Applications of Renewable Fibrous Materials

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Fibrous materials originating from annual plants are becoming increasingly important today. The use of long fibers is well-established in a variety of applications such as textiles, non-wovens and composite materials.

The present investigations will deal with short fibers not only accessible from the above mentioned sources but also from waste materials or man-made fibers originating from renewable materials. The different methods for grinding fibrous materials in order to obtain a specific fiber length and fiber width distribution are illustrated as well as methods for fiber analysis.

Short fibers can be used for a broad variety of purposes. This paper gives a brief literature review on the present state of the art concerning short fibers. For some selected examples own investigations were carried out. It is well demonstrated that short fibers from renewable materials have great potential for future applications.

Key words:
Natural fibers, grass fibers, renewable materials, fiber analysis, grinding

Introduction

Fibers are permanently present in our daily life. However, there exists a large variety of fibers with respect to chemical constitution, morphology, origin and use. This chapter provides a literature overview of fibers with a focus on natural fibers and presents the problems associated with the characterization of short fibers. Furthermore this section describes the most common grinding devices for producing short fibers and finally summarizes the applications of short fibers.

Fibers

Definitions

The term fiber is not clearly defined in literature. From the view of the textile industry, the most important user of fibers, a fiber is defined as a linear particle which can be processed to textiles.[1] Conventionally, fibers are divided into two groups:

– *natural* fibers and

– *man-made* fibers

Natural fibers are subdivided into crop and animal fibers. Man-made fibers are subdivided into natural materials brought into fiber form by a chemical reaction or a physical action (regenerate fibers), fibers made from synthetic polymers (synthetic fibers) and inorganic fibers. Table 1 provides some typical examples for each category.

In a more comprehensive definition, *fiber* stands for "a morphological term for substances characterized by their flexibility, fineness and high ratio of length to cross sectional area".[3] According to this definition fibers are subdivided into four groups according to their length:

– *Fiber fly*: airborne fibers or parts of fibers (light enough to fly), visible as fibers to the human eye; commonly fiber fly is an unintended by-product

– *Flock*: very short fibers, intentionally produced for purposes other than spinning (velvet-like products)

– *Staple fiber*: a textile fiber of limited but spinnable length; commonly used for textile applications

– *Filament*: a fiber of very great length, considered to be continuous; commonly used for textile applications

It is clear that natural fibers from crops belong to the category of staple fiber or flock according to the BISFA definitions.[3] Man-made fibers from renewable sources also can be present in the form of filaments.

Renewable fibrous materials

The term *renewable* is defined as "capable of being replaced by natural ecological cycles". Thus,

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Table 1 – *Classification of some selected fibers according to their chemical constitution*[2]

Natural fibers		Man-made fibers		
Crops fibers	Animal fibers	Based on natural polymers	Based on synthetic polymers	Inorganic fibers
Cotton (CO)	Wool (WO)	Viscose rayon (CV)	Polypropylene (PP)	Glass (GF)
Flax (LI)	Camel Hair (WK)	Lyocell (CLY)	Polyacrylonitrile (PAN)	Metal (MTF)
Hemp (HA)	Angora (WA)	Cellulose acetate (CA)	Polyvinyl chloride (PVC)	Carbon (CF)
Jute (JU)	Silk (SE)	Elastodiene (LA)	Polyamide 6 (PA 6, nylon 6)	
			Polyethylene terephthalate (PET)	
			Polyether ether ketone (PEEK)	
			Polyurethane (PUR)	

fibrous materials fulfilling this requirement may originate from the following sources (not considering animal fibers such as silk or wool):

– annual crops

– man-made fibers based on renewable resources (e.g.: cellulose)

– fibrous waste (from crops predominantly cultivated for other purposes than fiber production)

The most important fiber originating from annual crops is cotton. The world production of cotton in 2001 was about 21.2 million t which accounted for about 38 %, of the world's fiber production (excluding animal and inorganic fibers) of 54.0 million t.[4] From the total production of man-made fibers of 32.8 million t, only 2.7 million t (approximately 8 %) are cellulosic in nature.

