

NONWOVENS FROM RECYCLED WASTE

Alfred Watzl

Introduction

So far the reuse of textile waste (or rather secondary raw materials) has been considered under an economic aspect only. It was primarily the cost factor (reduction of production costs by saving of raw materials) that determined recycling viability.

Today waste utilization becomes more and more of a political issue in connection with environmental protection and avoidance of waste formation, but of particular note is that the recyclability of waste materials has gained great importance.

The growing importance of used raw materials with respect to ecological acceptability and need for non-polluting processing technologies themselves together with the demand for a higher degree of recyclability of the products, is already reflected in the current development of appropriate machines. The recycling process, as well as processing of recovered chips for the fibre production, concerns both waste accumulation during production in addition to the textile and nonwovens products themselves which are returned to the production cycle as secondary raw materials after use.

The development of new nonwovens under the heading of optimum recyclability is therefore being pursued already.

Avoidance, Reduction and Recycling of Waste

Avoidance and reduction of waste formation have to be mentioned first for being the most important issues of all these considerations. In this respect, the nonwovens producers are challenged.

When considering the problems caused by waste, the following order of precedence should be observed:

- avoidance
- before reduction
- before recycling
- before disposal

There are various possibilities to avoid the formation of waste which should be made use of more often.

Recycling means that both production waste and used materials are returned to the production cycle (see Figure 1).

First it can be said that a recycling technology is uneconomic when for reprocessing of the waste more energy and raw materials would be required than for making the same quantity of new products. Naturally the technology itself must not affect the environment more adversely than waste disposal. Finally a market must exist or new markets must be opened up for the products made of waste fibres. When dealing with these problems, however, the situation of the limited raw materials supplies must always be borne in mind.

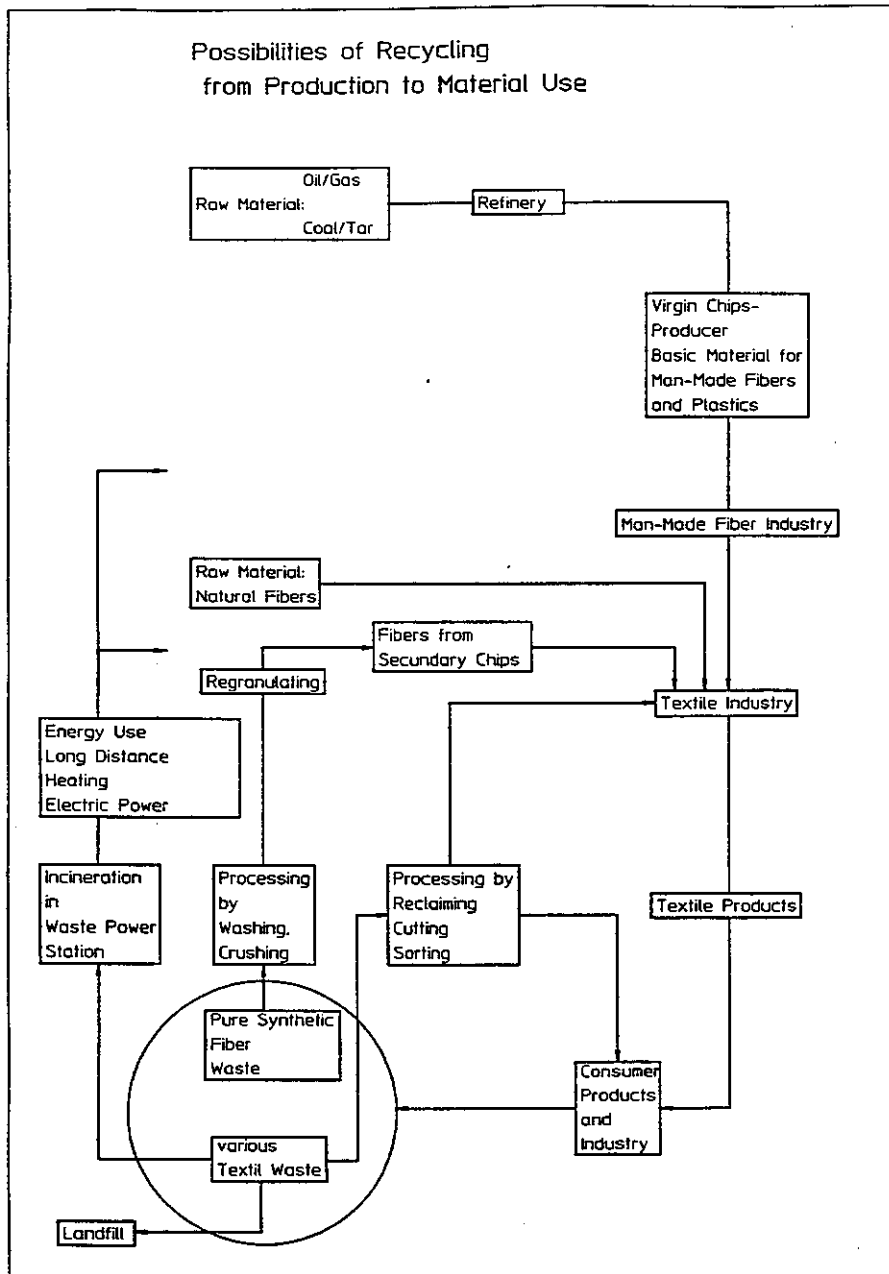


Figure 1: Possibilities of recycling from production to material use

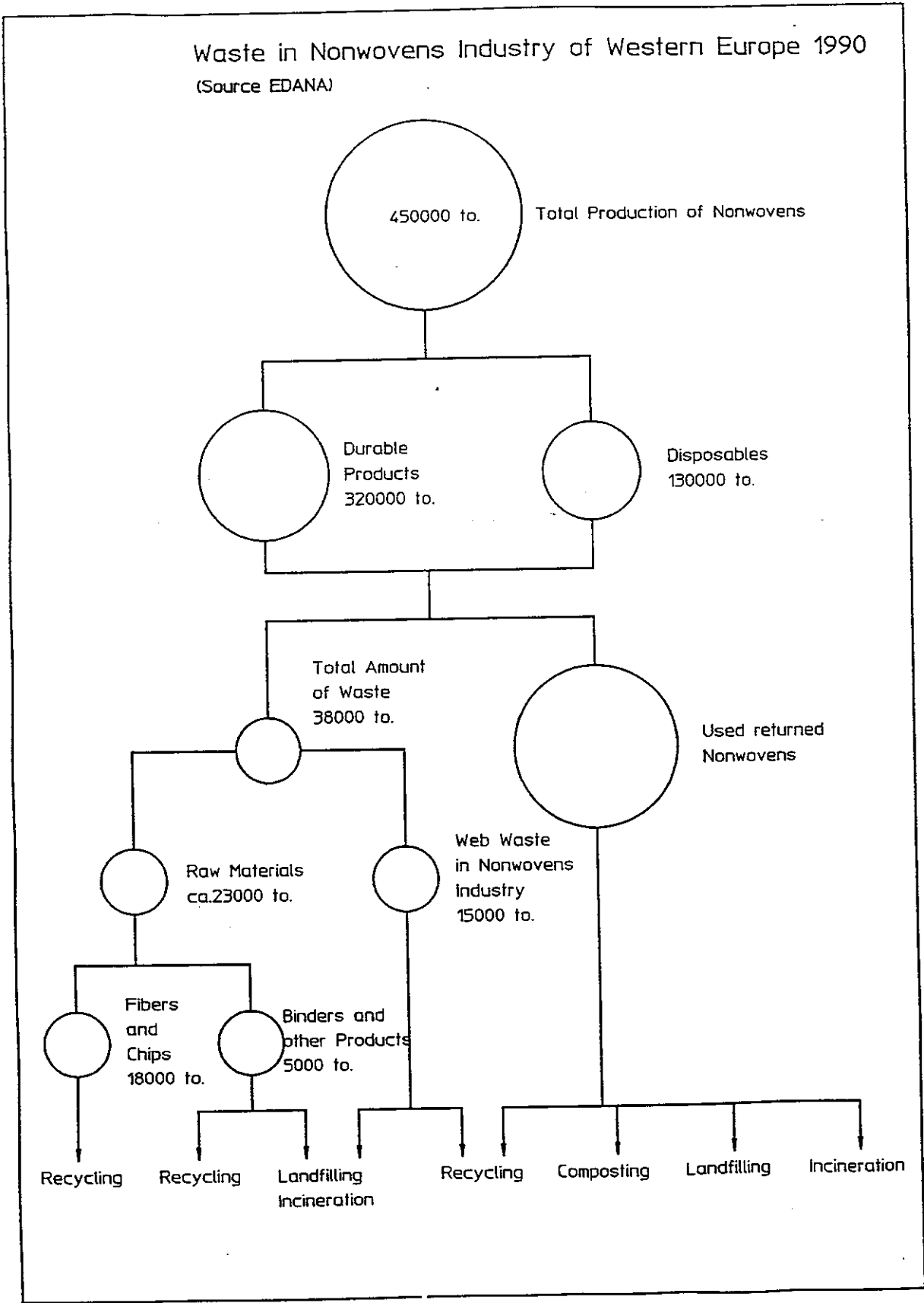


Figure 2: Wastes in the nonwovens industry of Western Europe, 1990 (source EDANA)

Disposal and Recycling of Specific Textile and Nonwovens Products

In case of nonwovens products, the main distinction has to be made between disposable and durables while for textiles only the last group is relevant.

Data on the waste situation in the nonwoven industry of Western Europe reveal how these two groups are split up (see Figure 2).

For the group of disposables, possibilities regarding the avoidance of waste formation, recycling, composting and depositing on land-fills will be listed. In this connection, the biological degradability plays an important role.

Synthetic polymer scientists have been striving for the past 50 years to make their organic products less oxidizable, hydrolysable and less biodegradable in order to make them durable. So it is not surprising that some re-engineering is necessary to reverse the process and make these materials less durable and therefore more disposable and less persistent in the environment. A material fulfils the public's expectations of biodegradable properties only if it is finally turned into CO₂ or biogas, water and residual biomass within a reasonable time.

