CHEMICAL ENGINEERING TRANSACTIONS Volume 17, 2009
Editor Sauro Pierucci
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DOI: 10.3303/CET0917124

Characterization of Short Fibers

Andreas Bartl, Davide Pico
Vienna University of Technology – Institute of Chemical Engineering
Getreidemarkt 9/166, 1060 Vienna, Austria

The methods for characterization of particles have improved drastically within the last years. Today, quick and accurate tools for determination of size, size distribution or shape are commercially available. However, most common systems fail when applied for fibrous particles. In this paper, the sieving, laser diffraction, optical microscopy and optical image analysis have been compared by analyzing well known fiber samples. According to standards, the time consuming process of microscopy is required. An optical fiber analyzer seems to be a good compromise between accuracy and time consumption.

1. Introduction

Particle characterization is an important field in mechanical process engineering and it is evident that fibers represent a disperse system consisting of innumerable individual particles.

It is well established to characterize particulate systems according to certain attributes, among which size and shape are of importance. Since particles are usually irregularly shaped, several methods are established to calculate statistic diameters such as a Feret or Martin diameter (Gebelein, 1956 and 1958, Church, 1968/69, Leschonski, 1984) However, particle size is not a proper tool for describing fibers since length and width are quite different.

It is striking that the term fiber is not clearly defined in literature. Textile industry, the most important user of fibers, defines a fiber as a linear particle which can be processed to textiles (DIN, 1999). A more far-reaching definition (BISFA, 2000), however, sees a fiber as a morphological term for substances characterized by their flexibility, fineness and high ratio of length to cross sectional area. According to this definition fibers are subdivided into fiber fly (airborne fibers), flock (very short fibers for other purposes than spinning), staple fibers (of limited but spinnable length) and filaments (length considered as continuous).

Within the last years several fiber analyzers have been developed for the pulp and paper industry and are commercially available (Hirn and Bauer, 2006). These systems can also be used to determine other attributes such as curl or kink. It has also been demonstrated previously that fiber analysis developed for pulp and paper characterization can also be used for measuring short fibers (Bartl et al., 2005).

Please cite this article as: Bartl A. and Pico D., (2009), Characterization of short fibers, Chemical Engineering Transactions, 17, 741-746 DOI: 10.3303/CET0917124

2. Reference Materials

In order to be able to evaluate the prospects of different analyzing systems, four fiber samples with well defined geometries were selected as references. Table 1 summarizes the details of the samples. Due to the manufacturing processes the individual fibers exhibit a uniform width and length with almost no deviations.

Table 1 Data of reference fibers for evaluation of measuring systems

Material	Viscose flock
Manufacturing technology	Guillotine cutting
Manufacturer	Casati s.n.c., Renate (Mi), Italy
Fiber width	1.7 dtex (i.e. 11.9 µm)
Fiber length	l_1 = 0.3 mm/ l_2 = 0.5 mm / l_3 = 1.0 mm / l_4 = 2.0 mm

3. Sieving

Sieving is a widely used method for quality control in the industry enabling a determination of mass fractions between two sieves of different mesh sizes. Conventional sieving is convenient for particle sizes from approximately 63 µm up to 125 mm. Air jet sieving allows even smaller sizes down to 10 µm and shows the advantage that interfering particles are continuously blown away from the mesh.

Although it can be expected that fibers, due to their extreme non-spherical shape, will not be convenient for sieving, in the literature, fibers derived from carpets have been analyzed by this method (Wang, 1995). The reference samples have been analyzed by air jet sieving according to a standard, which is used for characterizing plastics (ISO, 2001). However, since no feasible results were obtained, the portion of passed sample through the 20 µm sieve was determined against sieving time. The respective plot is shown in Figure 1. The total sieving time increased with the length of the flock samples. In the case of the 2.0 mm flock, sieving was completely impossible and the sample did not pass through the sieve at all.

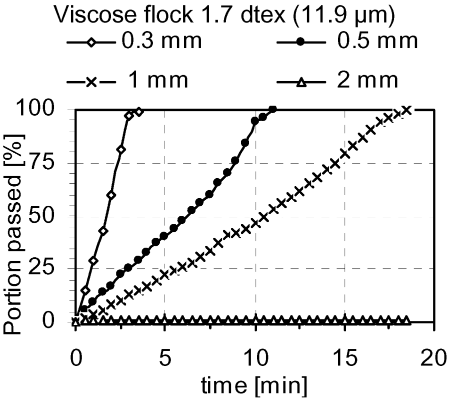

Figure 1 Portion of flock passing a 20 µm sieve of an air-jet sieve apparatus (Alpine 200 LS-N

This means that the result of a sieving analysis is significantly influenced by the sieving time. When the sieving procedure lasts a fairly long time period the sample will completely pass the sieve unless a certain maximum length is not exceeded.

It can be concluded that sieving is not a proper tool for characterizing short fibers. The method is of certain interest in the flock industry since, commonly, fibers show a uniform width and thus the results allow an estimation of fiber length.