As demonstrated by the data in Table 2, with a share of 80 % cotton is the dominant fiber among fibers obtained from annual crops. Among the other fibers only jute (approximately 13 % share; 3.1 million t) significantly contributes to the world production and ranges slightly above cellulosic man-made fibers.[5] The world production of all other crop fibers ranges well below 6 000 t.[5]

Furthermore, the production rates of fibers such as hemp, jute, ramie or others are of minor importance when compared to the fiber world market. However, these fibers are becoming more and more important today. For example, in Germany the production rate of hemp is expected to grow from 2 600 t in 1999 to 23 800 t in 2005.[6]

Refining agricultural products as raw material for industrial goods or the use of bio-mass for energy production are well-established research areas. In recent years the innovative utilization of fibrous residues from bio-refineries was investigated.[7] As fibers represent the major fraction of biomass in a bio-refinery its use is essential for an economically feasible operation of a bio-refinery-plant. About 5 t of fibers can be obtained from 10 000 m^2 of green land. In 2010 in Austria there will be about 150 000 ha of unused green land that can be harvested. As such, a rather large amount of these fibers can be expected in the future.

Table 2 – *World production of fiber materials originating from annual crops in 1996*[5]

Name	Code[1]	Production / million t	Portion / %
Cotton	CO	18.8	79.0
Jute	JU	3.1	13.0
Flax	LI	0.56	2.4
Sisal	SI	0.29	1.2
Coir	CC	0.12	0.5
Abaca	AB	0.11	0.5
Kapok	-	0.11	0.5
Hemp	HA	0.08	0.3
Ramie	RA	0.07	0.3
Agave	-	0.07	0.3
Other fiber crops	-	0.43	1.8
Total	-	23.8	100.0

Morphology

For all processes and procedures concerning fibers it is evident that there is a dispersed ed system consisting of innumerable individual particles. In the field of mechanical process engineering, it is common to characterize a particle by its shape and size in addition to its chemical constitution. Since particles are usually irregularly shaped, several methods are established to calculate a statistic diameter such as a Feret or Martin diameter.[8–13]

For describing the size of fibers the use of a particle size is not a proper tool. Commonly fibers are seen as regular-shaped particles (namely as cylinders). Thus, the diameter and height (i.e., the fiber length) are sufficient to describe a fibrous material. On the one hand the situation is easier since it is not necessary to define statistical size values. On the other hand, for each parameter – length and width – a size distribution function and a cumulative size distribution have to be computed.

Of course, the assumption that a fiber is cylindrical does not consider cross sections other than a circle. Crop fibers in particular may exhibit a flat or even hollow nature. Hence for a sufficient characterization of natural fibers, length and width parameters may not be sufficient. To make a statistically valid statement about the fineness of a fiber the textile industry commonly uses the fiber-denier.[14] The fiber-denier is defined as the quotient of mass and length having the unit tex (or dtex) which states the mass of the fiber in grams per 1 000 m (10 000 m).

Characterization methods

In general there are two possibilities for size analysis. The first method consists of measuring and counting individual particles and the second method involves separating particles into size fractions and measuring the quantity of each fraction.[15]

However, the most common methods for particle characterization fail when analyzing (short) fibers. Sieving analysis is not able to distinguish between length and width of a fibrous particle. Most common image processing techniques are designed to determine particles and fail to compute length and width of a fiber. To evaluate fiber morphology, optical microscopy is a useful tool but quantification is labor-intensive. Moreover, short fibers cannot be characterized by common methods used by the textile industry (e.g., Lenzing VIBROSCOPE) since their length is too short.

Grinding of fibrous materials

Man-made fibers can be produced in the desired dimensions and in particular, the desired diameter. In contrast, natural fibers commonly need to be sized to a specific dimension for a certain application. The grinding of fibers is a mechanical process similar to grain size reduction of powders. However, due to the extreme non-spheroid habit of fibers the process is more complex and one can distinguish between a length reduction (cutting) and a diameter reduction (fibrillation). There exist many types of mills that use different forces to rupture particles and fibers.[16–18] In the following section, three types of mills that can be used for grinding fibrous materials are briefly described.

Cutting mills

Materials showing rubber-elastic (entropy-elastic), visco-elastic or plastic properties exhibit a large deformation prior to their rupture or fragmentation.[17] In order to minimize the required deformation and thus the grinding energy, it is necessary to keep the deformed volume as small as possible. This prerequisite can be achieved in a cutting mill which predominantly exerts cutting forces. This type of grinding technique is commonly applied to fibrous materials.

