

OPTIMISATION OF METHODS OF FIBRE PREPARATION FROM AGRICULTURAL RAW MATERIALS

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1 - INTRODUCTION

For profitable West European agricultural production of non-food crops, and especially fibre crops, it is essential to be competitive with the cheaper fibre raw materials produced from other regions in the world. The availability of bulk quantities of products like jute and sisal, and the potential large scale production in Eastern Europe of flax or hemp is forcing the agro-industrial production in the EU into an specialised niche market. Traditionally, the flax fibre production in Western Europe for high quality linen has been able to cope with competing imported raw materials, because of its high standard of quality. The concentration in the past decades of the conventional linen promotion on the fashionable textile market has, increased the dependency of the sector on a strongly fluctuating market segment. One way for EU agriculture to compete on the world market of lignocellulosic fibres is to supply high quality raw materials with an added value for the user. This can only be achieved when the qualitative aspects have been defined in detail for each specific end-use.

2 - FIBRE PRODUCTION CHAIN

During the various steps of ligno-cellulosic fibre production from agricultural raw materials, a large number of factors affect the final product quality. Each stage in the production chain affects the added value of the end product. Depending on the required performance of the fibre in the end-application, the production and processing cost will determine its economic feasibility. There are big differences between quality and economy of the dedicated production of fibre crops or specialised energy crops and valorisation of agro-residues.

The traditional flax-linen production chain is composed of many labour intensive steps in which the products are produced with utmost care to maintain the fibre quality standards. The production scale and market price of linen fibre is dominated by the demand on the fashion sensitive textile market. The revenues obtained from the long fibre production should be paying for the main part the costs of the whole production chain.

The lower qualities of fibre, which are produced as residue from agricultural production have to compete with cheap wood fibre on the market for paper & pulp, fibre board and composites. Both hard- and softwood fibres are utilised on large scales for refining and pulping. Only about 11% of the world's virgin cellulose pulp is made from non-wood sources (mainly straw, bagasse, and bamboo). In the EU, US and Canada practically no non-wood pulp is used¹.

Another low end market outlet for fibrous bio-mass is in energy production. Promotion of renewable energy and bio-mass conversion plants by the various European governments is targeting for a doubling of the contribution to the energy production over the next 10 years. It has to increase from less than 10% of the total energy consumption now to over 20% in 2010. The efficiency of heat exchange and energy generation in existing systems for bio-mass conversion may not be optimal, but it is contributing to the policy of enhanced use of sustainable energy. Since bio-mass utilisation for energy production is still expensive, compared to fossil energy sources, the so called 'cascade principle' is considered. Combined fibre extraction and energy production may increase the revenues.

A large number of novel end-uses for cellulosic fibres have been identified² and demonstrated to be technically feasible. Growing awareness of the ecological aspects of consumption and production of raw materials has been leading towards the trend of sustainable utilisation of the natural resources. For example in automotive industry the use of cellulosic fibres as renewable raw material is receiving much attention and shows much promise. Another potential market for lignocellulosic materials is an increased interest for renewable materials in building and construction applications. Complex building

regulations and standardisation in the different EU member states, combined with different legislation on the use of building materials on national levels, make the introduction of novel products on this scattered and conservative market difficult. However, implementation on larger scales requires commercial challenges as the driving force for production chain development.

The production chain for fibre crops can be divided into three main links: agricultural production – fibre processing – and utilisation. Despite the interdependency of the different links in the chain, the interests may be divergent.

Agricultural production

- Breeding genetics
- Growth agronomy: soil, climate, weed & pests control, fertilisers
- Harvest / storage maturity and handling

Fibre processing

- Fibre extraction retting, braking, decortication,
- Fibre preparation cleaning, hackling, carding, refining, extrusion, steam explosion, chemical / biochemical treatments, etc.
- Fibre processing spinning, weaving, finishing; compounding,

Fibre application

- Use performance
- Disposal reuse and recycling, incineration / degradation

The quality aspects of the traditional flax linen production chain has been subject to detailed study over the past decades. In order to replace the customary subjective organoleptic methods of qualification, a number of instrumental methods have been developed³. Introduction of those instrumental methods as standards of qualification is essential for the development of a certified fibre production chain, especially for the novel industrial end-uses like fibre reinforced composites or building materials. Each application, however, will have specific demands on the performance of the raw material, which may be confidential industrial information. The quality control in the fibre production chain is requiring much attention and geared activities of all parties concerned.