4. Laser diffraction

Particle size analysis by laser diffraction is well established today, as it is a quick and reliable method. It relies on the fact that particles passing through a laser beam will scatter light at an angle directly related to their size. Based on the interpretation method, Fraunhofer and/or Mie theory, the particle size may range from approximately 0.1 to 2000 µm. However, the system can only gain information about size, not about shape. Therefore the system is not convenient for fibers.

In order to demonstrate the limitation of laser diffraction the 0.3 mm flock sample was measured with a CILAS 920 (range: 0.4 to 400 µm) in aqueous solution. Figure 2 shows the plots of four runs indicating quite different, non-reproducible results. One curve shows a distinct maximum at 18 µm which corresponds to the width of the swelled fiber. However, the other curves show maxima at larger, but different sizes, which seems to be a mean value between length and width. In no case any signal was determined at 300 µm, the nominal length of the flock sample. It is thus clear that laser diffraction is not applicable for samples which significantly exhibit a large deviation between length and width.

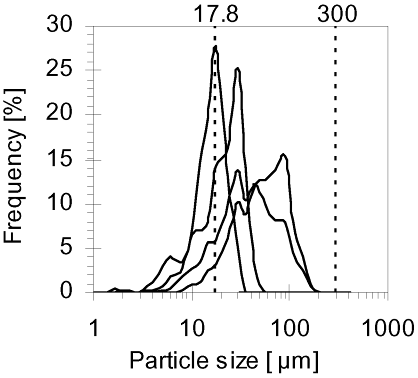

Figure 2 Size distribution function of viscose flock (l_1) determined with the CILAS 920

Optical Microscopy

Light optical microscopy is a very accurate but time consuming method for determination of size and shape of particles. Since the interpretation is not based on automated procedures, the method is convenient for fiber characterization. It is thus recommended that the determination of airborne fibers is performed by optical microscopy (BGI, 2004).

Figure 3 shows a picture of the flock samples (Table 1), which is composed of nine individual photographs. It is possible to use a high magnification in order to evaluate fiber width which ranges at about 13 µm; this is in good agreement with specification of the manufacturer. At the same time it is also possible to determine the fiber length, even of long fibers (2 mm) which are covered by two or three images.

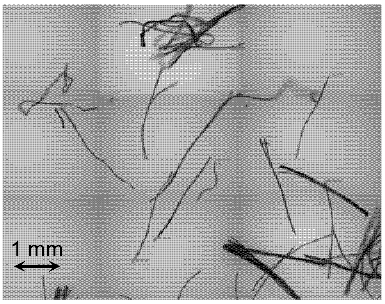

Figure 3 Light optical photograph of viscose fiber flock.

It has been demonstrated that fiber characterization by optical microscopy exhibits high accuracy, particularly when it is combined with modern microscopy software systems. However, for gaining statistically valid data at least 100 individuals have to be evaluated, which is quite time consuming (BGI, 2004). For a quick and economic determination this method is not convenient.

5. Optical Image Analysis

5.1 Fiber Analyzer

Among other analyzers optimized for fibers the MorFi system (Passas et al., 2001, Tourtollet, 2001) shows a rather high optical resolution (Hirn and Bauer, 2006). It has already been demonstrated that the MorFi analyzer is a proper tool for characterizing short fibers (Bartl et al., 2004), possibly originating from recycling processes (Bartl et al., 2005). However, since fibers have to be suspended in water, hydrophobic materials demand the use of surfactants and hydrophilic fibers will pretend higher widths due to swelling effects, even up to 100 % for viscose (Marini et al., 1980 and 1981, Segula, 1970). For all presented results 10,000 individual fibers have been evaluated. For this investigation only fiber length was considered.

5.2 Pure Samples

When talking about any average length it is essential to state the kind of distribution function, which can be based on number, length, area, volume or mass. The MorFi analyzer computes the average length weighted by both, number and length.

Figure 4 shows the length of the flock samples (as specified in Table 1) determined with the MorFi analyzer. The length specified by the manufacturer is in good agreement to the results of the MorFi analyzer. This small deviation, which is well below 8 %, is probably caused by minor variances of the cutting aggregate.

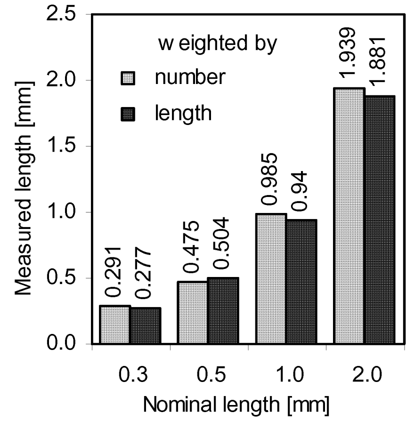

Figure 4 Comparison of nominal and measured length of flock samples.

The minor difference (up to 5 %) between the average length weighted by number and length is most likely caused by small variations in fiber length and minor impurities. In conclusion, the MorFi demonstrated its ability for determination of fiber length.

