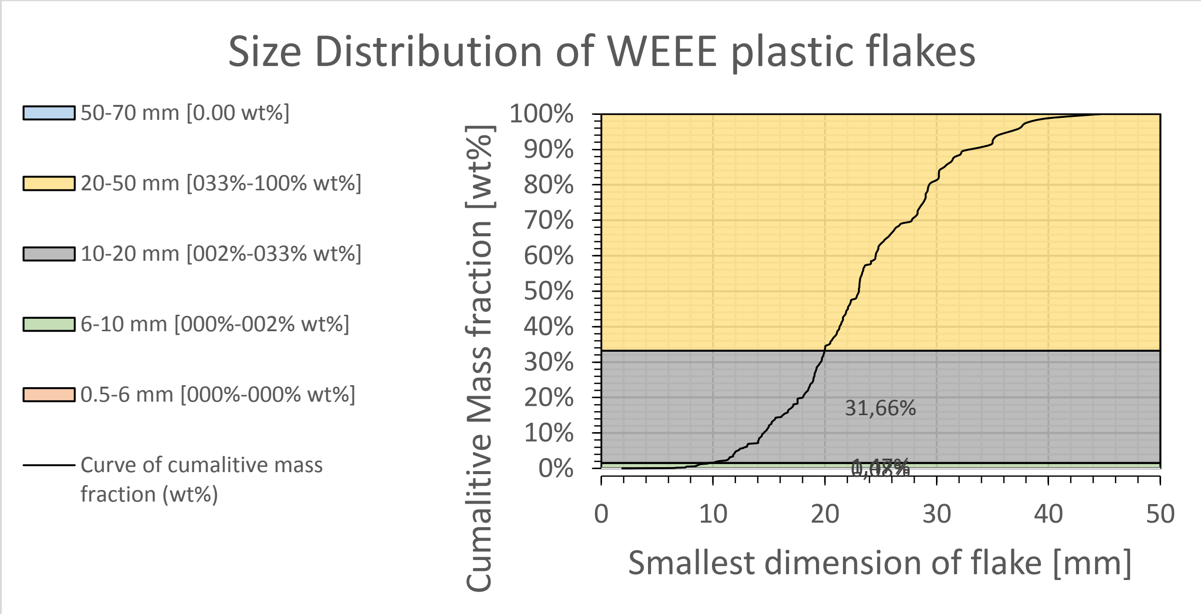
1. Ecodom -white flakes fridge drawers warm up

a. Metadata

(To be anonymized)		
Company		STENA TECHNOWORLD
Date sample sent		(dd-mm-yyyy)
Date sample received	*To be entered by KU Leuven*	(dd-mm-yyyy)
Sample identification code	*To be entered by KU Leuven*	(unique sample code)
Full address of the plant		Via dell'industria, 483, Angiari (VR)
E-mail address contact person		campadello@ecodom.it
Phone number contact person		+39 3336070931
General Description		
(Used confidentially)		
Product categories:		
Small household appliances		no
Large household appliances		no
Television screens		no
Monitors		no
Cooling and freezing equipment		yes
Washing machines and dryers		no
Other, please specify		(description)
Manual sorting		