The term durable products covers all textiles and nonwovens which have been designed for certain applications and are disposed of after being used or are returned to the raw material cycle. It mainly depends on their composition which possibilities come into consideration.

These products also comprise thermally bonded and chemically bonded nonwovens which are used, among other things, in the automobile and the furniture industry, for carpet production, for ready-made textiles and as coating substrates. Moreover, they comprise all the classic textiles like woven fabrics, knit goods, yarns, filaments, man-made fibres, natural fibres, mineral fibres, linters etc.

Waste material accumulating in the respective production processes is mostly recycled today, either in the production plant itself or in the production of other textile goods if direct recycling is not possible for quality or other reasons. However, there are no reliable data on the percentage ending on garbage dumps. This also applies to used textiles which are either recycled or deposited on landfills.

It will be appropriate to divide the textile and nonwovens waste according to the individual production stages and the possible waste processing technologies. This means that different technologies will be required for processing of the various waste groups.

The classification might look as follows:

Waste from the man-made fibre industry:

drawn and undrawn, crimped and uncrimped tow waste, cut and uncut, made of polyester (PES), polyamide (PA), polyacrylic (PAC), polypropylene (PP), viscose;

	filament waste
Spinning waste, yarn waste:	all yarn waste produced after spinning
Clothing waste:	waste from woven and knit goods
Nonwovens production waste:	thermally and chemically bonded, light-weight webs, needled webs, coated, uncoated
Carpet mill waste:	needle felt, tufted carpet, cut waste, coated, uncoated
Used textiles:	here sorting is required, possibly also cleaning and dedusting.

Uses and Ranges of Application for Recycled Textile Fibres

In the following, a few examples of possible uses in the nonwovens, carpet, building, textile, agricultural and paper industries are given, but new ranges of application are opened up every day (see Figure 3). The various uses mainly depend on how the textile and nonwovens waste is formed during the individual process stages. This will then determine the possible recycling process. Consequently, various technologies are required for reprocessing.

Nonwovens industry:	Automobile industry: insulating webs for sound and heat insulation, hard-pressed parts for floors, side and seat linings, trunk compartment, luggage dump etc., bottom felts for carpeting, stitch-knit nonwovens (Moliwatt, Molivlies).
Furniture industry:	Mattress covers, mattress webs, bottom webs for seating in furniture, upholstery material, wadding material. Wiping cloths Needled webs.
Carpet industry:	Bottom felts for carpeting.
Building industry:	Sound and heat insulating webs, filter products, nonwoven coating substrates, footfall sound insulation. Textile shreds as filling material for insulating webs, as aggregate for textile concrete in road construction.
Textile industry:	Spinning waste, blended yarns or 100% waste yarns for spinning to the DREF or rotor spinning process (wiping cloths,

blankets, home furnishings), comforters made of acrylic knit goods waste.

Agriculture industry: Covering webs, seed carrier webs.

Paper industry: Wearing felts for paper production.

Crude felts for bitumen roofing felts.

On the whole, there are two technologies used for textile waste reprocessing as described below.

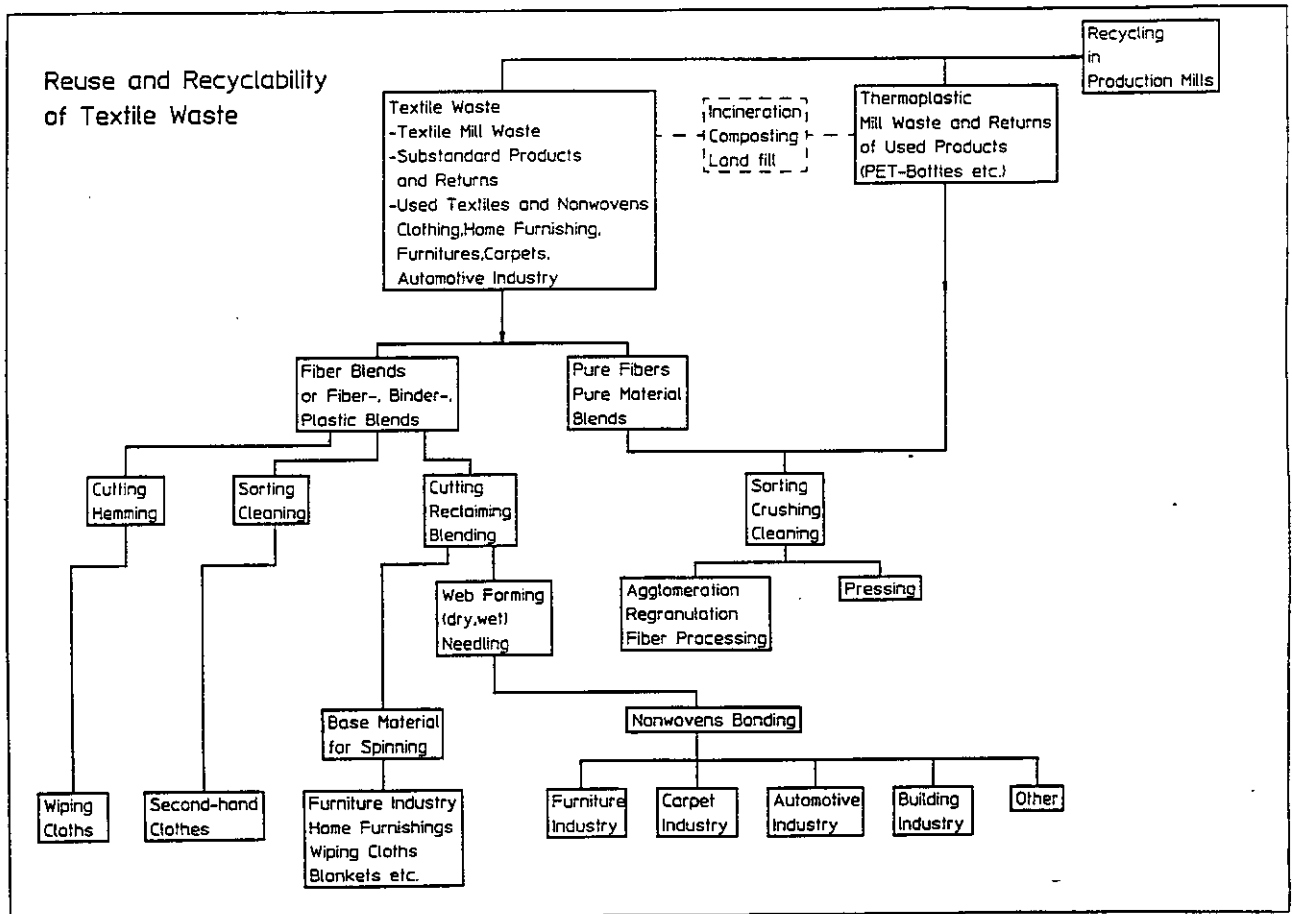


Figure 3: Reuse and recyclability of textile waste

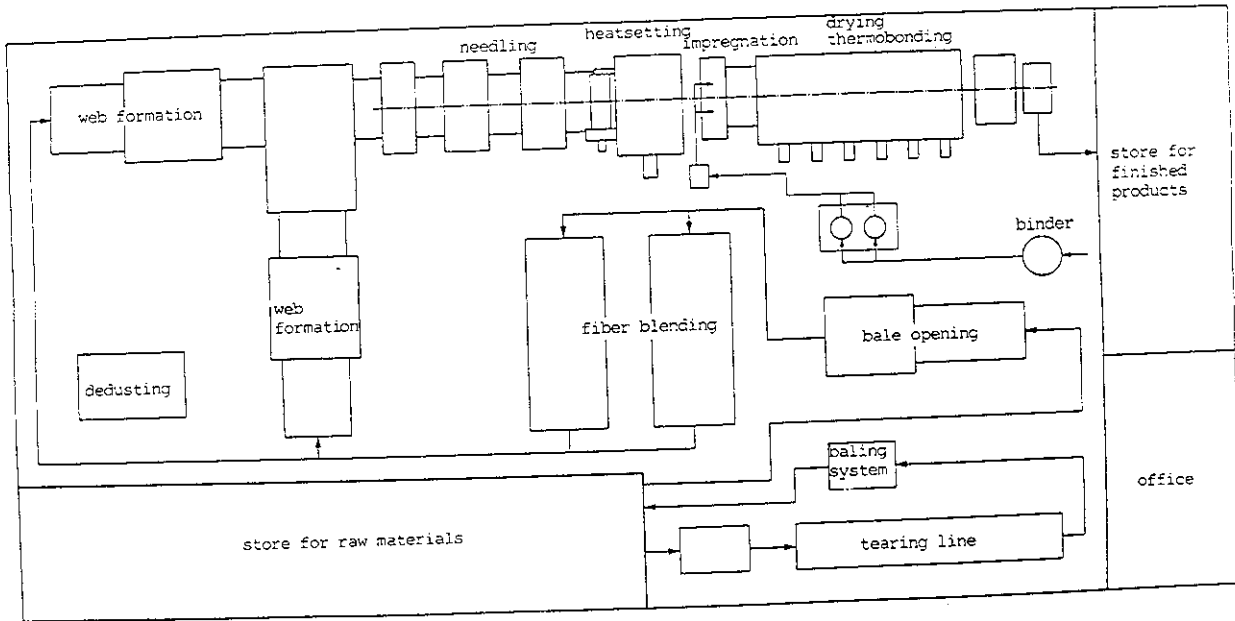


Figure 4: Multipurpose line for waste fibre webs for heatsetting, thermobonding or binder bonding.

