STANDARDISING THE SAMPLE PREPARATION FOR ANALYSIS OF FIBRES AND PARTICLES BY STATIC IMAGE ANALYSIS

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ABSTRACT

Static image analysis is known as a versatile method, which is in use for characterisation i.e. of fibres, nonwovens, textile recyclates etc. Due to incomplete standardization (esp. in the area of sample preparation) the usage is actually limited. Within the project StaPAFaB two research institutes are engaged to compile a reference manual listing typical classes of materials and optimised methods of sample preparation for each of them. This will be combined with recommendations for reasonable parameters in image acquisition / processing and possible limitations for each type of material. Aim is to enable reproducible and consistent analyses on an inter-laboratory level as well as to reduce the demand of time for the analyses. This article focuses on typical classes of textile materials and adapted methods to enable their quick and reliable sample preparation.

KEYWORDS

Image analysis; Sample preparation; Textile fibres; Recycled fibres.

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INTRODUCTION

Static image analysis delivers more detailed results than e.g. sieving. Namely there are distributions instead of average values for length, width and several shape factors. As for all analytical methods, standardisation is an essential prerequisite for reproducibility as well as for comparability of the results. Unfortunately, up to now standardisation has taken place only incompletely for static image analysis. ISO 9276–1 to –6 [1] define parameters for evaluation and graphical presentation of the results. Image acquisition and calibration of the experimental setup is only specifically described by the equipment suppliers. This does not cover the way how to prepare samples for the static image analysis.

Consequently this leads to problems in commercial and research laboratories. Static image analysis is a useful method for universal access to parameters like size and shape of a wide range of materials, combined with statistical analysis. This misleads to a frequent use of this method for new questions and single research samples. Finally different ways of sample preparation, combined with various parameters in image acquisition and analysis lead to strongly different results. This guarantees neither a reproducibility of results in one laboratory, nor comparable results between different laboratories. On the other hand it is known from inter-laboratorial round trials, that a well-described procedure can guarantee identical results within a small tolerance [2].

To overcome the problems in the area of sample preparation, the project StaPAFaB has been started, where two research institutes are engaged to compile a reference manual listing typical classes of materials and optimised methods of sample preparation for each of them. The manual will comprise different types of sample preparation as well-documented guideline for scientists and practitioners. This article presents results for typical ‘easy’ and ‘complicated’ sample materials to give a first insight into the project aims.

DEFINITION OF MATERIAL CLASSES

Typical sample Materials have been collected during the project and have been classified according to a newly set-up scheme (cf. Figure 1) allowing to find easily adequate methods of preparing samples for analysis. The material classes are:

- **particles with free-flowing property**, e.g. powders, small crystals (minerals), granules, rice husks etc. Main criterion: these particles do not stick together.
- **particles with limited free-flowing property,** e.g. short fibres, shives etc. Main criterion: these particles tend to stick together slightly, but can be separated non-destructively by small mechanical action.
- **single fibres**, which are not short fibres (see above). Main criterion: the length must be smaller than the max. scanner image length.
- **roving snippets**, e.g. cut-offs from processing high performance fibres (glass, carbon, etc.). Main criterion: the snippets are stable enough for either sieving or at least manual separation using tweezers.
- **recyclates** (consisting of several material fractions): depending on structure several options are possible.

Each material class is sub-divided into groups. For each group possible methods of preparation are specified and recommendations are given.

For a correct assignment of a sample to the most relevant group and best fitting preparation method it is essential to know, what the aim of the analysis is and which parameters have to be analysed. Otherwise the preparation time and number of parameters to analyse may exceed your budget and time! In experience of the authors each hour of discussion with the sample supplier can save two or more hours working with the samples.

Based on this information it is easy to assign each sample material to one of the groups and then to select the preparation method fitting best to analyse the parameters required by the sample supplier. A typical example for saving time and efforts is a fibre sample (class: single fibres). In the worst case there is only manual preparation possible to analyse length and crimp of each fibre. In the best case the fibres can be cut to short fibres and the width distribution can easily be analysed.