One can distinguish between cutting mills fed with filaments which in turn are not suitable for natural fibers and cutting mills which can process staple fibers and are thus suitable for crop fibers. Cutting mills are widely used in the industry for treating materials such as plastics, wood, pulp and paper, textile and leather, fat and wax, domestic waste, paperboard and empty barrels or cans. It is well established that cutting mills are used for the pretreatment of materials prior to further grinding by disc mills or impact mills.

Each cutting mill consists of several knives where one can distinguish between knives mounted on the rotor (so called flying knives) and knives mounted on stationary blades (so called bed knives).[19] The amount of knives and the diameter and speed of the rotor are key parameters for the functionality of the mill. As such, these parameters need to be optimized for each dimension reduction task.

Impact mills

The size reduction in impact mills is based on impact forces. These forces are caused by collisions between a particle and a hard surface or between two particles.[20] There exists a large variety of impact mills each optimized for certain size reduction tasks.[19] However, materials to be ground in an impact mill must not be abrasive, not too hard, not too heat sensitive (melting or softening point >80 °C) and not too large in size but should be brittle.[19] Hence, in general fibrous materials from crops are more suitable for size reduction using impact mills than man-made fibers. Usually fibers need to be sized (length reduction) in a cutting mill prior to the impact milling process.

Disc mills

The forces in a disc mill are tension as well as shear and abrasive stress.[18] They consist of two discs, one fixed (stator disc) and one rotating (rotor disc), having a narrow gap between each other (about one-tenth of a millimeter). Often the discs are equipped with sharp cams or teeth (so-called toothed disc mill) and are especially convenient for fibrillation of organic products such as wood, paper, pulp, leather, rubber or cork.[17]

Applications of natural short fibers

Conventional fiber reinforced plastics contain glass, aramid, carbon or others as reinforcing fibrous material and their use is well established for several decades.[21] In order to decrease energy consumption and environmental pollution synthetic fibers synthetic fibers are increasingly suppressed. The processing of natural fibers is different from that of conventional fiber products and thus manufacturing processes need to be adopted.[22,23]

Today natural fibers are commonly used as webs formed from long fibers in the automotive industry[24] or can be processed in the form of endless rovings[25] by pultrusion. In order to produce fiber reinforced plastics by injection molding short fibers need to be used.[26] This technique is of no industrial importance for natural fibers today.[27]

The most important market for natural fiber reinforced composites is the automotive industry. In the medium-term range the European automotive market will demand natural fibers of 40 000 to 70 000 t.[6] The main reasons are:

– Lower density and thus lower mass; consequently a reduced fuel consumption of the respective vehicle[28,29]

– Less energy consumption as well as less waste production in the course of the manufacture process[28,29,30]

– Better thermal disposal due to significantly lower ash content and higher caloric value when compared to glass fiber composites; recycling does not require special technologies[31]

– Lower costs of natural fibers when compared to fibers conventionally used for reinforcement purposes.[32]

In spite of these advantages several technological and economical obstacles have to be solved, such as:

– More complex fiber preparation and thus higher production costs[22,23]

– Problems with the adhesion fiber – matrix; the use of coupling agents to increase the affinity fiber – polymer is necessary and currently the topic of intensive investigations[33–36]

– Sensitive to moist atmosphere resulting in a loss of strength.[37]

The second most important market for natural fibers is insulating materials.[6] This market is expected to grow up to 28 000 t natural fibers within the next 5 to 10 years.[6] The third field of possible applications for natural fibers is cellulose such as special papers for cigarettes, bank notes, as well as for standard papers. The latter can be seen as an "emergency" market for low quality and excessive production.[6]

Experimental details and results

Samples

This paper presents results of grinding experiments using Lyocell fibers and two samples of grass fibers. The Lyocell fibers represent an example of man-made cellulosic fibers. The Lyocell fibers used for the experiments had a denier of 1.3 dtex (i.e., 10.4 μm) and a length of 39 mm and were supplied by Lenzing AG, Austria. The two grass fiber samples were obtained from a green bio-refinery. The following two samples were investigated:

– Lucerne (Medicago sativa): harvest 2002, green (not fermented), pressed one time; dried (water content w = 5.8 %)

– Wheat grass (Triticum aestivum): harvest 2002, green (not fermented), pressed one time; dried (water content w = 5.6 %).

Grinding device

For this study a cutting mill was used as the grinding device. The respective data are summarized in Table 3.

Characterization methods

Within this investigation the following methods of analysis were used to characterize the ground fiber sample.