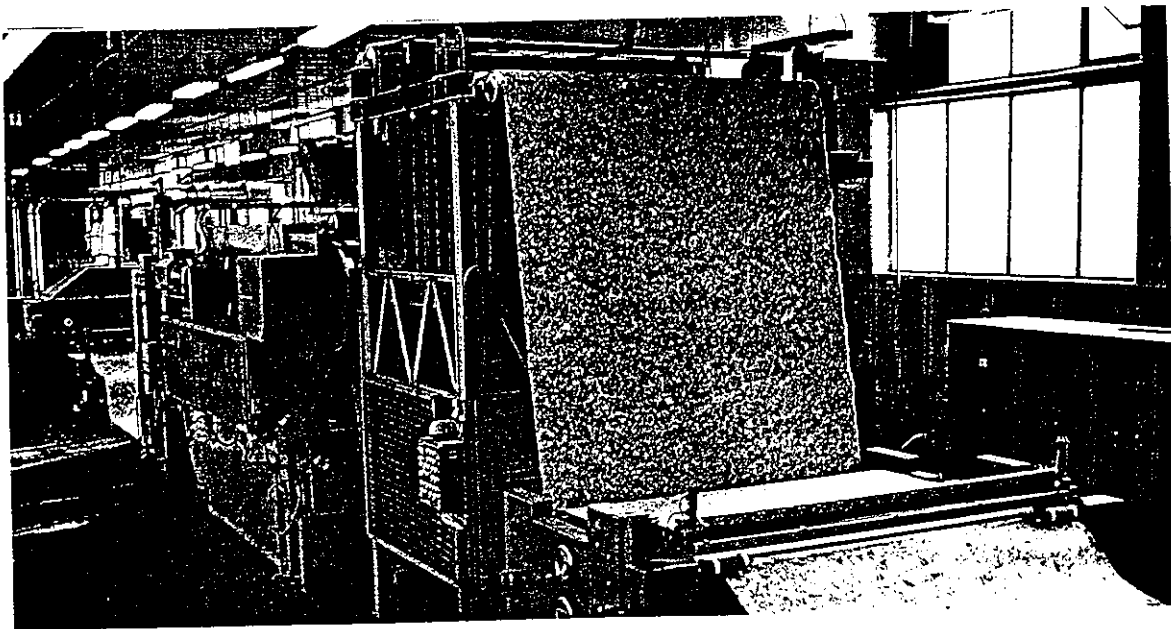


Figure 5: Fleissner waste fibre-to-nonwovens recycling line.

Mechanical Reclaiming (shredding, unravelling)

The mechanical process is used chiefly for fibre blends (man-made fibres, natural fibres) as regranulating is not possible.

Among all known possibilities for processing of textile waste, unravelling on shredding lines is the most common and most economic solution. The shredded fibres are used for the production of various kinds of nonwovens.

A complete processing line for the production of nonwovens from waste fibre comprises the following line components (see Figure 4):

- Fibre cutting and shredding (reclaiming) section
- Fibre blending section
- Web formation section
 - : dry-laid webs: - aerodynamic web formation
 - carding
 - : wet-laid webs: - wet forming machine
- Bonding by needling
- Bonding line
 - : heat-setting
 - : thermobonding
 - : foam impregnation
 - : drying and curing of binding agents

Together with other renowned textile machinery producers, Fleissner supplies complete processing lines for recycling of waste fibre into simple and high-quality nonwovens as shown in Figure 5.

One example describes the use of secondary fibre for products in the automotive industry (insulating webs, moulded parts).

Concerning bonding of waste fibre webs, there are various possibilities. In practical operation, several processes are used to increase the number of possible applications and improve the characteristics of needled nonwovens.

Heatsetting: A method for bonding by heat treatment without melting of fibre is the heatsetting process. It is particularly used for fibre blends of 100% PES which are further finished as coating substrates. Shrinking during the subsequent impregnation with binder agents is avoided when the nonwovens are heatset at a temperature of 220 -230°C

For this purpose, the web is retained in special lateral holding devices to keep it from shrinking. Heatsetting at the same time increases the web density and has a positive effect on the breaking strength and elongation-at-break of the nonwovens.

Heatsetting is done on a Fleissner perforated drum line operating on the flow-through

principle. The flow-through heating system has the highest possible heat transfer coefficients.

Thermobonding: Thermal bonding of fibre blends made of waste fibre and a small percentage of fusible fibre can be used for manufacturing many well known market products based on nonwovens, such as industrial and household wiping cloths, insulation webs for the automobile sector, fibrefill webs for the furniture industry, etc.

To increase the number of possible bonding points, the melt adhesive bonding fibres are usually mixed with fibres of somewhat finer metric counts.

Heat treatment is carried out at a temperature at which the melt adhesive fibre or parts of bicomponent fibres become viscous or melt. The bonding polymer flows by surface tension and capillary action, mainly to the inter-fibre contact points where it forms bonding spots.

For heating to the required temperature, flow-through heating systems are used. Because thermobonded webs are very sensitive when heated, uniform air flow and temperature across the working width are essential to avoid irregular thermal shrinkage or uneven bonding and web thickness. In most cases, lines with 1 and 2 drums are used, although in some cases the lines comprise several drums as well.

In order to smooth the nonwovens surface or obtain a particular density or thickness, a pair of pressure rollers (heated or cooled) can be installed directly at the discharge of the bonding line.

Binder bonding with efficient impregnation and drying: Bonding of waste fibre webs with binding agents is required for various reasons:

- to optimize the breaking strength/elongation-at-break
- to increase the wear resistance
- to allow mouldability of needled webs
- to allow stabilization and dyeing at the same time.

Binder-bonded nonwovens made of waste fibre are used among other things in the automobile industry, in the furniture industry and as floor coverings.

Today, chemical binders are exclusively used. All existing binder types can be employed. For reasons of costs, but also because of their particular properties, preference is given to binders based on styrene-butadiene.

Since the binders, as mentioned above, are aqueous dispersions, water is always applied together with the solids during impregnation; this water has to be removed in a subsequent drying stage.

Fleissner has introduced a foam impregnation process which can also be employed for pre-

bonded, pre-needled materials. Due to its advantages, it has been adopted for a wide range of applications and gained great importance. For the drying process, perforated drum dryers are predominantly used.

Regranulating/Use of Pure Waste

Waste material from textiles, technical webs and nonwovens made of thermoplastic and synthetic fibres and plastics can be put to a new use by melting and remoulding. The great advantage is that at the end of this chain new fibres are produced which can be used for the same or other applications. However, this process can only be used for largely pure waste material.

For natural fibres, this process is out of the question.

All thermoplastic waste such as polyethylene, polypropylene, polystyrene, polyamide, polyester, polyvinyl chloride, etc. as well as all sorts of compound materials can be processed to granules on agglomerating plants.

An example of modern recycling for the production of high-quality carpet fibres describes regranulation of PES from recovered beverage bottles.

For nonwovens production also it seems better, from the current point of view, to use pure materials since they are easier to recycle.

As pure materials often do not meet the many requirements of the intended application, considerable developmental work will be required to find equally good substitutes. Work on this problem is progressing in many fields and particularly in the automobile sector, practicable solutions have already been found.

One example is the recyclable nonwovens mat for mouldable inner parts of automobiles, made of a blend of PES fibre and PES bicomponent fibre. Those parts produced on the basis of pure polyester can be completely regranulated. The fibre newly spun from these granules can be reprocessed into a nonwovens mat after adding a small amount of new fibres.

There are also other examples of products easy to recycle.

Description of a Fibre Production Line for the Textile and Nonwovens Industry

For the production of fibre from recycled fibre material, compact processing technology is the most suitable. It must be characterized by high flexibility and allow production of a greater variety of PES and PP fibres.

The compact spinning technology is a continuous process for the production of staple fibres which covers all process steps for the supply of granules, through drying of granules, dyeing, spinning, drawing, crimping and cutting up to the compressed fibre bales in one operation.

Summary

After years of reducing, removing or dismissing environmental pollution, the ecological demand for avoidance of waste and environmental pollution now comes to the fore.

This paper has shown that great efforts are made to reduce or avoid waste formation by developing new nonwovens products which are pure polymers and therefore can be returned completely to the material processing cycle.

Concepts for recycling of textile waste, which will still be produced in great quantities, already exist although examples of possible solutions and the respective processing technologies have been presented.

RECYCLING ZEFTRON CARPETS

Ian Wolstenholme

Introduction

Carpet Recycling Market: In 1993, 1.3 million tonnes of fibre for carpet manufacture were produced in North America. This fibre was used in about 2.5 million tonnes of carpet. Of this amount, approximately 70 percent replaced existing floor coverings, resulting in approximately 1.7 million tonnes of replaced carpet. The vast majority of this material ended in landfills. Several approaches for diverting this material from landfill are under investigation. These include converting carpet into secondary products such as plastic wood, extruded profiles, or extenders and reinforcing agents in other products, and incineration/cogeneration. These markets are, however, limited by competition with other recycled polymers and, in the case of incineration/cogeneration, public resistance to siting.

Carpet Construction: Complicating the recycling issue for carpets is their multicomponent construction. A typical broadloom carpet with a face weight of 850 gm⁻² consists of approximately 50% by weight face fibre. Most of the face fibre produced in North America is nylon, both 6 and 66, with smaller amounts of polyester, polypropylene, wool, cotton and acrylic fibres also used (see Table 1). The typical broadloom carpet also consists of 12% polypropylene primary and secondary backings, and 8% styrene-butadiene latex adhesive. Additionally, the carpet consists of about 30% calcium carbonate filler (see Table 2).