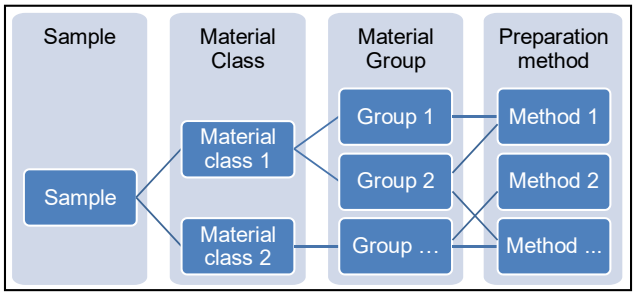

Figure 1. Scheme of material classification to identify adequate method(s) for sample preparation

EXAMPLES FOR SAMPLE PREPARATION OF DIFFERENT MATERIAL CLASSES

Within this section typical examples are given for an easy sample preparation using the dispersion by sieving (cf. section 3.1) as well as a quick separation method for complex recyclate mixtures enabling at least access to essential data of composition (cf. section 3.2).

Example #1: 'easy' sample preparation

As described in the previous section, recommendations for different Materials may end up in the same preparation method. This is the case for e.g.:

- Short cut fibres ('flock fibres') <2 mm length: material class 'single fibres', group short fibres, within the group described as case of 'short enough to exhibit free-flowing property'
- Sand or minerals (Aluminiumtrihydroxide 'ATH', Soda etc.) in dry state: material class 'particles with free-flowing property', group dry powders

For both of them use of a sieve as aid for dispersion is recommended, assisted by shaking or use of a brush. In Figure 2 (a) the experimental setup is shown in brief: a spoon and/or spatula to distribute the sample on the sieve, a brush for additional dispersion, and an analytical sieve in adequate mesh size. To guarantee a reproducibility of the results, the sieve must be certified to a standard like ISO 3310-1 [3]. Some preceding trials are recommended to identify the optimal sieve size: if a sample passes directly through the sieve, the mesh size is too big. If the sample remains (nearly) completely on top of the sieve and does not pass even under vibration, the sieve size is too small. If the sample has a large size distribution, it may be necessary to use two or more sieves to fractionize the sample. In this case the complete sample must be analysed in several images to obtain a valid result representing the sample correctly.

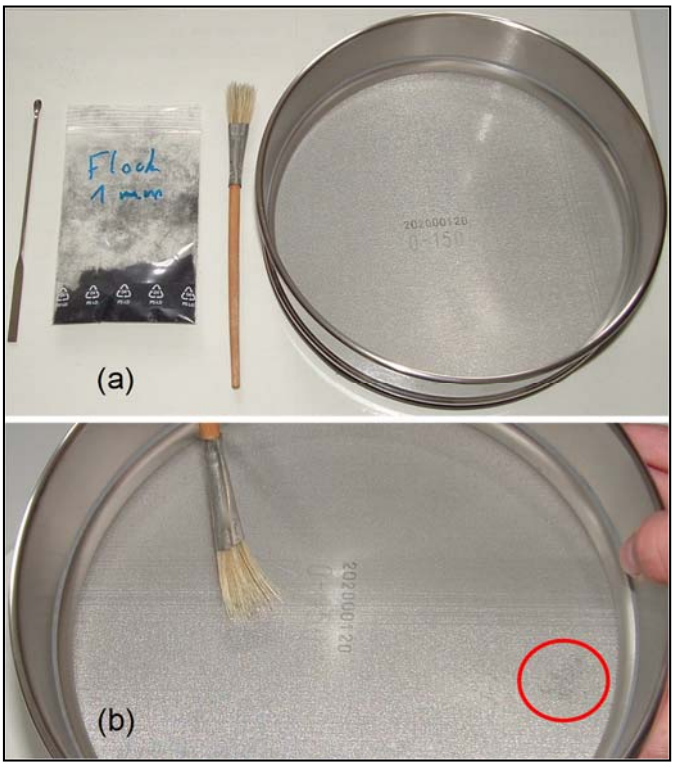

Figure 2. (a) Flock sample, tools and sieve for preparation and (b) dispersing by brush with flock agglomerate still to disperse in red circle.