Table 3 – *Data of cutting mill used for experiments*

Manufacturer	Alpine – Hosokawa, Germany
Type	Rotoplex 20/12 Ro
Number of flying knives	2
Number of bed knives	3
Diameter rotor	200 mm
Width rotor	120 mm
Engine power	4 kW
Maximum rotation speed	1500 min^{-1}
Sieves (situated either close or far from the rotor)	round holes: 0.5 to 3.0 mm conidur holes: 0.5 to 3.0 mm
Fan power	1.5 kW
Maximum airflow	200 m^3 h^{-1}

Sieving analysis

A conventional sieving machine manufactured by Retsch, Germany, type "Vibro" was used. The mesh sizes of the sieves were 40, 63, 80, 180, 280, 400, 500, 630, 800 and 1000 μm. The sieving time was 10 min. The results were computed as particle sizes distribution and cumulative distribution (weighted by weight).

MorFi fiber analyzer

The MorFi fiber analyzer[38,39] was developed by the Pulp and Paper Research & Technical Centre (CTP), St Martin d'heres, France in order to characterize fibers (width, length, curl, and kink), shives and fine elements in the pulp. An aqueous suspension of the fiber sample is pumped through a measuring cell and a high resolution digital camera takes pictures. These pictures are analyzed by the software system and the length and the width are calculated using the skeletonization process by scanning each segment path. The software can determine fibers within the range of 0.1 to 10 mm for length and 5 to 75 μm for width. For both length and width ten size intervals can be chosen and the software will compute the size distribution function and cumulative size distribution either weighted by number, length or area. In addition, average length and width (arithmetical or weighted by length) are available.

Light optical microscope (LOM)

For this method of analysis, a light optical microscope, manufacturer Leitz, Germany, was used. The images were taken by a digital photo camera using reflected light.

Grinding of man-made cellulosic fibers

The Lyocell fibers were ground at a rotation speed of 1500 min^{-1} and air flow rate of 200 $m^3\ h^{-1}$. The sieves were situated close to the rotor (approximately 5 mm). The mesh size (conidur holes) was 0.5 and 1.0 mm respectively.

The samples were analyzed using the MorFi fiber analyzer. For comparison purposes a cut-flock sample (Viscose) manufactured by Guillotine-cutting was analyzed. The cut-flock sample had a denier of 1.7 dtex (i.e., 11.9 μm) and a length of 0.5 mm and was supplied by REO Flock & Faser GmbH, Pfullingen, Germany.

Figure 1 summarizes the results using the MorFi analyzing unit for two ground Lyocell samples. It is evident that the length distribution of the ground samples is much broader compared to that of the cut-flock sample. Although the mesh size of the sieves was significantly different (0.5 and