Table 1: Carpet face fibres in USA

Nylon 66	40%
Nylon 6	30%
Polypropylene	20%
Polyester	10%

Table 2: Carpet construction

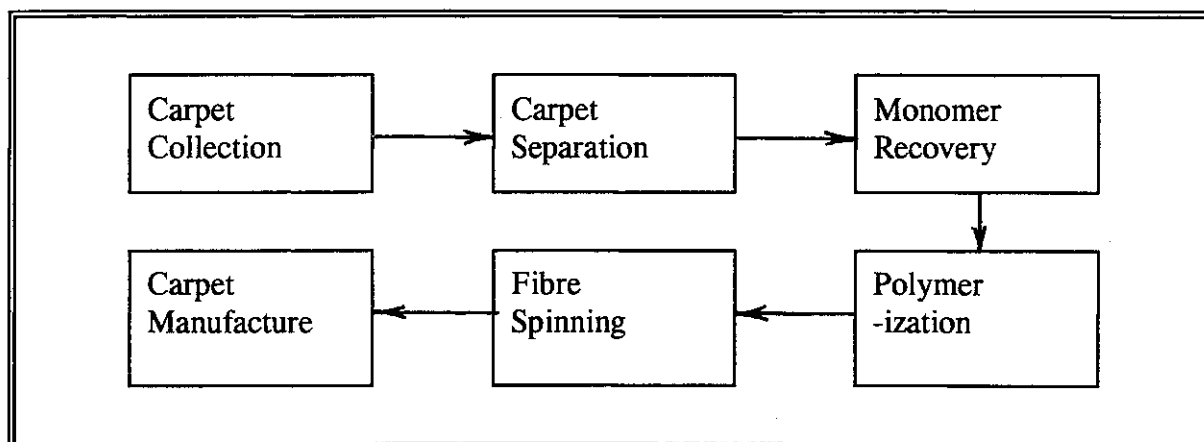
Face Fibre:	50%
Backing:	12%
Latex:	8%
Latex Filler:	30%
Note: Based on Typical 850 gm ⁻² Carpets	

Other backing materials in use include jute, polyester, and nylon 6. Polyvinyl chloride, ethylene vinyl acetate, and polyurethane backing substrates are also used. Finally, the carpet contains small amounts of dyes, pigments, antistatic fibres, and surface treatments.

Closed Loop Recycling

Chemical recycling of nylon 6 is not a new concept. BASF has been recycling post-industrial nylon 6 at its Enka, North Carolina and Arnprior, Ontario locations for more than thirty years. However, the material recycled has had a high nylon 6 content. Only recently has BASF developed the technology for chemically recycling materials with lower nylon 6 contents. For carpets this is a six-stage process. First, carpets are collected and identified as to face fibre content. The face fibre is then mechanically separated from the backing, adhesive, and filler. The resulting nylon is depolymerized back to its monomer, caprolactam, and purified for use. New nylon is then formed from the recovered caprolactam. This nylon is then spun into fibre and finally, new carpets are produced (see Table 3).

Table 3: Carpet recycling process



Chemical Recycling is really a very simple concept. It begins by converting old product back to its basic raw materials. Once this is accomplished, contaminants and impurities are removed from the crude raw material. Finally, the purified raw material is reconverted or reconstructed, back into new product.

The major advantage of this type of recycling is that the new product is the same as the original product. There is no loss in product quality by the proper implementation of a chemical recycling approach, unlike methods which simply reprocess old product into another form.

Decomposing Tree in Forest Example: Examples of chemical recycling are all around us. A decomposing tree in the forest is an example. At its most fundamental level, this tree is slowly being converted to carbon dioxide, water and other basic nutrients.

In the presence of seed and sunlight, these same basic raw materials are recombined to produce a new tree. Although the tree is new, it consists entirely of used raw materials.

A tree is a renewable resource, unlike plastics derived primarily from crude oil; therefore the chemical recycling of plastic materials will become even more important in the future as oil supplies deplete and assume a higher value.

Polymer - Monomer - Polymer: Nylon 6 polymer can be chemically converted back to caprolactam, its monomer; in fact this technology has been practised by BASF for 30 years. Nylon 6, unlike nylon 6.6, has the advantage of consisting of only one monomer and this means that the contaminant removal process, for monomer purification, can be more economical and less complicated than if the converted polymer consisted of two monomers.

Simple filtration and distillation processes are all that are required to purify the crude single monomer intermediate stream. Two monomer mixtures involve more complex separation technologies as would be the case for nylon 6.6 and polyester, for example.

Once pure caprolactam is obtained, it can be converted to new nylon 6 polymer once again by conventional polymerisation and so the process can be repeated over and over.

The BASF Carpet Recycling Process

BASF's carpet recycling process consists of six steps. The first two, Collection and Separation, are part of the infrastructure that will have to be developed for the carpet industry in future. BASF is taking the lead by working with others to put the necessary infrastructure in place.

As mentioned earlier, the third step - Monomer Recovery of Nylon 6 scrap - has been practised at BASF facilities for over 30 years. This technology only requires modification to handle the more highly contaminated nylon 6 material obtained from waste carpet.

The final three steps - Polymerization, Spinning and Carpet Manufacturing - are already well developed and it is envisioned that no major changes need to be employed in the carpet

recycling concept.

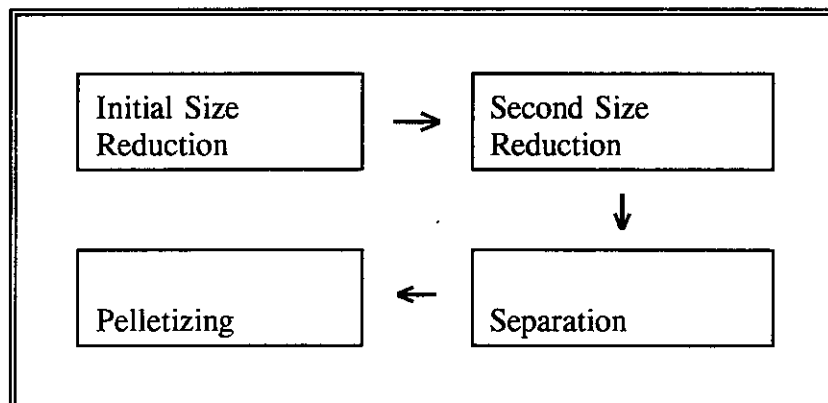
One problem in waste carpet recycling is the need to identify the face fibre. A simple test, which specifically identifies nylon 6 face yarn, can be done in about 1 minute by determining whether a carpet tuft dissolves in a dilute solution of hydrochloric acid.

This test will only dissolve nylon 6 as nylon 66, polypropylene, polyester and wool remain undissolved in the 4.4 Molar HCl solution.

Separation: The second step is Separation. Shred-Tech, in cooperation with BASF, is designing a carpet separation system. Shred-Tech has had over 15 years of experience in designing and fabricating material handling equipment for recycling everything from scrap metal appliances to polyester drinks bottles.

Table 4 schematically shows the separation process in which the first component is size reduction; at this time no special pretreatment is envisioned for the waste carpet. Initially a shredder reduces the carpet to strips and further size reduction is accomplished with a rotary cutter.

Table 4: Carpet separation process



Using an air classifier, which works by pulling a slight vacuum through a rotating cage, the small, low density face yarn fibres are pulled through the cage and collected in a receiving cyclone. The larger, higher density latex backing particles are thrown outwards and drop by gravity into the collection vessel below the rotating cage.

Typically, each constituent is of equivalent weight in the separated piles of face yarn and carpet backing fractions from a 950g m⁻² carpet.

The separation step thus generates the first side stream of the carpet recycling process. End-uses for the enriched carpet backing stream are currently under investigation.

In order to economically transport the separated carpet face yarn, to a monomer recovery

plant, densification is essential. A Condux Plastcompacter is one type of sintering mill capable of performing the densification. Over 200 of these mills are in operation worldwide, densifying materials such as yarn and film scrap. This essentially, is a sintering mill which works when frictional force is converted to heat causing the fibrous material to densify into pellets or granulate; thermal degradation of the polymer is avoided. Sprout-Bauer also manufactures a pelletising mill that works by using the same principle. Sintering increases the bulk density of the separated face yarn to about 70% of that of polymer chip.

Typical separation efficiencies for a typical 50:50 face : backing 950m² are shown in Table 5.

Table 5: Relative carpet component separation efficiencies

	Feed	Face Yarn	Carpet Backing
Nylon 6	50%	80%	20%
Calcium Carbonate	30%	10%	50%
Polypropylene	12%	6%	18%
Latex	8%	4%	12%
Rate Ratios	1.0	0.5	0.5
Nylon 6 Separation Efficiency = 80%			

In initial feasibility tests, roughly 80% of the carpet's face yarn was recovered with the separation process so far described. The separated face yarn stream contains about 80% nylon 6 and 20% backing materials, while the carpet backing stream contains about 20% nylon 6 and 80% backing materials.

Monomer Recovery: The third step, in BASF's carpet recycling process, is monomer recovery and this is the heart of the chemical recycling approach. Nylon 6 depolymerization and caprolactam purification, from nylon 6 process waste and scrap materials have been economically practised for many years. Adapting the technology to old nylon 6 carpet materials is a matter of building upon what BASF and in fact other nylon producers already do.

Essentially the densified nylon 6 face yarn is simply highly contaminated N6 scrap material, which because of the chemistry and process technology involved, can be converted to purified raw material, that is caprolactam.

The first part of the monomer recovery step is hydrolytic depolymerization of the densified nylon 6 carpet face yarn produced in the separation process followed by extraction and distillation to generate monomer of acceptable priority for re-use as mentioned previously.

Summary

In summary, BASF is developing the collection and separation infrastructure required to put a closed loop recycling process in place by working with others in the recycling industry. This part of the process must become a business in itself, working with the existing carpet industry.

Secondly, BASF is building on its technological base, extending the inherent recyclability of the nylon 6 polymer to more highly contaminated carpet materials. Here BASF has submitted patents for its work in the recycling of nylon 6 based carpets.

Finally BASF is ensuring the quality of future recyclable nylon 6 carpet by utilizing a higher quality chemical recycling approach in order to solve the long term waste carpet problem.

COTTON WASTE RECLAMATION

Ferdinand Leifeld

Introduction

During the chain of processing steps for cotton it changes its physical form at each step until it finally fulfils its intended purpose for the consumer (Figure 1).

It is cultivated, harvested, ginned and baled before being opened, cleaned, carded, spun, woven, finished, made into textiles of all kinds by the processing specialists, used by consumers and later, in the form of waste, disposed of, or regenerated/recycled. Then it is returned to a certain stage in the production process for repeated use.

If one follows the above-mentioned processing steps, it becomes clear that at every stage the cotton is refined and extra value is generated (Figure 2). Some of the refinement processes involve cleaning cotton to separate the undesired substances from the desired, pure cotton. Separation is never entirely successful. In the process, waste is separated that contains trash as well as fibres. The fibres represent a resource that can be reclaimed if they are again separated out of the waste. The major part of this paper will deal with this form of fibre recovery.