2.1 – Agricultural fibre production

2.1.1 – crop improvement

In agricultural production, increasing the crop yield has always been an important way to raise the income of the farmers. Besides improvement of yield, which is particularly relevant for energy crops, in flax breeding research the emphasis has been primarily on the agronomic aspects of the fibre production chain, such as disease and lodging resistance. Ever enhanced production efficiency has been achieved by mechanisation of soil tillage to harvest, optimised use of fertilisers and crop protecting chemicals (herbicides, pesticides).

The quality assessment of the crop at these stages of production are based on organoleptic expertise. The straw quality rating is expressed in length and thickness, homogeneity and branching, but also in colour and feel or shape of roots and stalks.

For breeding purposes the assessment of long and short fibre yields for the different varieties is insufficient. Since the value of the fibre is determined by its utility for textile use, the desired properties for textile processing have been aimed at. Dominant parameters in textile applications are fibre strength and fineness and refinability. For traders the homogeneity of a batch is another very important aspect, which is related to the colour and lustre of the fibre, but also to the regularity of the fibres. Morphologically flax varieties may differ in the number of bundles per stem or fibre cells per bundle. The filling degree of a fibre cell or lignification of the cell walls are largely dependent on the ripeness. For non-textile applications the quality parameters are diverse, but the currently used organoleptic methods cannot be accepted by industrial consumers, who are accustomed to ISO-certified products.

Quality assessment techniques are needed, that can be used quickly, require small samples and are applicable in an early stage of the production chain, preferentially in early stages of crop development for selection purposes in the breeding programme. Therefore it is important to understand the molecular (genetic) regulation of the cell wall biosynthesis at the different stages of development and the factors influencing the fibre properties. This requires joint efforts in multidisciplinary fundamental and applied research.

2.1.2 – economics of fibre crop production

Minimum cost of production for fibre flax in the Netherlands⁴ by the traditional methods of harvesting (pulling, dew retting) and processing for long textile fibres, have been calculated to amount over € 3000 per ha. With an average yield per hectare of 8 ton straw, the minimum straw price for the farmer then should amount at least € 380 per ton in order to be profitable.

Adding to the production costs of linen textile fibre are the different specialised tools and equipment designed for parallel harvesting and processing of fibre flax.

Per hectare the crop will be yielding on average 1 ton of long fibres, 1 ton of short fibres (tow), 3 tons of shives and 1 ton of linseed (table 1) bringing globally an income less than € 2000 per ha (including costs of deseeding and fibre extraction processes; scutching = € 115 /ton).

Table 1. Average yields of fibre flax production per hectare in the Netherlands.

	Yield ton/ha	Market price ⁵ € per ton
Straw	7-9	
Long fibre (scutched)	0.7-1.2	1.300-1.700
Short fibre (tow)	0.4-1.0	100- 150
Shives	3	20- 40
Linseed	1.1-1.5	150- 190

The gap between production costs and market value of the raw materials has been bridged in the past decades by financial support from the European Commission, to secure and stimulate the agricultural production of linen textile fibre. The EU support regulations for fibre production are expected to change dramatically in 2000. This means that without subvention the economic production of flax in the EU will be no longer feasible if the yields and/or market prices do not rise. Stimulation of other market outlets with a more efficient production chain, however, could even bring enhanced economic activities.

2.1.3– economics of energy crop production

At the low end of the fibre production chain the utilisation of its energy content for the production of bio-energy has received much attention. Ligno-cellulosic fibres, ranging from agro-industrial waste bio-mass (verge grasses, food crop residues, processing wastes) to specially grown energy crops like miscanthus, phalaris or switch grass, have been studied for their potential as source for the production of 'green energy'. However, the negative economic values for the costs of waste disposal, makes valorisation of waste streams attractive. Large variation in composition and contamination makes combustion complex and requires the development of flexible processing units in which tar and ash formation can be controlled. Due to the seasonal variation in supply, a highly organised logistic chain needs to be set up.

As long as the price for crude oil remains around the level of 15 US\$ per barrel, bio-energy production cannot compete cost wise without governmental intervention*. To make the production of bio-mass

* For generation of energy 1 ton oil (~ € 100) is equivalent to approximately 3 tons of ligno-cellulose feedstock (dry matter e.g. straw). High yielding energy crops may provide 15 tons dry matter per ha and an income of ~ € 500 per ha, which should balance the cost of production. In the case of agro-residues like wheat straw, which may yield 3 tons/ha, valorisation of surplus straw as fuel is adding €

profitable, additional income has to be generated in most, like cascade utilisation of cellulosic fibres and recycling, valorisation of residues (silica, tar).