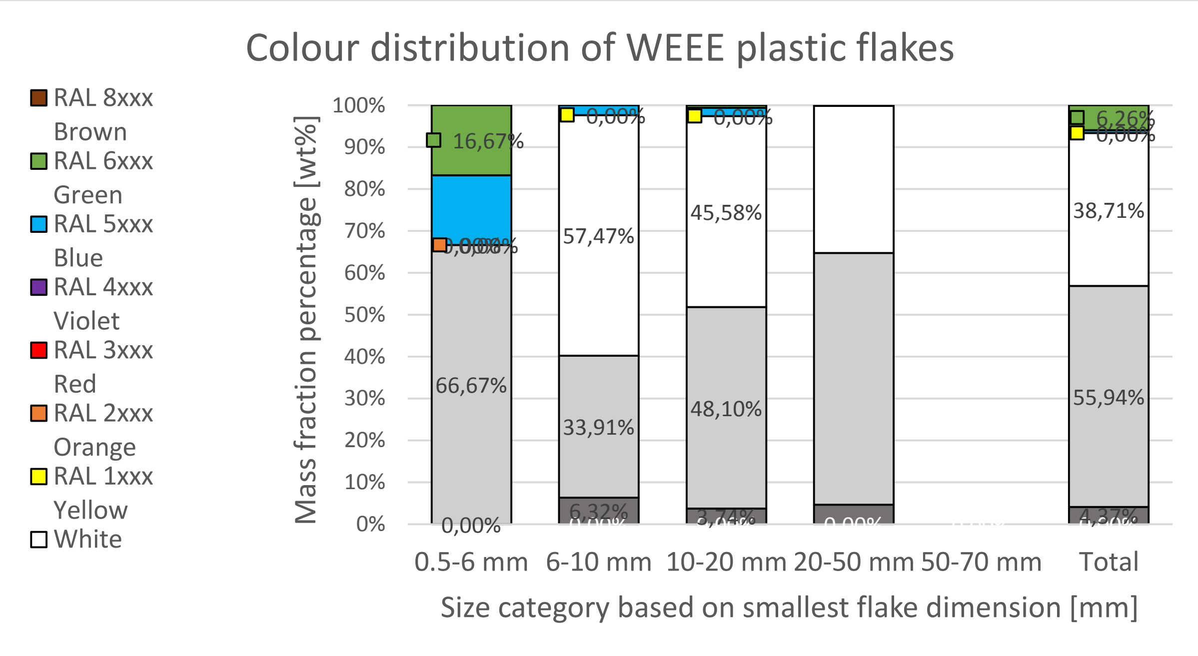
Components manually removed pre-shredder Components manually removed post-shredder	white fridges drawers mannualy removed to be shreddered separately (component(s) description)

b. Fines, Size and shape analysis



The fines content (<3mm) of the sample was 0,05 wt% base on sieving.

c. Colour analysis



The colour analysis showed unrepresentative results. Below one of the pictures used for analysis can be seen.

While it is clearly visible and represented in the results that different shades of white and grey are present in the flakes, further adaptation to define valuable thresholds for the different shades is necessary. In addition, the white flakes clearly show dark spots when looked at with the bare eye. These spots cannot be detected with the colour measurement. The dark spots are considered to be shredder residues as a consequence of processing in an uncleaned shredder. Also dirt is a possible cause of contamination. Processing in a clean shredder might be sufficient, however, washing should be considered.

d. Plastic composition

The plastic composition was 88,5 % polystyrene and 11,5 % PP filled based on the analysis of 35 valid measurements.

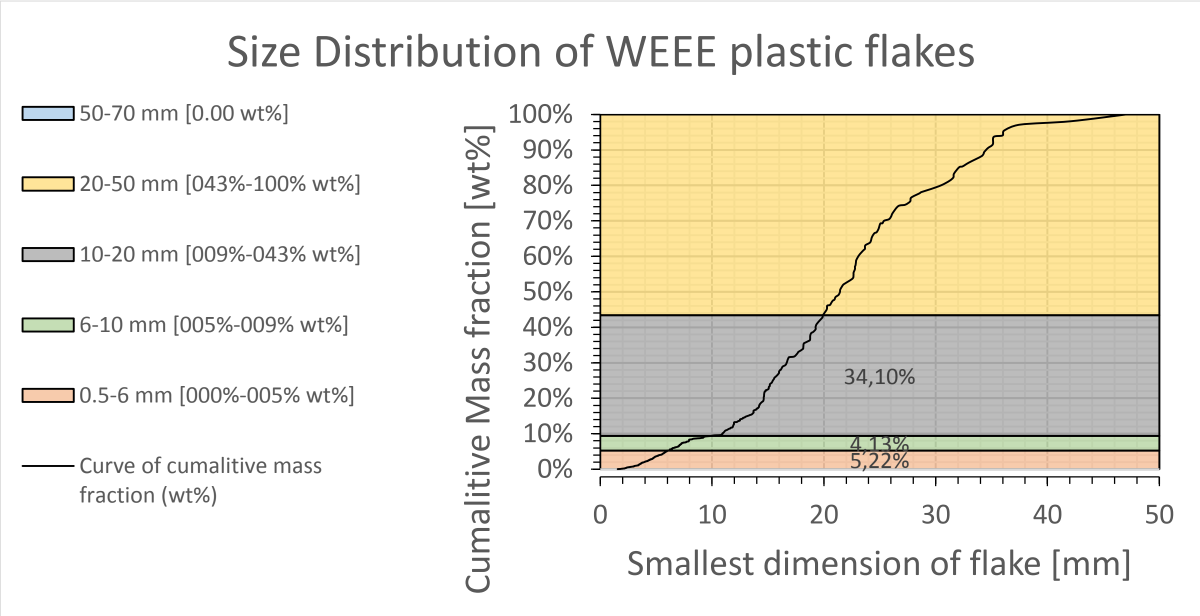
e. Conclusion

Processing on clean shredder required for large scale sampling trial. Relevant amounts of 11,5% PP talc filled are present.