The cleaned fibres are sent in the form of silver, roving or yarn from machine to machine. These processes also generate waste. This occurs as a result of sliver breakage or when starting and stopping machines and production lines. This material, known as reworkable waste, is fed more or less directly back into the process (see Figure 3). This will be explained with reference to an example.

In the processes following spinning, e.g. weaving and garment-making, further wastes are generated. These can be regenerated like the used textiles mentioned above. This is usually effected with cutting and tearing machines which reduce the fabrics to fibres. Such fibres are used as wadding, added to blends of virgin raw material for spinning, or spun in their pure state for special fabric qualities suitable for this process. This paper concentrates on the subject of waste recycling in the spinning process.

Questions

Whenever there is mention of waste separation and recycling in this field, various questions reflecting the various points of view are asked. First of all there is the opinion that it is no longer worthwhile recycling wastes because modern cleaners today separate waste with low fibre proportions. On the other hand the view is expressed that high rates of cleaning efficiency should be achieved and it would therefore do no harm to separate more waste even if it contains a higher proportion of fibres. Efficient cleaning is not that crucial, it is claimed in justification, because recycling is carried out anyway.

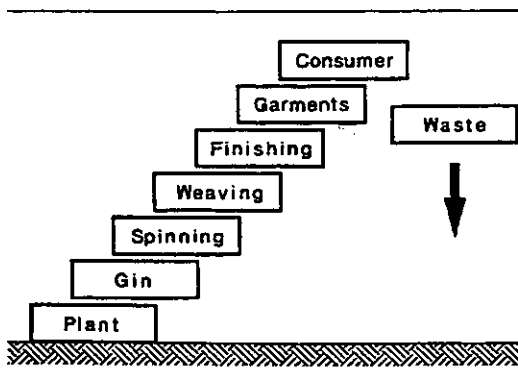


Figure 1: Processing stages

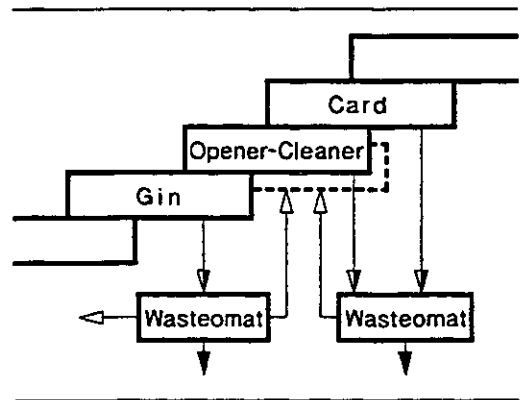


Figure 2: Reclamation of primary waste

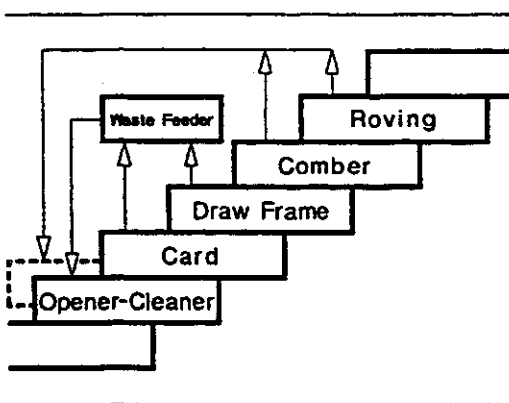


Figure 3: Reclamation of reworkable waste

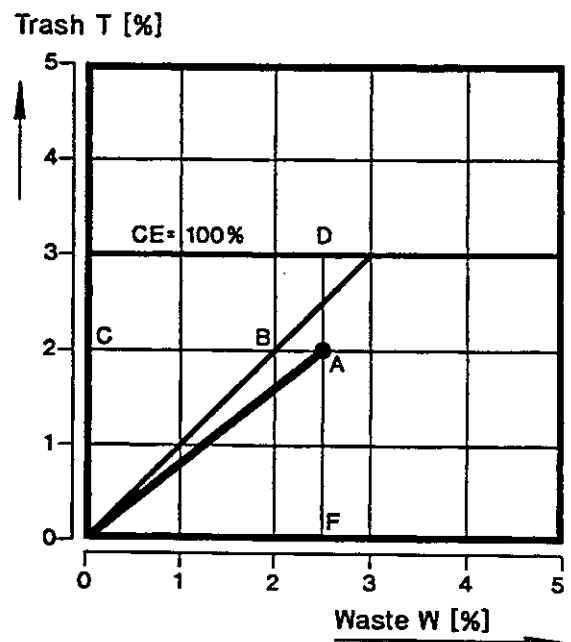


Figure 4: Cleanogramme

Over the course of the last 10 years the trash content of the raw material has been steadily reduced. Today trash contents of 1.2 to 1.5% in the bale are perfectly normal. From this fact alone one could logically conclude that it is no longer worth recycling wastes. But is this really the case?

Figure 2 however, helps to understand that ginning, cleaning and carding all produce wastes which fortunately can be reprocessed with recycling machines. In the spinning practice little attention for example is devoted to gin motes, which according to our analyses, can contain 70 to 80% fibres. These wastes are generally reprocessed by companies specialising in this activity and regenerated for special purposes.

Waste separation

In modern cleaning and carding rooms high cleaning efficiency with low fibre loss has been achieved. The rates of cleaning efficiency are closely related to the separated quantities of waste. It is easy to generate high-quality waste if one is prepared to accept a low rate of cleaning. In our terminology high quality waste refers to a low content of fibres, i.e. 10% and less.

It is not difficult either to achieve a high rate of cleaning efficiency if a high proportion of fibres in the waste is tolerated. The ultimate objective, however, is to achieve a high cleaning efficiency and a low content of fibres in the waste. This is precisely what we expect from good cleaners and cleaning lines and what distinguishes them from less good ones. Because of the divergence in the two main requirements an ideal solution is only possible in the form of an acceptable compromise. This must be technically measurable, reliable and reproducible.

The quality of cleaning can only be judged by figures. For this reason when referring to an achieved rate of cleaning it is essential to maintain the quantity of separated waste and its composition. A full picture is obtained when the following values are quoted:

1. The content of trash in the material being cleaned.
2. The content of trash in the cleaned material, which is the most important figure for yarn manufacturers.
3. The cleaning efficiency [in %], calculated from 1 and 2.
4. The waste content [in % of the treated input quantity].
5. The waste composition, i.e. content of fibres and trash [in%].

Usually these results are graphically presented for ease of comparison and to enable relationships to be identified. To obtain a comprehensive picture, four or five graphic representations have to be generated, which regrettably is not always done because of the excessive effort involved. For this reason only certain data are supplied, and so that necessary for a proper assessment is often missing. The picture remains incomplete, but it

is possible to present these relationships in a single diagram (Figure 4) which is called a "Cleanogramme". On the x-axis is the quantity of separated waste and on the y-axis the quantity of separated pure trash.

With the same scale on both axes, the plotted 45° line represents the ideal separation line. The points on this line refer to a cleaning process in which the separated quantity of waste consists of pure trash. Real points can only be expected on the right-hand side beneath the 45° line. The distance of a real point A from the ideal line says something about the purity of the waste. The closer the point to the ideal line, the lower the fibre content in the separated waste. From the horizontal line C-A, the following can be identified on the x-axis:

- the absolute quantity of the separated trash C-B,
- the separated trash B-A and
- the separated quantity of total waste C-A.

In this "Cleanogramme" the initial trash content of the material being treated can be entered as the horizontal. In this case it is 3%. From the distance between points D and A one can read off the residual trash still present after the cleaning cycle in this case, 1%. In this "Cleanogramme" the cleaning processes can be clearly displayed and give important information. It can also be used for clearly describing, comparing and assessing very good multistage cleaning processes with all the relevant details.

In an example, let us compare the cleaning efficiency of the two processing lines shown in Figure 5. The upper diagram shows a conventional preparation line with three individual cleaners and beneath it a modern line with just a single CVT4 four-roll cleaner and a DK 760 card. The cleaning result of both systems are presented in the "Cleanogramme" in Figure 6.

The axes here no longer have the same scales. The left, diagonal straight line passing through point 1.1 is again the ideal line and in this case it no longer runs at 45°. The horizontal line shows the raw material's initial trash content, in this case 1.4%. This line is also important for the achieved cleaning efficiency. When this line is reached, 100% cleaning efficiency has been achieved. The individual points show the achieved cleaning efficiency of each process. The comparison of the two systems shows the progress achieved in the development of the most recent generation of cleaners and cards, CVT4 and DK760 compared with the former concept consistent of the individual cleaners AXI-FLO, RN and RSK respectively. The cleaning performance of the lick-in is assessed separately here.

One can see that the coarser cleaning steps at the beginning come closer to the ideal line than the fine ones. A better cleaning performance with reduced fibre waste is achieved with the CVT4. It has always been known that the final cleaning points and particularly the card cylinder with its separating equipment separates waste with a higher fibre content than the preceding separation points. As can be seen, the characteristic curve for the cleaning efficiency flattens and the distance from the ideal line grows.