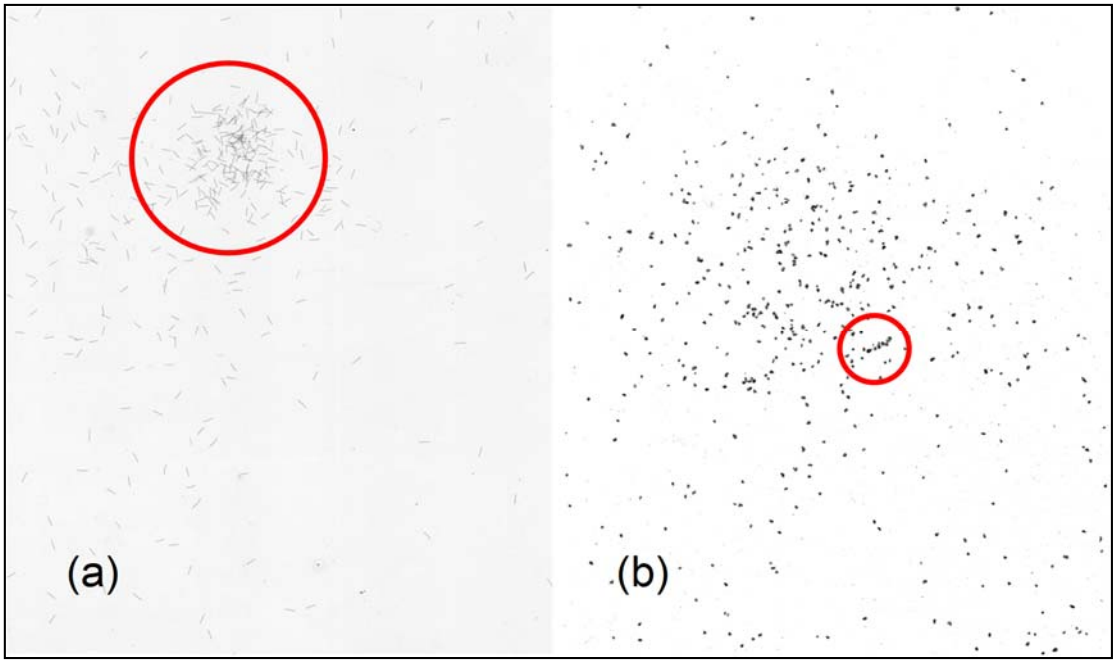

Figure 3. Acquired images of (a) flock fibres of figure 2 and (b) sugar particles. Red circles indicate remaining agglomerates which should be excluded from analysis.

For sample preparation mostly it is sufficient to position the sieve above the scanner or a transparent foil, which is later transported into the scanner. Then the sample is distributed over the sieve by the spatula or spoon. Finally the particles can be dispersed by using a brush as shown in Figure 2 (b). Agglomerates of sample particles have to be dispersed carefully in order to achieve a good distribution for scanning. If particles remain on the sieve, they must be transferred to a second transparent foil to be analysed as additional image to guarantee an analysis of the complete sample.

In Figure 3 the greyscale scans of two particle sample are presented as typical examples for the sample types listed above: (a) flock fibres of figure 2 with 1 mm of nominal length representing short fibres and (b) sugar particles as example of crystalline particles.