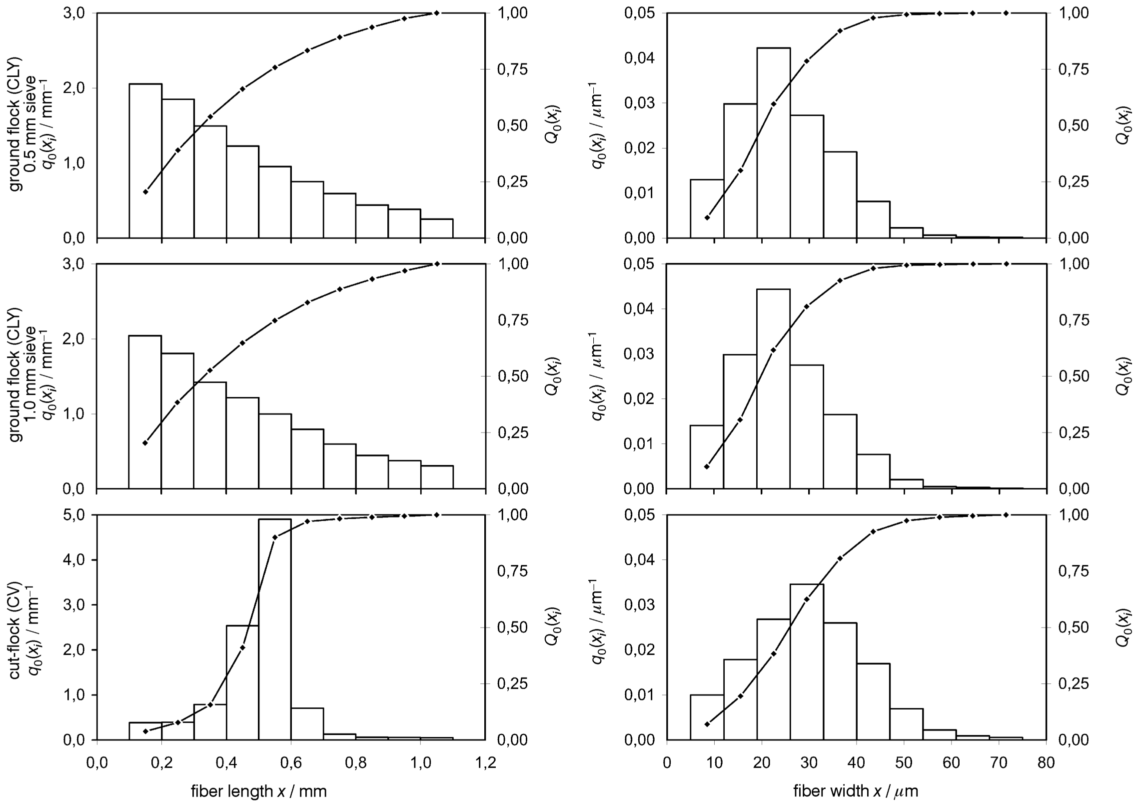


Fig. 1 – *Fiber Width and Length Distribution Function and Cumulative Site Distribution of Ground Lyocell and Cut Flock*

1.0 mm) the length distribution function of the Lyocell samples is about the same. Also, the arithmetical length of the samples (computed by the MorFi analyzing unit) is de facto the same (0.43 and 0.44 mm). It can also be estimated that a certain fraction of fibers will be well below 0.1 mm and thus, cannot be analyzed by the MorFi unit. In contrast the length distribution function of the cut-flock sample is very narrow, showing an arithmetical length of 0.5 mm which is exactly the value stated by the manufacturer.

The fiber width distribution of the man-made fiber samples is rather narrow but somewhat wider than one would expect for a man-made fiber. This is caused by a strong swelling of the cellulosic fiber. For the Lyocell samples a diameter of 24.9 and 24.4 μm was determined. This means a diameter enlargement of about 135 to 140 % of the value in the dry state (10.4 μm). The swelling is even more dramatic in case of the cut-flock (Viscose) which shows diameter enlargement of about 150 % from 11.9 to 29.7 μm. This extreme strong swelling is higher than expected and significantly exceeds values reported in the literature of about 80 to 100 % for Viscose[40,41,42] and about 65 % for Lyocell.[43]

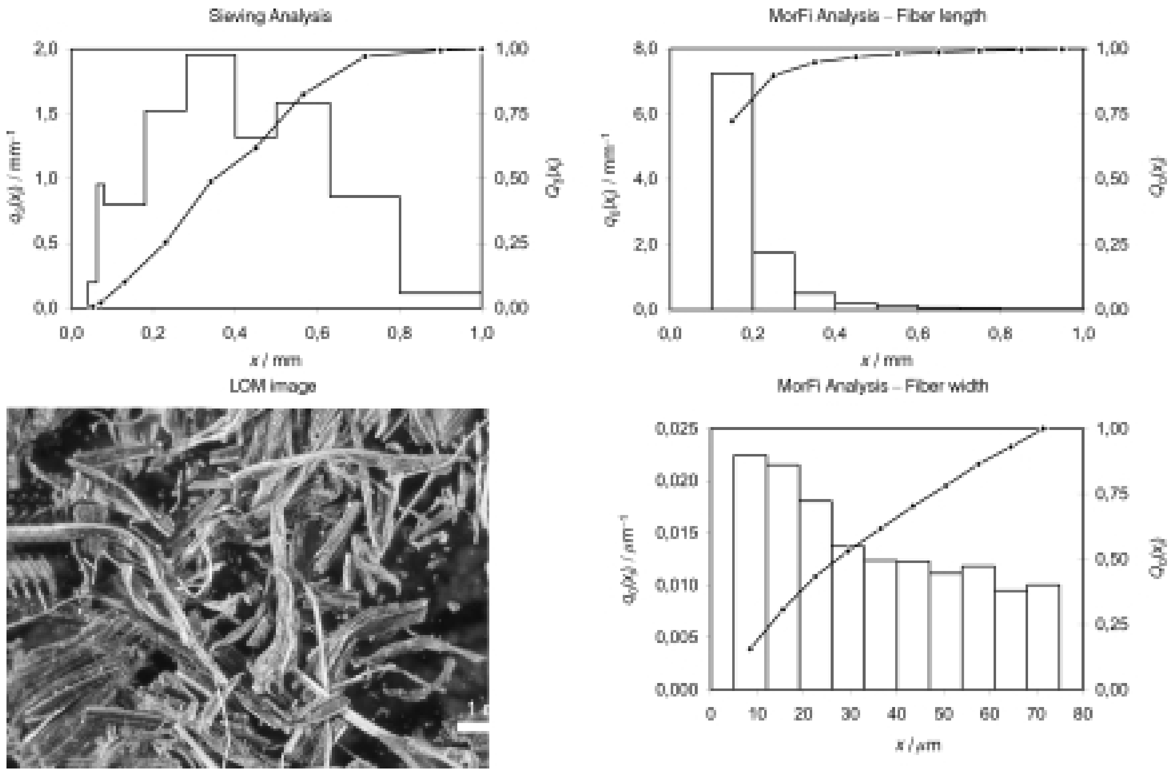
The results demonstrate that the MorFi analyzing unit is a practical tool for the size determination of short man-made cellulosic fibers. It has, however, to be considered that the measured diameter is significantly larger than the actual diameter (dry condition) due to swelling and most probably other, not yet defined, influences. Another restriction of the MorFi analyzer is the fact that fibers below 0.1 mm are not considered as fibers and thus are not accounted for in the computation of length distribution, cumulative distribution, and average length.