2. Ecodom-transparent flakes fridge drawers warm up

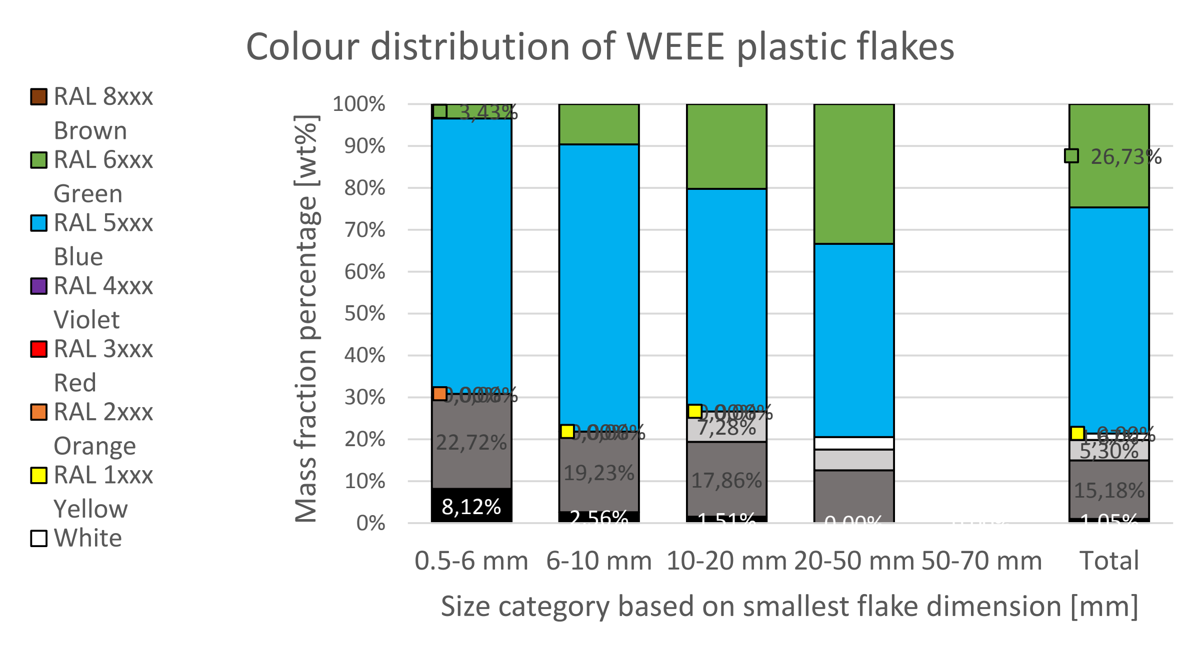
a. Metadata

b. Fines, Size and shape analysis



The fines content (< 3mm) of the samples was 0,05 wt%.

c. Colour analysis



The colour measurement results in a green and blue coloured fraction with some pieces in grey, white and black shades. Below one of the pictures used for analysis is shown.

The result of the colour measurement are not representative for the sample. The computer vision system needs to be adapted in order to identify transparent shades of plastics which is considered to be very challenging. Additional manual analysis showed that different shades of transparency are present in the sample. Below a picture indicates some colour shades that are present.

A very small percentage (<<1%) of plastic flakes were black and are assumed to be impurities from shredding or transport. In addition approximately 15% of the plastic pieces were in a smokey coloured transparency shade, which can be seen in the computer vision analysis as grey.

d. Plastic composition

For the transparent flakes we found 100% polystyrene based on the analysis of 42 valid measurements.

e. Conclusion

The extrusion trials at MGG Polymers resulted in an insufficient quality of the samples based on a smokey colour combined with a rather opaque than transparent appearance. The smokey colour is assumed to originate from the smokey colour shades present in the input material and would need to be sorted before extrusion in order to avoid a final smokey colour. Possible causes for the opaque appearance were summarized in the table below with possible testing procedures and reacting that could be taken in the recycling process:

Transparency changes in Polystyrene			
Cause	**Origin**	**testing**	**reaction**
Material heterogeneous (voids, orientations)	processing	tensile testing low strains leads to lower transparency, granulates after compounding transparent?	adapt mixing in compounding, injection moulding parameters
Air inclusion	processing	tensile testing low strains leads to lower transparency	degassing
Moisture inclusion	material	tensile testing low strains leads to lower transparency	dry