2.2 – Fibre extraction process

The fibre extraction process has a major impact on the final product quality. Besides differences in variety, the influences of climatic conditions and soil, ripeness at harvest and harvesting method, the retting procedure is of crucial importance to the processability of the long bast fibres (flax, hemp, kenaf). The available methods of retting are relying on the biological activity of micro-organisms from the environment, degrading the pectic polysaccharides from the surrounding tissues and liberating the bast fibre bundles.

2.2.1 – Retting

The most commonly applied retting process is dew retting on the fields. The dew retting process is based upon the action of fungi, which grow on the carbohydrate rich tissues. Primarily the fungi utilise the easy accessible pectic polysaccharides in the phloem, which will liberate the bast fibre bundles from the cortex and xylem. The influence of uncontrollable weather conditions (humidity, sun) on the process is extremely high. In some years part of the crop cannot be gathered in before over-retting occurs, due to exceedingly over wet climatic conditions. In those cases the fibre strength will be affected because of microbial attack of the cellulose. Direct correlation has been demonstrated between retting time, cellulose polymerisation degree and fibre strength .

In under-retted samples the pectin content is too high for good separation of bast fibres and shives, which will give substantial problems in the further processing (decortication, cleaning and combing, fibre separation).

Water retting utilises bacteria to degrade the pectic polysaccharides in the Under the anaerobic conditions, bacterial growth is coupled with production of a complex mixture of volatile compounds (butyric acid, H₂S, methane, etc.). Water retting has been abandoned for flax processing in the past because of the pollution it caused to the surface waters and the foul smell given off. Only on a limited scale warm water retted flax fibre is still being produced. The objections of additional costs for labour, energy (for heating and drying) and pollution control (smell and waste water production) are compensated by a better controllable process and reliable product. However, the price payed for warm water retted flax fibre is not much higher than a good dew retted product.

Enzyme retting has been introduced (Flaxzyme, Novo) but has yet to lead to commercialisation of the process. The same restrictions for warm water retting apply for enzyme treatments, except that the process can be much faster and cleaner (less nauseous compounds are produced). The enzymes will be specifically directed at the degradation of the pectic substances, without the risk of degradation of the other structural polysaccharides (hemicellulose and cellulose) of the fibre cell wall. On an experimental scale good quality fibres can be produced. The major disadvantage of the process is that for the catalytic action it is necessary to bring the enzymes into close contact with the substrate. Therefore high concentrations of purified enzyme and (mechanical) pre-opening of the straw are necessary. So far the costs of the enzyme preparation in relation to its retting efficiency and investment costs (equipment and waste water treatment systems) as compared to dew retting have been limiting factors for commercialisation.

So far no satisfactory solution for the risky field (dew) retting procedure has been introduced on larger scales. The simplicity of the dew retting process and relative low investment costs have caused its maintenance as a dominant technology. A number of alternative technologies have been evaluated to reduce the harvest risks due to the field retting. Additional arguments for it have been a reduction of costs or pollution and the guaranteed supply of a (more) constant quality raw material.

2.2.2 - Chemical retting

In the sixties a combined mechanical and chemical process for fibre extraction from green decorticated flax fibres (VITNO) had been developed in the Netherlands for textile application. Due to the large economic problems in the European textile industries in the seventies, the project was abandoned.

100 per ha. Straw in other uses (animal bedding and horticultural use) has a market value up to € 60 per ton.

Some of its concepts, however, should receive renewed interest. Selective chemical degradation of the unstable pectic polysaccharides is a possibility. The use of green decorticated fibres or semi-retted fibres in non-textile applications has been considered in recent years.

2.2.3 – Thermo-mechanical fibre opening

Most of the novel industrial applications for cellulose fibres do not require parallel processing of the fibres. A number of thermo-mechanical fibre opening processes such as refining, extrusion, and steam explosion, have been developed to produce feedstock for different end-uses. In non-woven, paper, fibre boards and polymer composite application different quality demands require adapted processing technologies to provide the specific properties. Properties like absorbency, fibrillation, strength or fibre dimensions are all most strongly affected by the processing conditions, and may be less sensitive to the variable raw material input. The added value of fibre performance in the non-textile end-products may not be demanding the high quality standards, developed for textile use.