Parallel to the MorFi analysis, sieving analysis was carried out. It was not possible to compute any data for this analysis since the main fraction of the fibers remained on the top sieve forming a kind of web.

Grinding of grass fibers

Both samples were ground in the cutting mill using a mesh size of 1.0 mm (conidur holes, close to the rotor). Due to the grinding process a dramatic change in the morphology occurred. The inhomogeneous starting material consisting of agglomerates of fibers of very different size turned into an optical homogenous material. However, it turned out to be quite difficult to analyze the ground material as discussed below.

In the case of the wheat grass sample (Figure 2), the LOM image shows a distinct fibrous character. The fibers are, however, not round but flat consisting of several fibrils. A considerable amount of fibers show a length above 1 mm. The sieving analysis contains only a small portion in the coarsest



F i g . 2 – *Comparison of Sieving Analysis, MorFi Analysis and LOM Images of Wheat Grass Sample.*

size class (0.8 to 1.0 mm) indicating that the long fibers (>1.0 mm) passed the 0.8 mm sieve.

The results using the MorFi analyzer are quite different. For the fiber length no considerable signals were determined above 0.4 mm. This is in contrast to the LOM image and cannot be explained by the different weighing methods (sieving: by weight; MorFi: by number). It is highly likely that the fibers are larger than 75 μm in diameter, the maximum range for the MorFi analyzer, and were not taken into consideration. This is well demonstrated by computing the fiber width distribution. There is no distinct distribution but it can be estimated that there are considerable portions of fibers being either below 5 μm or above 75 μm (measuring range of MorFi analyzer).

In case of the lucerne sample (Figure 3), sieving analysis indicates a similar morphology (slightly larger particles). However, the LOM image demonstrates that the morphology of the sample is completely different. A large portion of the sample does not exhibit a fibrous character at all but contains plates of about 1 mm edge length. These particles most likely contribute to the considerable portion of particles in the 0.8 to 1.0 size range. Again the MorFi analyzer is not able to recognize the (fiber) particles as fibers. No considerable amounts of fibers were determined to be larger than 0.4 mm. As for the wheat grass sample, there is no distinct maximum width distribution indicating a large portion of fibers to be outside the measuring range (5 to 75 μm).

Summary of results

The results demonstrate that it is quite difficult to characterize short fibers which are not accessible by textile characterization methods. In particular, the grass fibers are far too inhomogeneous.

Sieving analysis is not a proper tool for fibrous products. It is not possible to characterize man-made cellulosic fibers since the fibers do not pass through the sieves. Although very short fibers and more particle-like samples (lucerne grass) can be sieved, the method does not distinguish between length and width.

The MorFi analysis is capable of characterizing man-made fibers if their length and width is within the measuring range. Especially for ground fibers the minimum length restriction of 0.1 mm may lead to false results if a certain amount of fibers are not measured. For grass fibers this method is not a proper tool for characterization.