Degradation (Oxidation, double bonds, gas formation)	processing/material	FTIR, yellow appearance =>oxidation, double bonds	stabilize, adapt processing, degassing
Additives	material		research required (find origin, define sorting measures)
impurities	material	composition analysis (of fines)	avoid shredder contamination, washing
crystallization	processing/material	tensile testing low strains leads to higher transparency, DSC	Hindrance of crystallinity: introduction of sulfonates has been shown to increase transparency of polyamides by retarding crystallization, crosslinking. Control of

			crystallinity: nucleation to receive small crystals

f. Crystallization

As commercial PS is either atactic (amorphous) or syndiotactic (froms crystals), it is considered unlikely that atactic PS changes tacticity, however atactis PS can have parts of the chain that are syndiotactic and form crystals. Unlikely due be due to processing, more likely that impurities act as nucleing agents. Crystallization could be quickly checked with DSC measurements.

g. Impurities

As some black plastic pieces were found (<<1%) and MGG measured some other plastics, this is a likely cause, that could also induce crystallinity. The processing on a clean shredder for the larger scale trial will therefore be crucial. Remaining oils could influence transparency or degrade during processing, but it is assumed that this can be avoided by washing.

These are considered to be the most likely causes for the opaque appearance of the polystyrene samples. If the problem still exists after taking the proposed measured, extrusion tests with degassing could help evaluating the presence of gases.

It needs to be clarified what the targeted appearance of the recycled fraction is as no shades of transparency is defined yet.

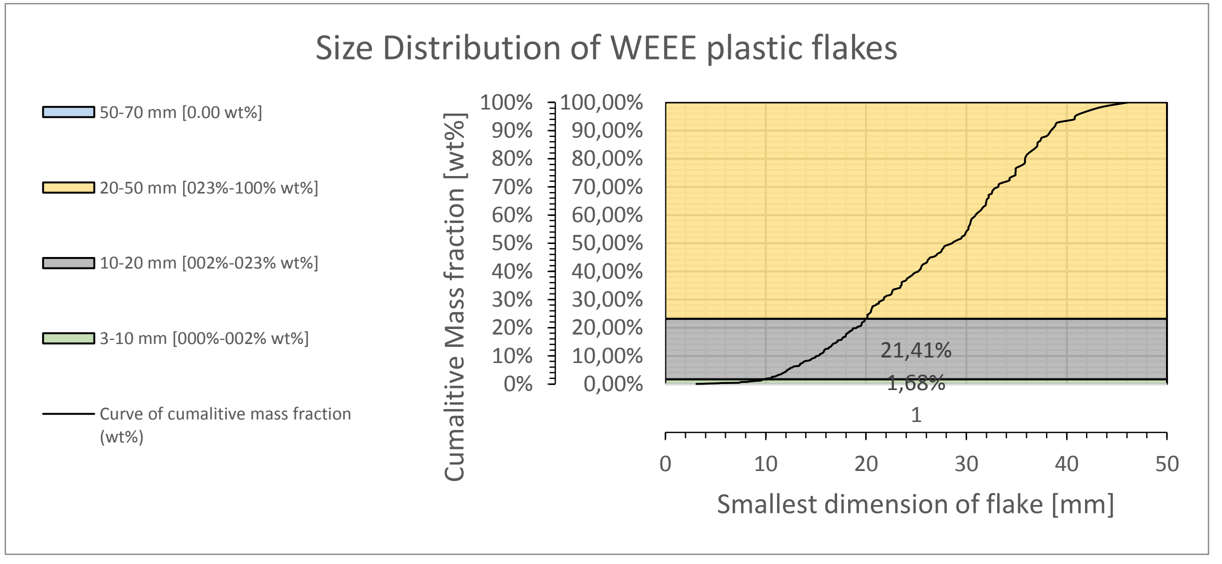
3. Ecodom-plastics from washing machines warm up

a. Metadata

(To be anonymized)		
Company	SEVAL	(name)
Date sample sent	10/01/2019	(dd-mm-yyyy)
Date sample received	*To be entered by KU Leuven*	(dd-mm-yyyy)
Sample identification code	*To be entered by KU Leuven*	(unique sample code)
Full address of the plant	Via la Croce, 14, 23823 Colico LC	(address)
E-mail address contact person	campadello@ecodom.it	(e-mail)
Phone number contact person	0039 333 6070931	(phone number)
General Description		
(Used confidentially) **Product categories:**		
Small household appliances	no	(yes/no)
Large household appliances	yes	(yes/no)
Television screens	no	(yes/no)
Monitors	no	(yes/no)
Cooling and freezing equipment	no	(yes/no)
Washing machines and dryers	no	(yes/no)
Other, please specify	nothing	(description)
Manual sorting		