2.3 – Fibre processing and qualification

Besides crop improvement and fibre extraction, processing into the final product further affects the performance of the fibre. Understanding the relation between the properties of a raw material and its production process is fundamental to the improvement of quality. The different stages of the production process have their influence on the end-product price and performance. The whole chain approach is necessary to be able to see where the most promising improvements can be made at the lowest costs. When, for example, fineness is an issue for the application of a fibre, genetic improvements may not yield the necessary results, when no adequate fibre extraction technology is available. The production costs may become too high for a specific end-use even if the qualitative criteria are met. On the other hand, if an efficient fibre processing technology can be developed, which substantially upgrades the fibre quality for a specific end-use, irrespective of the variable properties of the input raw material, all the efforts for crop improvement are in vain. So a tailor made solution for each different commercial development for fibre applications should be aimed at.

2.3.1 – Textile fibres

For the textile application of bast fibre crops, such as flax and hemp, the parallel handling and processing during the whole production is considerably adding to its costs. In addition, the competition on the textile market for pure linen fabrics is even more difficult because of the relatively slow spinning process for the long bast fibres as compared to cotton spinning. Alternatively, shortened, cottonised flax is finding an outlet in blended yarns. These blended yarns with cotton, wool, or synthetic fibres, are giving a linen look to the fabrics. In this case not the highest quality long fibre flax is required to produce the yarn and secondary fibres (hackling tow) may find application.

An increasing interest in coarse (hand spun) textile products from fibre hemp (imported from Nepal or China) can be observed. In order to grow fibre hemp for textile application in the EU, a completely new approach will be required, very similar to the alternative flax processing. It will be very difficult to compete on this market, since the whole production chain will have to be developed, including seed propagation and spinning technology.

The production in the EU of kenaf fibre for textile production will not be an option, since the quality of yarns and fabric will be comparable with low grade jute, which is in abundance available on the market at very low prices.

2.3.2 – Non-wood pulp

Paper products are composed of a mix of virgin pulps, recycled pulps and additives, depending on its end use. In the EU the total fibre mix consists of 56% wood pulp and 44% recycled fibres. Non-wood bast fibres of flax and hemp are only used on limited scale for speciality papers (bank notes and safety paper, tea bags, cigarette paper)¹. The relatively high price for hemp pulp of € 3000 per ton, as compared to € 500 for bleached softwood can be explained by the relatively small production volume (~5000 tons per year) and speciality niche market. A modern wood pulp mill produces up to 1 million tons per year. For logistic reasons annual fibre crops should be processed on relatively small scales (approximately 20.000 tons / year)⁶. Small scale pulping mills have more difficulties to cope with the environmental legislation. Most of the non-wood fibre pulping plants in the EU have therefore been closing in the past decades. Novel methods of processing like extrusion pulping show much promise for efficient production of a diversity of annual fibres. Small scale black liquor recovery may become feasible, when methods of lignin extraction and valorisation have been developed.

The woody core (or shives) of hemp and flax, which are composed of rather short fibres (~0.6 mm), have been demonstrated to be easily processable and to give good yields of a pulp resembling hardwood and straw pulps. The growing market for hardwood pulps in printing and writing paper may offer novel outlets for valorisation of residues of flax and hemp fibre production¹. In the past decades the possibilities for kenaf pulping (whole stem, separated bast and core) have been investigated in the US up to industrial production trials. Good results have been reported, however, commercialisation is still not realised.

2.3.3 – *Non-textile fibres*

The major change in the production of non-textile fibres as compared to the traditional linen production is that parallel handling of the raw material can be abandoned. Only for unidirectional composites, the oriented fibre bundles, as in hackling sliver, may be applied. For most other end products the extreme fibre length can be reduced and random oriented fibres further processed. In many cases the shorter tow fibres can be utilised. Although currently the price of flax tow is relatively low and stocks may have build up to more than 50.000 ton⁷, the expected increase in industrial demand for short fibres in the coming years will have a dramatic effect on availability and price.

One way for alternative (non-textile) end-uses to anticipate on the raw material supply and to reduce the raw material costs is to utilise long and short fibres, which are not separated in the scutching process ('lin-total'). This necessitates alternative fibre extraction and cleaning processes, capable of efficiently removing the adhering plant tissues (cortex, shives), without affecting the bast fibre properties.

The presently used fibre processing technologies are optimised for textile production and are far from optimal with respect to other applications. The effects of the mechanical treatments (braking, scutching, hackling, stretching) on the performance of the fibre, for example, in composites may be causing inferior performance. Much damage to the fibre structure is occurring because of the mechanical forces required to separate the different tissues. The harsh conditions may be causing rupture of cell walls and strongly adhered impurities as remnants of xylem and cortex tissues. The fibre structure and composition will have its effects on the performance in the application such as water absorbency and durability (biodegradation), fibrillation and strength.