5.3 Mixed samples

Commonly, it is not the task to analyze fibers of uniform lengths but mixtures of various lengths. In order to evaluate the practicality of the MorFi analyzer, a set of specimens have been prepared by mixing different portion of the flock samples. Starting with the first specimen containing 100 % of the 1.0 mm flock sample, the portion was gradually decreased to 0 %. Simultaneously flock samples with 0.5 and 2.0 mm nominal lengths were added, so that the average lengths weighted by number remained at 1.0 mm. The actual average lengths differ only slightly from 1.0 mm since the results of the MorFi analysis and not the nominal lengths, were taken into account for the calculation. However, the lengths weighted by length significantly increase with the decreasing portion of the 1.0 mm flock sample. Table 2 shows all specimens indicating their portions of the flock samples (0.5, 1.0 and 2.0 mm) and the resulting average length for both, weighted by number and weighted by length.

All specimens have been analyzed with the MorFi system. The respective results are plotted in Figure 5. The left diagram compares the calculated and the measured fiber lengths weighted length. The results of the MorFi analyzer are slightly higher than the calculated values. The difference does not exceed 0.1 mm error; thus being below 8 %.

Table 2 Specimens containing different portions of flock samples and their calculated average lengths

Portion [length %]			Length weighted	Portion [number %]			Length weighted
0.504*	0.958*	1.939*	by length	0.504*	0,958*	1.939*	number
0.000	1.000	0.000	0.958	0.000	1.000	0.000	0.958
0.033	0.900	0.067	1.008	0.064	0.903	0.033	0.962
0.067	0.800	0.133	1.059	0.128	0.806	0.066	0.965
0.100	0.700	0.200	1.109	0.192	0.708	0.100	0.969
0.133	0.600	0.267	1.159	0.257	0.609	0.134	0.972
0.167	0.500	0.333	1.209	0.323	0.509	0.168	0.976
0.200	0.400	0.400	1.260	0.389	0.409	0.202	0.980
0.233	0.300	0.467	1.310	0.455	0.308	0.237	0.983
0.267	0.200	0.533	1.360	0.522	0.206	0.272	0.987
0.300	0.100	0.600	1.410	0.590	0.103	0.307	0.991
0.333	0.000	0.667	1.461	0.658	0.000	0.342	0.995

* Actual fiber length (weighted by number /length) of flock sample measured with MorFi analyzer.

The right diagram compares the average length weighted by number obtained through calculation and measurement. In this case the calculated values are marginally higher with the exception of the sample containing 30 % flock of 1.0 mm. Without fail the deviation in fiber length is below 0.05 mm which is well below 5 % error.

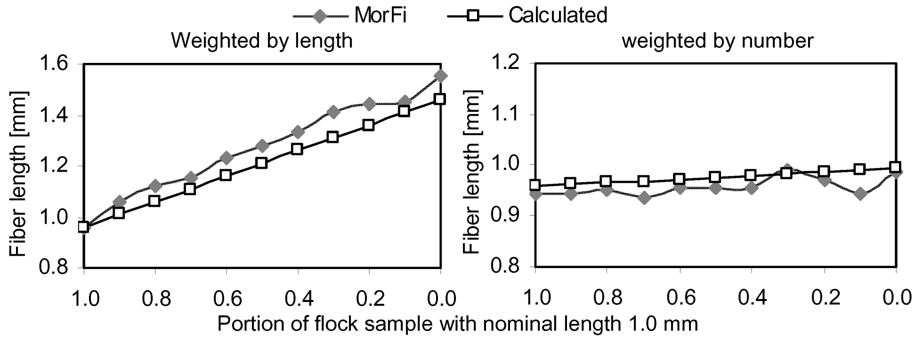

Figure 5 Comparison of calculated and measured fiber length

The results prove that the MorFi analyzer is a feasible instrument for analyzing short fibers. The measured fiber lengths are in good agreement to the calculated values. The system can evaluate a high number of individuals (approximately 10,000), in a rather short time (10 to 15 min) resulting in a high statistical reliability. Boundary effects, striking fibers or insufficiently dispersed fibers (coupled fibers) obviously do not cause severe problems. It cannot be concluded that these effects might become more dominant at higher fiber lengths (up to 10 mm). Since the system is operated with water, disentangling of fibers is facilitated. While viscose fibers as hydrophilic material can be easily dispersed in water, synthetic polymers require the utilization of surfactants.

6. Conclusions and Outlook

This paper compares several possibilities for measuring the length of short fibers. It is demonstrated that sieving and laser diffraction are not convenient for fiber characterization. On the one hand, optical microscopy can gain reliable results and is recommended by standards, but is still very time consuming. It has been demonstrated that a fiber analyzer called MorFi can gain quick and reliable results.

Other fiber materials (synthetic polymers), longer fibers and fibers of different widths are a greater challenge and will be used for further evaluation of analyzing systems. Frequently fibers are coexistent with non-fibrous particles requiring a complex analyzing procedure. The development of optical image analysis systems is rapidly continuing and within the next years, further improvement can be expected.

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