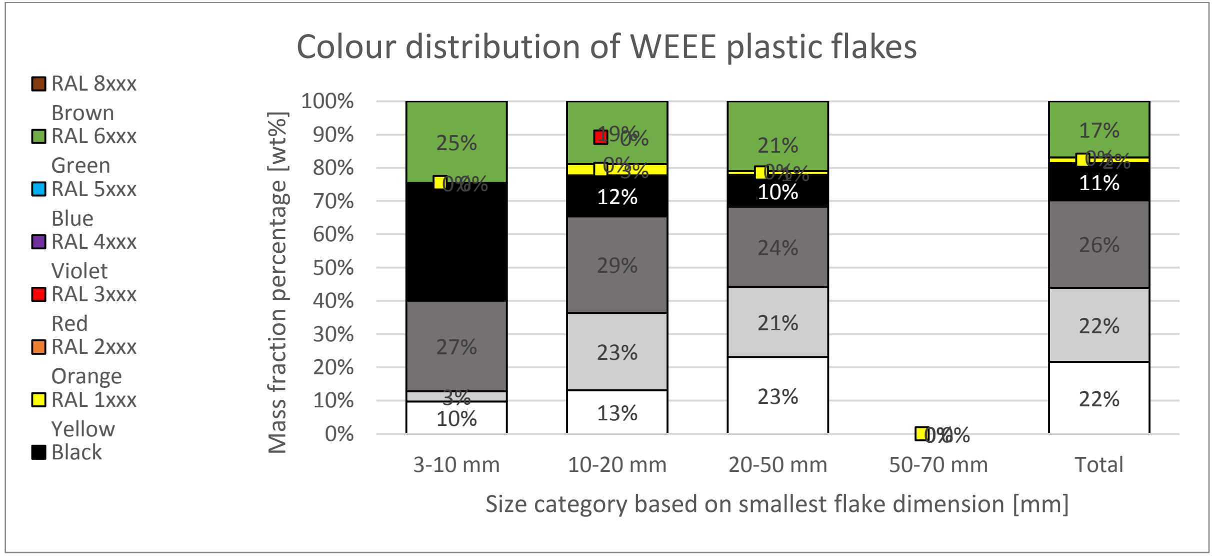
Components manually removed pre-shredder	door of the washing machine drain hose cables	(component(s) description)
Components manually removed post-shredder	Step1: iron panel, drive motor, concrete block, cables/rubber seals Step2: cables, pieces of glass belonging to the door	(component(s) description)

b. Fines, Size and shape analysis



The fines content (< 3mm) of the samples was 0,03 wt%.

a. Colour analysis



b. Plastic composition

To be tested

c. Conclusion

Tests to be finalized

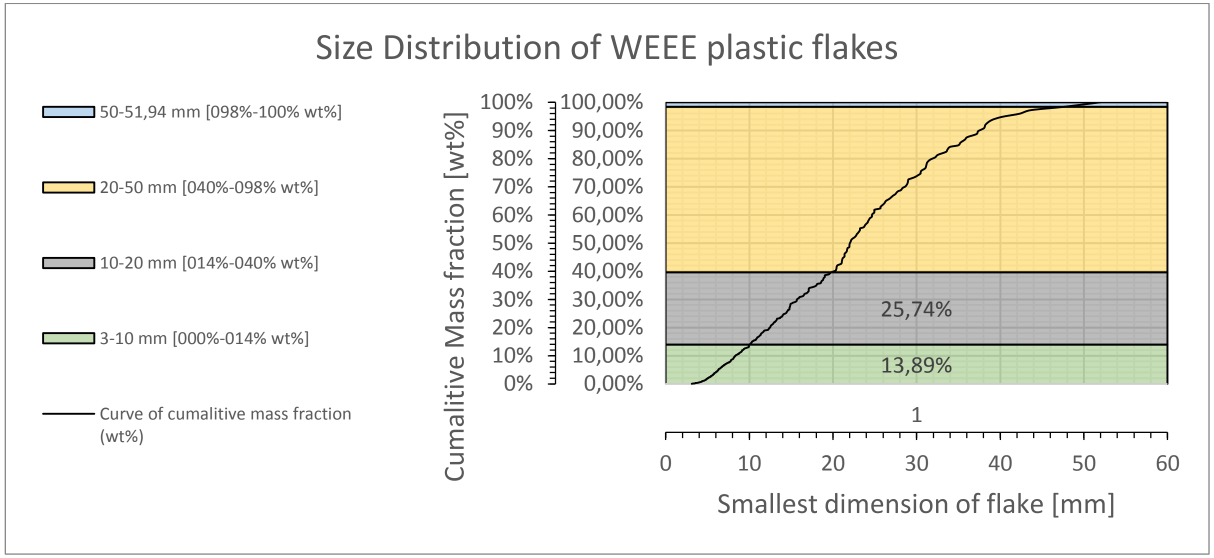
c. Ecodom-plastics from washing machine drums warm up