Another phenomenon occurring in flax processing is the formation of so called kink-bands in the cellulose fibrils⁸, which are the weak spots of disturbed crystalline domains. These are caused by an impact force horizontal on the fibre direction or over bending, which occurs frequently during braking and scutching. In order to prepare fibre reinforced composites with the highest possible mechanical properties the introduction of the weak spots in the fibre has to be avoided.

2.3.4 – *Bio-mass for energy*

The production of energy from bio-mass has been focussed on the generation of the highest output of power. Problems with the variable composition and combustion efficiency of bio-mass have been recognised. An alternative approach is the adjustment of the combustion process to the raw material, which will prevent excessive tar formation or agglomeration of silica. Prior extraction of proteins and minerals will reduce the NO_x emission or prevent corrosion of the installation. Therefore the input raw material should be characterised in relation to its combustion performance. Methods to monitor its composition are required to adjust the air supply or regulate the temperature. Such an adjustable process may not yield the highest power, but the production of residues can be minimised.

2.3.5 – *Qualification / ISO certification (ASTM)*

One of the most important demands of industrial users of fibres is a guaranteed supply of sufficient quantities of qualified raw materials. The ISO-certification is an issue of general interest. The qualification systems currently in use for most fibre crops are still based on the subjective organoleptic assessment of the traders. Instrumental methods for quality control of fibrous raw materials have been developed for cotton, which has been introduced world wide. This operating system cannot be transferred as such for other fibre sources. The reliability of instrumental assessment has been shown to be poor in a round robin test within the FAO-flax working group⁹. Standardisation of methods is urgently needed. Recently, a new working group within ASTM has been giving attention to the flax qualification.

Definition of the raw materials and products will help to overcome the existing barriers for introduction of cellulosic fibres on new markets. Concepts like technical fibre, fibre bundle, elementary fibre, and fibrils should be better defined.

For the on-line quality control of fibre production, methods utilising computer operated optical control systems should become available. Adaptation of existing systems in other branches of (agro-)industry seems possible.

3 – Conclusions

Much has been achieved over the last decade in the exploration of the potential markets for different fibre resources, although a real breakthrough still has to come. As a renewable raw material fibre crops do have strong marketing arguments as 'eco-efficient' products for development of sustainable consumption and production. Diversification of the market for fibre products in automotive industry, geotextiles, building and construction materials, paper & pulp, bioenergy, etc., are all in the picture, if the quality control of the agro-industrial production chain has been organised according to ISO-standards and supplies of specified products to industrial buyers can be guaranteed.

A number of aspects in quality control and improvement in the production chain of cellulosic fibres from agricultural produced fibres do need attention. In the whole life cycle of production and use - from breeding to combustion and disposal - the upgrading of quality and enhanced performance may be achievable by dedicated technology and logistic control systems. A multidisciplinary research and development approach is essential to bridge the gaps between the farmer and consumer. Within the EU fifth framework programme 'Quality of life and management of living resources' such a cluster of activities is fitting exactly and needs to be organised. A concerted action of the whole agro-industrial production chain will be necessary to attain the targets of sustainable consumption and ecological safe production of the scope of renewable products based on cellulosic fibres.

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² J.E.G. van Dam, G.E.T. van Vilsteren, F.H.A. Zomers, I.T. Hamilton and W.B. Shannon – 'Industrial Fibre Crops' – study on: increased application of domestically produced plant fibres in textiles, pulp and paper production and composite materials (EC DGXII – 1994 EUR 16101 EN).

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⁴ Personal communication with Mr David Kasse, Hoofdproductschap Akkerbouwgewassen, The Hague, The Netherlands; average of recent 5 years Dutch Fibre flax production data.

⁵ Data calculated in € from Vlasberichten, April 1999 (1 BEF = 0.02479 €).

⁶ H.J. Bakker and M.J.J.M. van Kemenade – Papier uit hennep van Nederlandse grond, eindrapportage van vier jaar hennep onderzoek: samenvatting, conclusies en aanbevelingen (1993).

⁷ C.E.L.C. – Bilan matières U.E. campagne 1997/98.

⁸ H.L. Bos, and H.M. Donald – 'In situ ESEM study of the deformation of elementary flax fibres', *J. Mat. Sci.* 1999, accepted.

⁹ R. Kessler – Results of the round robin test on quality measuring by the FAO European Cooperative Research Network on Flax. Proceedings 4th regional worksop on Flax, Rouen, 25-28 September 1996.