Both samples exhibit a good dispersion of the particles over the image. But, in both samples there are small regions with insufficient dispersion, indicated by red circles. For the image analysis it is essential, that there are not too much overlapping particles. In general it is not possible to analyse these overlaps correctly, and thus they must be excluded from the analysis. For the sample preparation this means, that either the particles in these regions may be separated manually (only reasonable for large particles), or they must be excluded from the subsequent analysis.

Finally the images can be loaded into the desired image analysis software to perform the analysis of the desired parameters. For short fibres this is typically width and length, while for particles it is typically the grain size, aspect ratio etc.

Example #2 / complex sample preparation

Textile recyclates often consist of different fractions. Thus there was normally only the time-consuming possibility of manual separation and subsequent analysis of each fraction. Within the project a new approach using compressed air to separate the fractions has been developed. It enables a quick approach to at least rough analysis of the shares.

The experimental setup (cf. Figure 4 a & b) is simply using a sampling bag 42 x 21 cm and a commercial airbrush pistol (a). The sample here is cotton from T-shirts after the tearing process. After separating the agglomerate (b) the bag has to be transported horizontally to a flatbed scanner to acquire a greyscale image (cf. Figure 4 c). The fractions fabrics, yarns and fibres can now be easily identified and quantified by their gray scale values. In this case the fabrics appear black, represented by the greyscale values 0 to 50, while the yarn pieces are dark grey, represented by the greyscale values 51 to 140. The fibres appear light grey, represented by the greyscale values 141 to 200. Values above 200 to 255 represent the background.

The exact limits of the different fractions vary for different recyclates depending on structure, colour and degree of disintegration. Thus the greyscale limits for the different fractions should be defined individually. Now it is easy to analyse the greyscale histogram, using e.g. free software like ImageJ [4]. Typically not more than 5 – 10% of the pixels should be particles to avoid too strong overlapping. From these pixels the share of the different fractions can be calculated in %, representing roughly the mass shares.

As a second example a carpet recyclate after separation in compressed air is shown in Figure 5 with (a) photograph and (b) greyscale scan. For this material the same approach for assessing the shares of different fractions is possible. In this case not only the fabrics (carpet backing), but as well the tufting yarn appear black. For this reason they can only be counted as one fraction. Finer yarns from the backing and fibres are the other fractions evaluatable.

Summing up, by this method of sample preparation it is easily possible to access the share of fractions in textile recyclates. This is important to control the efficiency of the tearing process and gives information necessary to decide, if the degree of disintegration is sufficient.

In principle, some more parameters like size and shape distribution of the fabric fragments or width distribution of the fibres can be analysed.

Other options like length distribution of the yarn pieces and fibres would be only available after manual sample separation.