a. Metadata

(To be anonymized)		
Company	SEVAL	(name)
Date sample sent	10/01/2019	(dd-mm-yyyy)
Date sample received	*To be entered by KU Leuven*	(dd-mm-yyyy)
Sample identification code	*To be entered by KU Leuven*	(unique sample code)
Full address of the plant	Via la Croce, 14, 23823 Colico LC	(address)
E-mail address contact person	campadello@ecodom.it	(e-mail)
Phone number contact person	0039 333 6070931	(phone number)
General Description		
(Used confidentially) **Product categories:**		
Small household appliances	no	(yes/no)
Large household appliances	yes	(yes/no)
Television screens	no	(yes/no)
Monitors	no	(yes/no)
Cooling and freezing equipment	no	(yes/no)
Washing machines and dryers	no	(yes/no)
Other, please specify	nothing	(description)
Manual sorting		
Components manually removed pre-shredder	washing machine drums mannualy removed to be	(component(s) description)

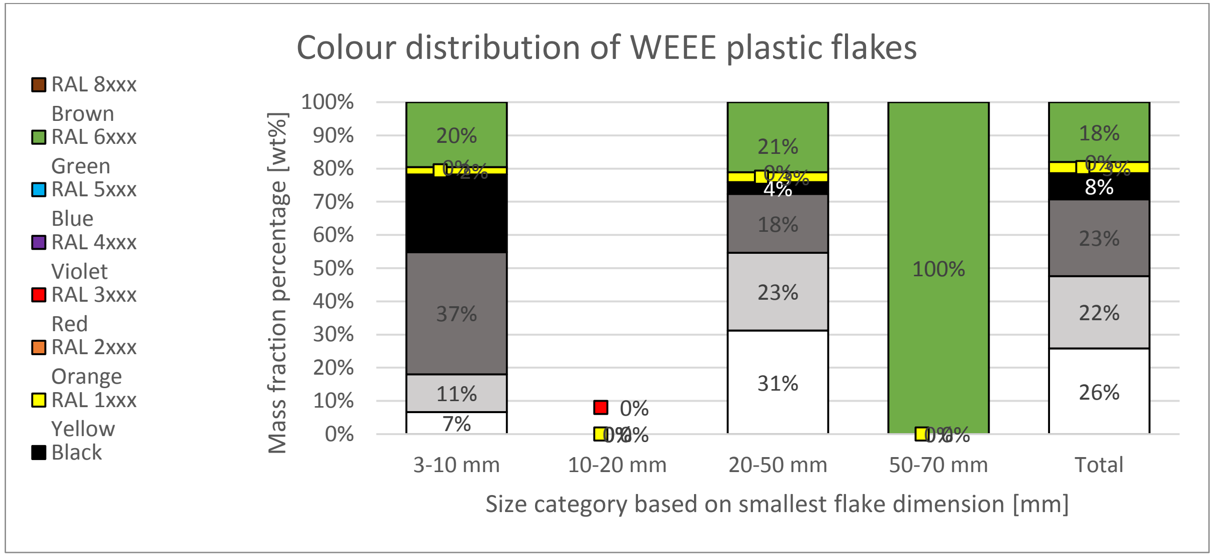
	shreddered separately	
Components manually removed post-shredder	rubber seals cables and plastics containing metals	(component(s) description)

b. Fines, Size and shape analysis



The fines content (< 3mm) of the samples was 0,56 wt%.

c. Colour analysis



d. Plastic composition

To be tested

e. Conclusion

To be finalized