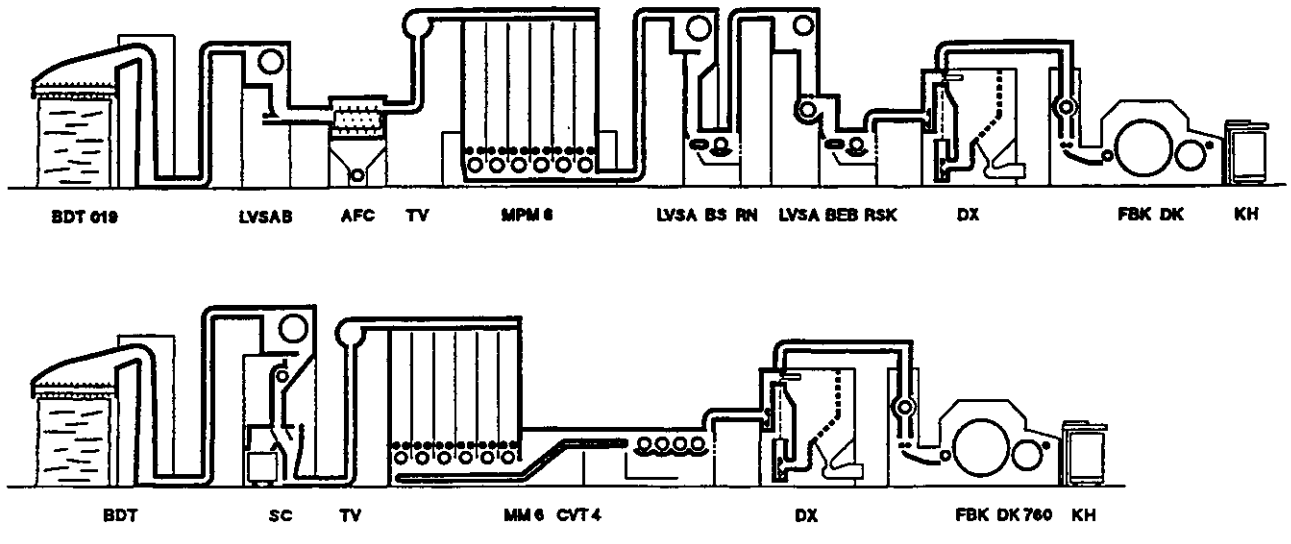


Figure 5: Conventional and modern cleaning line

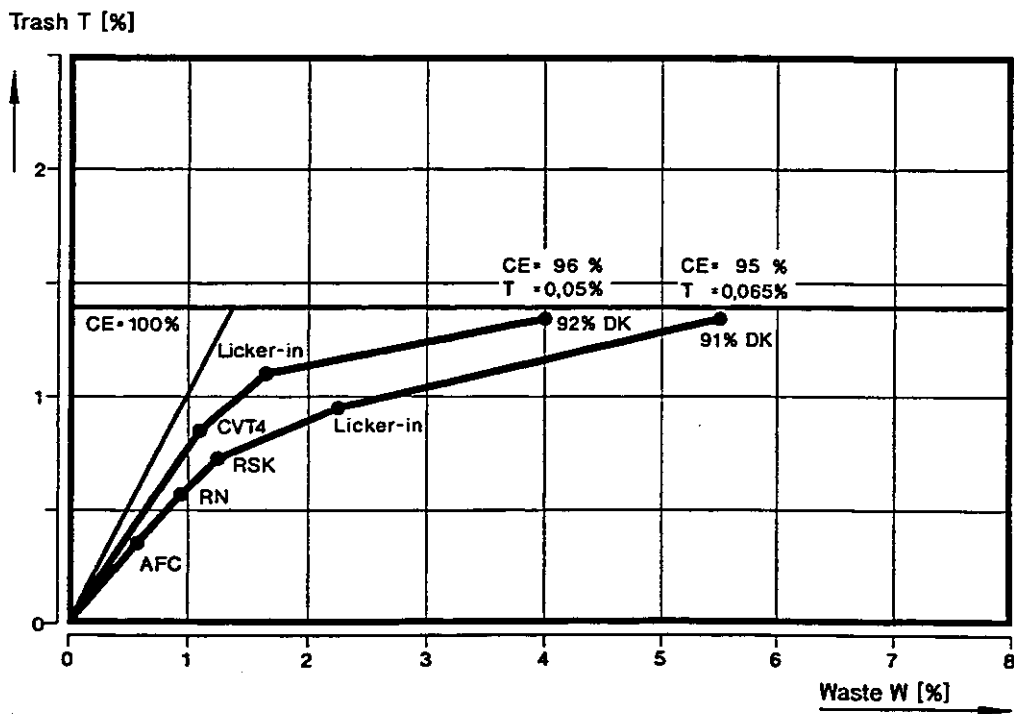


Figure 6: Comparison of the cleaning efficiencies between conventional and modern cleaning lines

Therefore it is evident that the new cleaning line separates a total of 4% waste which contains almost 1.4% trash. This means that the total waste consists of 35% trash and 65% fibres. The former cleaning line extracted 1.4% trash in a total waste quantity of 5.5% and this means that with the former system, the waste contained 25% trash and 75% fibres.

The effort to lower the fibre content in the separated waste with more advanced machines has thus been successful, and in the above example with the new machinery up to 1.5% less fibres are separated in the waste. This means 1.5% less input of raw material, or in other words 1.5% savings of raw material with the new line in comparison with the conventional technology. This was one of the reasons for the rapid success of the new generation of cleaners because they very soon paid for themselves solely as a result of the lower input of raw materials.

However, it is generally accepted practice for primary waste to be collected and processed together even though the proportion of 75% fibres in the total waste has been reduced to 65%. This being so, then the question of the nature and proportion of these waste fibres should be addressed. The wastes from the cleaners generally have a fibre content of only 20 to 30%. Even these quantities are still worth recycling.

In the treatment of this subject so far the influence of the material on the waste quantities has been ignored. A certain degree of fluctuation has to be expected as a result of the material's cleansibility. The cleaners have cleaning points with adjustable guide vanes and can be adapted to the properties of the particular material. As a result the waste quantities can be regulated at each separation point while the machine is running.

A major factor is the trash content in the raw material. For the modern cleaning line there is a rule of thumb that the proportion of waste separated in the cleaner should be equal to the proportion of trash in the material. On this basis, the CVT4 has to separate approximately 1.4% waste from a raw material containing 1.4% trash. If a cleaning efficiency of 70% is to be achieved in the process, the fibre content in the waste would then be approximately 30%. One could now start calculating to find out how much waste can be saved at this point and how much is to be recycled. However, the CVT4/DK 760 system is an integrated system. In certain respects the action of the CGT4 cleaner only takes full effect in the card. When employing the CVT4 it was discovered at the card that the licker-in waste and the upper card waste were drastically reduced and the cleaning efficiency was improved over that of the combination conventional cleaners and card. This can be clearly seen in the "Cleanogramme" in Figure 6.

A modern cleaning point is situated in the card's licker-in and makes it possible to influence cleaning efficiency and waste composition by regulating the rate of separation. The "Cleanogramme" in Figure 7 shows this relationship and the results of a licker-in study.

We recommend adjustment to the first mote knife in such a way that a cleaning efficiency of about 30% is achieved. With an input of 1% trash in the material this means a pure trash separation of 0.3% and this is achieved with a waste quantity of 0.4%. One can then see that about $\frac{3}{4}$ of the waste consists of pure trash and $\frac{1}{4}$ of fibres, i.e. that the waste has a fibre content of about 25%. If the waste quantity were to be raised above this, the cleaning

efficiency would improve but the fibre content in the waste would also increase disproportionately. If the rate of separation is reduced, the proportion of fibres in the waste declines but the rate of cleaning efficiency drops dramatically. The most favourable, real separation point characterises the ideal setting. In the "Cleanogramme" this is the point where the initial straight line ends and the curve starts to move away from the ideal line.

The relationship between cleaning efficiency and fibre content shows another trend derived from the licker-in "Cleanogramme" (Figure 8). It is clearly apparent that it cannot be the objective to aim for only 10% fibres in the waste at the licker-in, for instance, because the cleaning efficiency inevitably falls to a value of about 10% for this to occur.

If on the other hand the rule of thumb for the cleaning line is applied and as much waste is separated as is trash content in the material - in this case 1% - a cleaning efficiency of 50% would then be achieved with 50% fibres in the waste. This according to his own priorities and within certain limits, the user can exert influence on trash separation, fibre separation and cleaning efficiency.

These diagrams have been dealt with in detail because the aspects covered strongly influence the rate of material utilisation. Reducing fibre separation with efficient cleaning represents the biggest contribution towards cutting the cost of materials since, as already mentioned, the expensive primary raw material is at stake and because this, without any additional treatment such as recycling, yields direct savings.

The licker-in also has a pre-cleaning function. The card cylinder, revolving flats and webcleaner in working together attain an extraordinary cleaning efficiency. If we proceed from the trash content still present in the area between licker-in and cylinder (i.e. 0.1%), we can determine that stripping roller and webcleaner separate about seven to ten times additional waste (i.e. 0.7 to 1%) of the reference trash content. That means that the separated waste contains about 85 to 90% fibres. It should be borne in mind that this waste, in particular, contains a considerable proportion of short fibres and neps, however.

Waste reclamation

The above estimations of fibre content of 65 to 70%, depending on the machine configuration, may be compared with values obtained in the last few years from a large number of customer tests of cotton recycling. The frequency distribution for fibre percentages in the waste in Figure 9 clearly shows that even in industrial practice the greatest frequency lies within the 60-80% range of fibre content in the waste.

Trützschler installations are used by its customers to process cleaning and carding wastes as well as gin motes. In the past, two basic installations for recycling have evolved which differ principally in their production rate. Firstly there is the small installation which is mostly found in spinning mills with a production rate of up to 100 kg/h depending on the quality of the material processed of which open-end (OE) spinners are the main users. Larger installations have a production rate of up to 700 kg/h and are characterised by a central precleaning section and individual cleaners arranged in parallel or in series according to the required production rate.