The LOM images give the best characterization of fibers in particular for crop fibers and grass fibers since a detailed morphology (not only length and width) can be obtained. However, for a quick and inexpensive characterization this method is not practical.

In order to develop the grinding technology of fibers from crops to an industrial scale it is not only necessary to improve the process itself but it is necessary to also develop a proper characterization method. This method should be based on a system similar to the MorFi analyzer but allow for a wider range for both length and width.

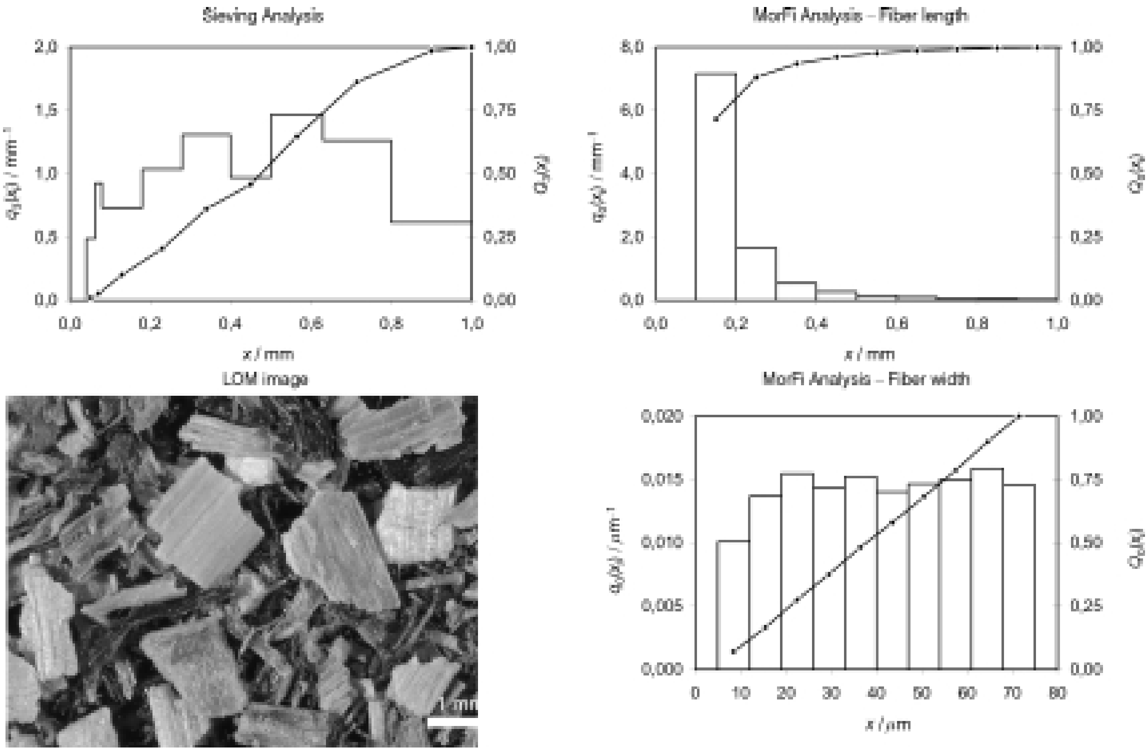


Fig. 3 – *Comparison of Sieving Analysis, MorFi Analysis and LOM Images of Lucerne Sample.*

Conclusion

Renewable fibrous materials have a great potential today. Within the years ahead, a significant increase in the use of renewable fibrous materials in the automotive industry can be expected. This increase will be mainly centered on short fibers.[6] The engineering of short fibers needs to be improved and a quick and precise characterization method is needed. The most important crop fiber for the automotive industry is flax and hemp.[6]

A recent investigation of the application of grass fibers such as Switchgrass and Fescue as reinforcements for polypropylene composites, showed that these materials could be used as a substitute for natural (e.g., hemp, flax, wood) as well as inorganic (e.g., glass) fibers.[44] However, in that paper the fibrous habit of the Switchgrass and Fescue was not considered. Only the mesh size was reported. It is very likely that fibrous reinforcement materials exhibit a better reinforcement behavior and also the use of grass fibers, which remain as by-product from "green bio-refinery", may be an interesting alternative as reinforcement materials. However, the grinding processes as well as characterization methods need to be further developed.

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