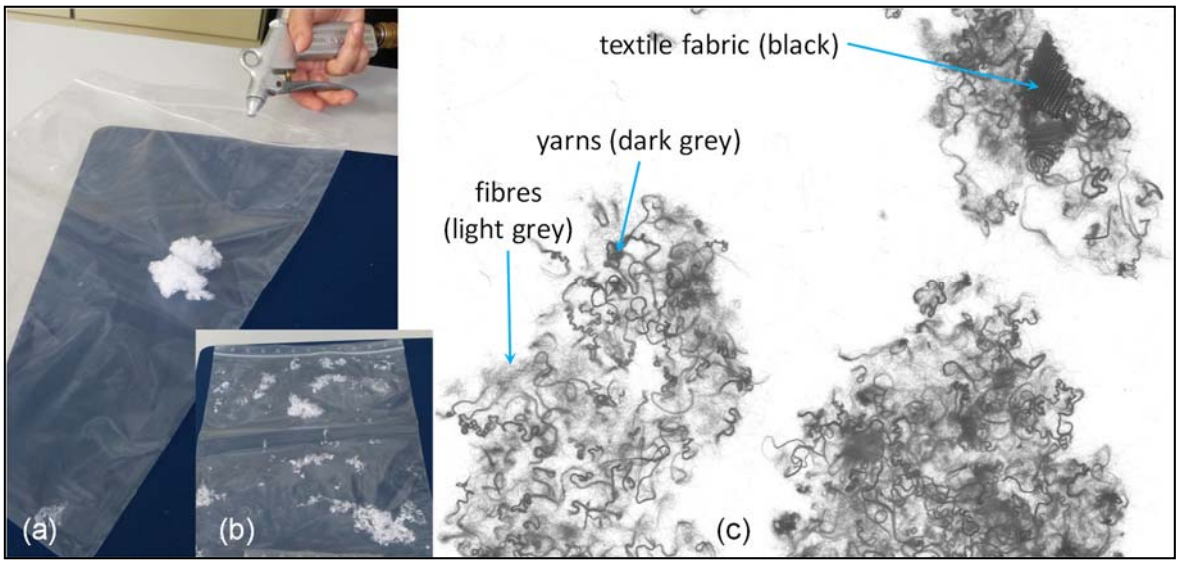

Figure 4. (a) agglomerate of recycled textile in sampling bag, (b) after separation by compressed air and (c) resulting grayscale image in sampling bag with fabrics in black, yarns in dark gray and fibres in light gray.

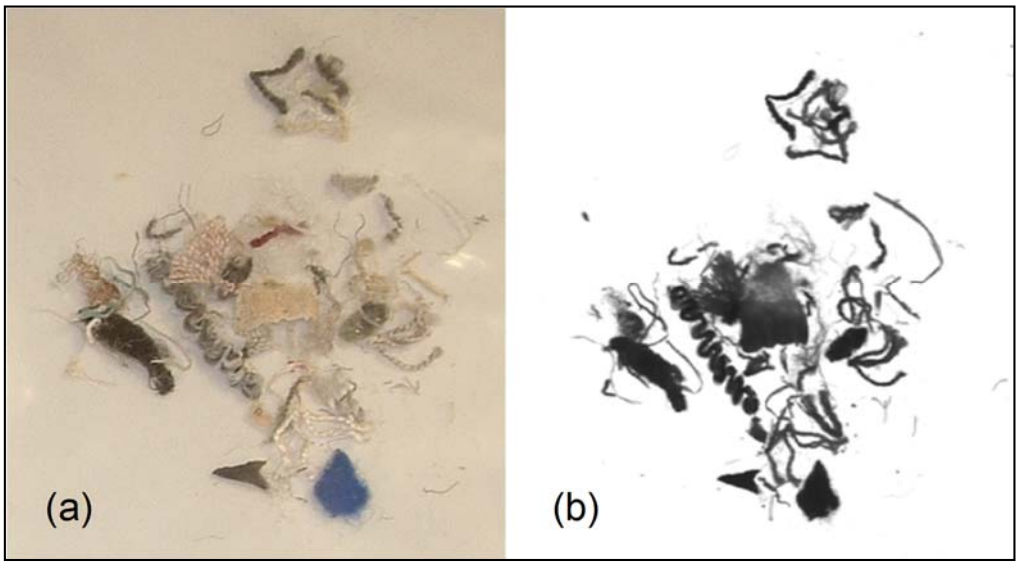

Figure 5. Agglomerate of recycled carpet, (a) photograph and (b) grayscale scan.

CONCLUSIONS & OUTLOOK

Within the project StaPAFaB a scheme has been set-up for quick and reliable sample preparation for static image analysis. The project work led to progress in sample preparation techniques especially in the field of textile recyclates. At the beginning of the StaPAFaB project the only method to prepare samples from these recyclates was manual separation, demanding up to >1 day per sample. Using the compessed air method described here it is possible to prepare a sample within minutes, which is good enough to assess the parameters important for the tearing process.

In order to disseminate the results to a broad circle of interested scientists as well as practitioners, a public workshop will take place in January 2023 in Bremen / DE for everybody interested in these topics. In addition, the project results will be published in April 2023 as a reference manual listing typical classes of materials and optimised methods of sample preparation for each of them.

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