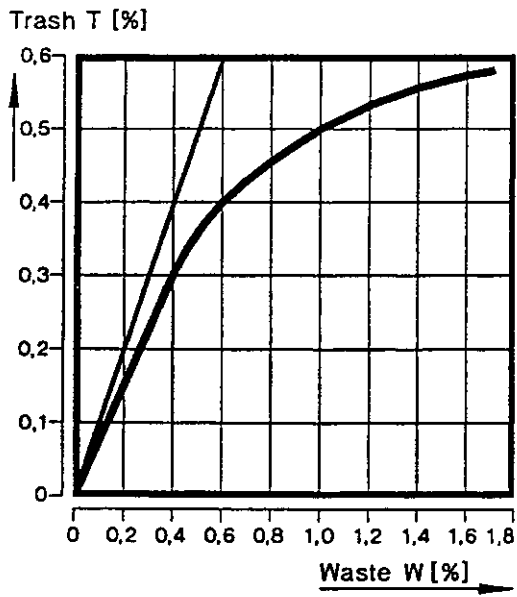


Figure 7: Cleanogramme

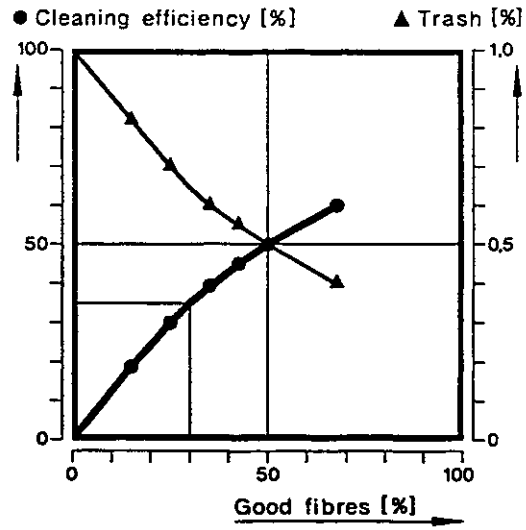


Figure 8: Correlation between cleaning efficiency and good fibre in waste

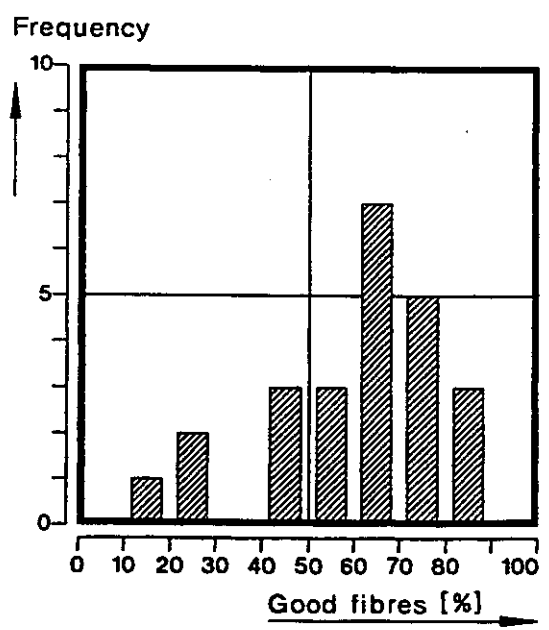


Figure 9: Frequency of good fibre in waste

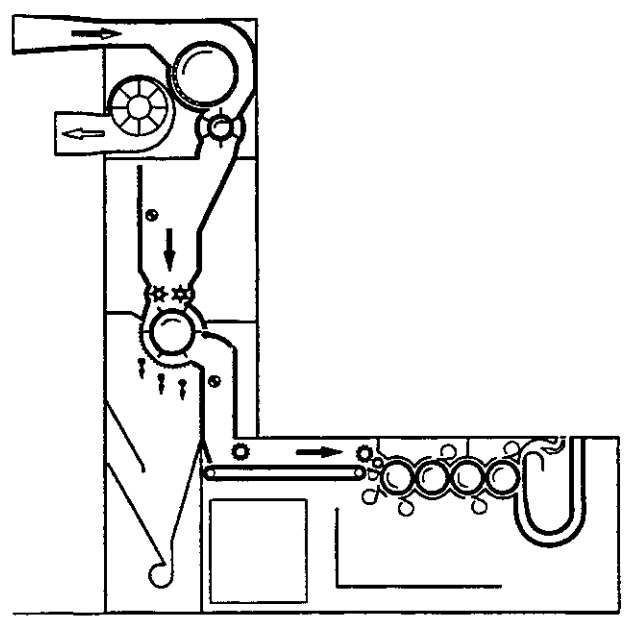


Figure 10: Wasteomat

The central element of these installations are the Wasteomat machines. These are a new development and supersede the successful Cotonia. The Wasteomats (Figure 10) are derived from the successful Cleanomats and are available in two versions, i.e. in a two-roll version for low production rates and in a four-roll version for high production rates. The Wasteomats differ from the Cleanomats in their feeding and choice of clothing. The Wasteomat feeding system has a tray intake, which is superior to the roll intake of the Cleanomats in its opening action and cleaning efficiency. In the Wasteomats all the rolls are saw-tooth rolls, even at the first position, unlike the Cleanomats which always have a fully spiked roll or a spiked lattice roll at the first position. This is to ensure gentler opening. Like the earlier Cotonia, the clothing of the rolls becomes progressively finer as the material proceeds towards outlet and they are generally finer and have a more powerful action than the Cleanomat clothing.

The effective, continuous direct waste extraction has been adopted from the Cleanomat and represents an improvement in comparison with the Cotonia system. Furthermore, all modern monitoring and control devices have been taken from the Cleanomat. The small installation (Figure 11) can be assembled as follows:

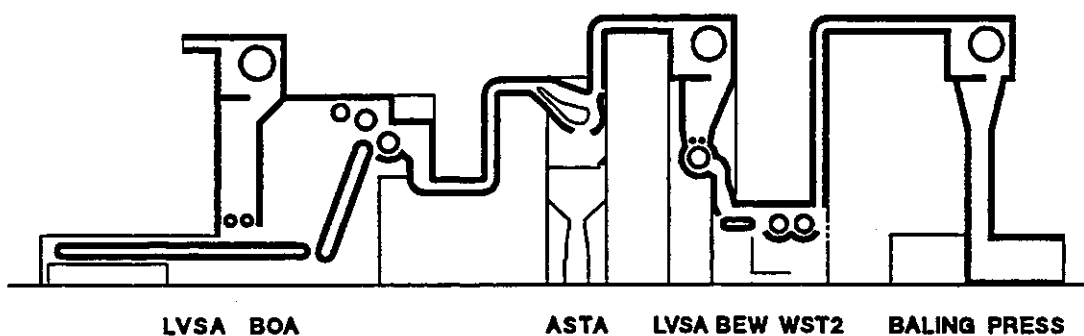


Figure 11: Wasteomat - small installation

Storage and delivery point for the wastes, e.g. BOA, precleaner and BEW feed device, consists of condenser, top feed trunk, intermittent grid cleaning, bottom feed trunk, and a Wasteomat 2 (WST2) with two cleaning rolls and condensers for collection of the cleaned fibre material. In the BEW stage a multiple cycle via the grids is effected by opening and closing a flap at the outlet. Ideally the waste output should go to a baling press. Returning the material in bales to the spinning process thus becomes flexible and a favourable blend is ensured.

The large installation (Figure 12) also starts with a storage facility as the delivery point for the line. This is followed by a series of two AXI-FLO cleaners. These replace the Wilomat. The double passage through the AXI-FLO yields the same cleaning effect as with the Wilomat. It is worth mentioning at this point that grids are the ideal cleaning elements for material containing large quantities of heavy impurities which can easily fall out. Use of these is made both in the AXI-FLO and the BEW stages in the installations. When the bulk of the heavy waste has been separated, the clothed rolls with mote knives and carding elements are the most effective cleaning instruments.

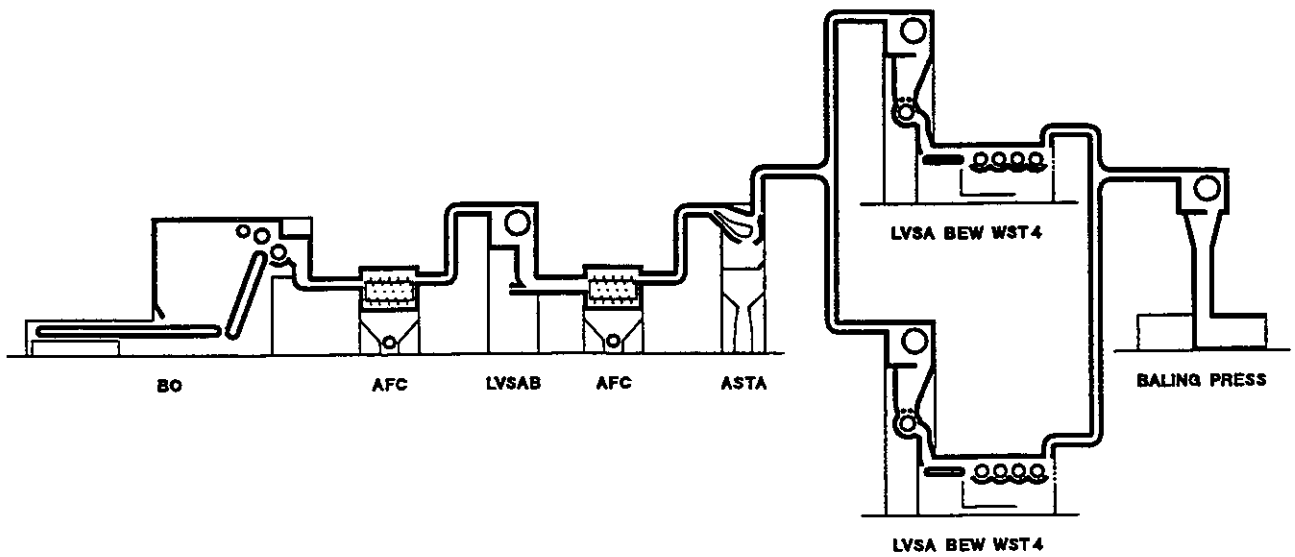


Figure 12: Wasteomat - Large installation

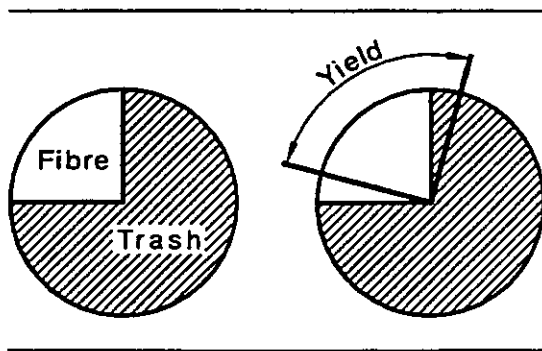


Figure 13: Definition of waste separation

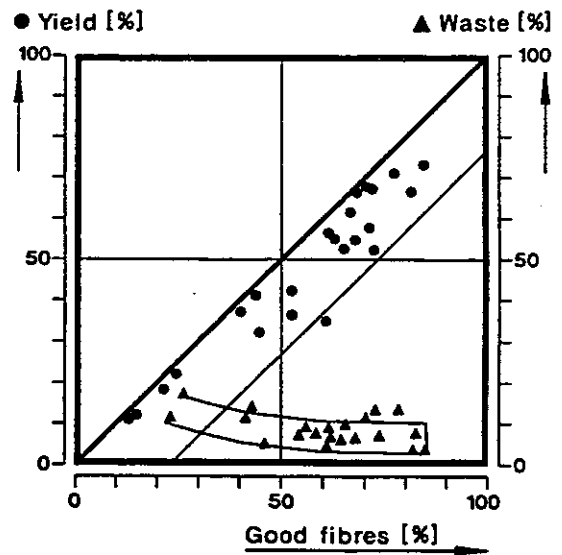


Figure 14: Yield

Use of these is also made in the Wasteomat WST4 which, because of the higher production rate, has four rolls. Owing to the progressive increase in the speed of each successive roll, the last roll can generate an extremely powerful centrifugal force. This yields extremely high separation forces and powerful separation action. This machine is also served by a condenser for waste extraction which collects the regenerated material and separates it from the air current. The process of separating trash from fibres is effective but not 100% as explained in Figure 13. The primary waste containing trash and fibres is divided into two categories, the first containing a high fibre and low trash content, and the secondary waste with reverse proportions.

For the sake of completeness the losses due to the extraction devices which trap dust and short fibres in the filters require mention. These are disregarded in Figure 13 but can account for between 1 and 10% of the material weight.

Actual results are shown in Figure 14 where the diagonal again shows the ideal separation line. The real points give an indication of how good the actual yield is and a high yield reflects a high fibre content in the material. The bottom region of Figure 14 presents the achieved residual trash content after cleaning and this area varies essentially between 2 and 10% residual trash in the yield. The scatter of data points in the respective areas is largely attributable to the properties of the material being treated. The results of cleaning and yield are approximately the same for the small and large installations. The small installation is usually used in the spinning mill and the large installation generally by specialist waste processors.

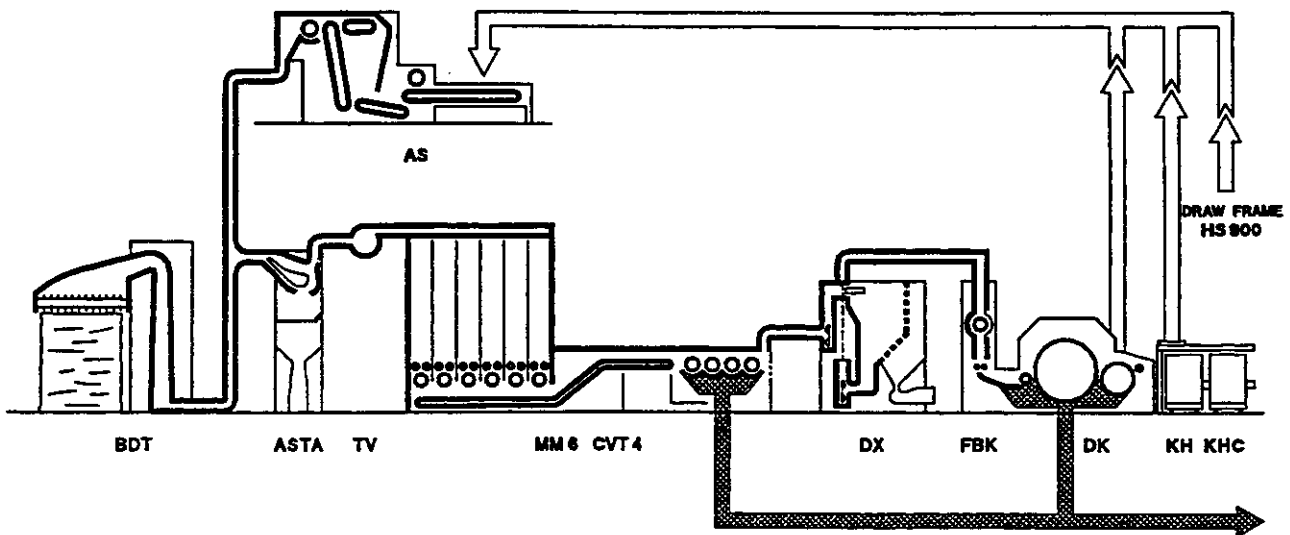


Figure 15: Distribution of waste

The waste flow in spinning preparation is shown in Figure 15 in which the upper part shows the direct feedback of reworkable waste such as card or draw-frame silver via the waste feeder. The feedback into the process should take place before the heavy trash separation and before metal separation. It is at this point that valuable reworkable waste is returned almost without loss to the process. The primary waste is indicated beneath the processing line. The feedback of this waste into a certain processing stage varies from company to company according to its final utilisation as shown in Figure 16.

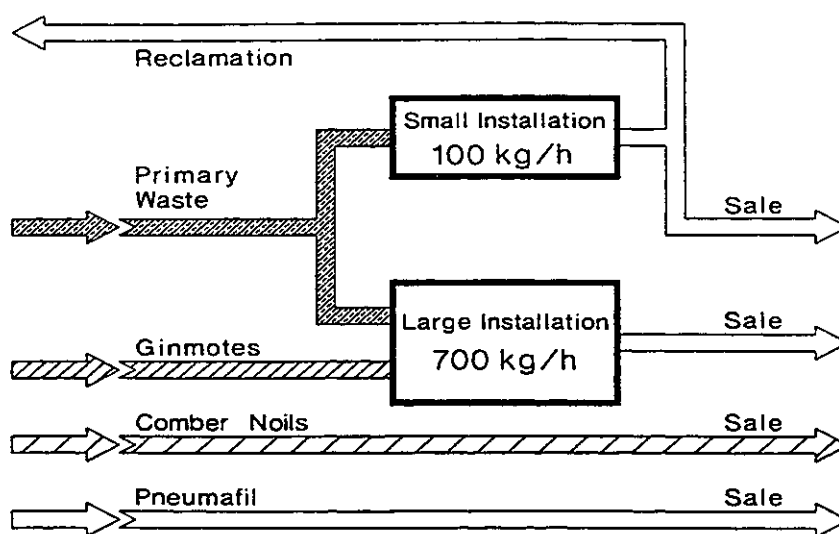


Figure 16: Utilisation of waste

Two main alternatives exist, the first of which is where the wastes are reprocessed directly in the spinning mill and are then used for minor qualities or sold as purified waste. Alternatively the wastes are collected and sold unprocessed. These wastes usually end up at reprocessing companies. These mostly operate large reprocessing lines where wastes from all kind of sources are processed. These wastes include Pneumafil, comber noils and gin notes.

Economic aspects

In order to decide whether it is worthwhile recycling wastes from a modern processing line with reduced fibre separation, a rough calculation of costs may be undertaken. Consider again the example above in the "Cleanogramme" for the CVT4 line with card where the trash content in the bales is low at 1.4% (see Figure 6). The total quantity of waste from the cleaner and card is 4% comprising about 1.4% trash and thus 2.6% fibres. The fibre content in the waste is therefore 65%. Figure 17 shows this and the following calculations.

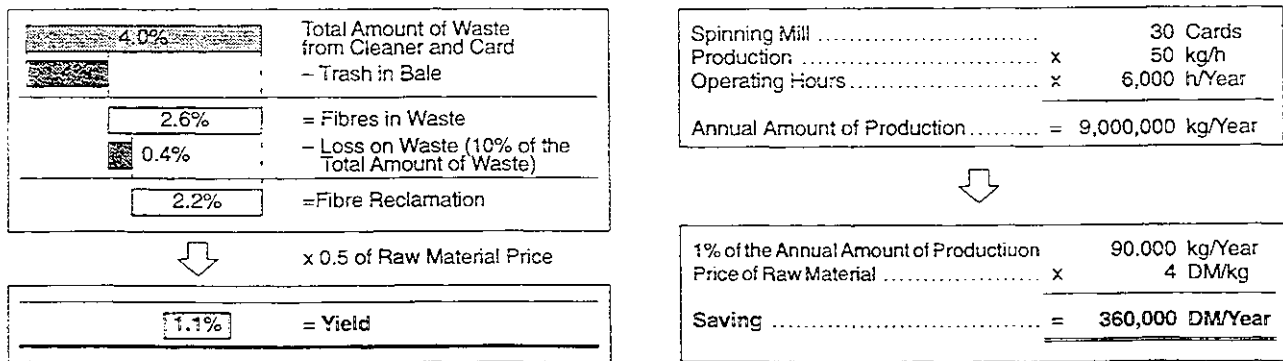


Figure 17: Economic calculations

In attempting to recover the 2.6% fibres from the waste, losses may be expected which in the most unfavourable case can be as much as 10% of the original secondary raw material. This means 10% of 4% which is a 0.4% loss to give a fibre recovery of $2.6 - 0.4 = 2.2\%$. The secondary raw material is not as valuable as the primary raw material. If it is assumed to be half as valuable, this would yield a raw material recovery potential of 1.1% relative to the primary raw materials which for simplicity may be assumed to be 1%.

If this result is applied to a spinning mill which operates 30 cards each with an output of 50kg/h and 6,000 hours of production per year, then the annual production rate is $30 \times 50 \times 6,000 = 9,000,000\text{kg}$. 1% of this is 90,000 kg. This is a considerable value-added volume. With a material price of 4 DM/kg, this is equal to DM 360,000 per year (£150,000 per year).

Following from this, with 6,000 hours of production per year, the hourly production rate of the recycling installation is $360,000 : 6,000 = 60 \text{ kg/h}$. The small installation, therefore, with an actual hourly production rate of 100 kg/h is suitable for the spinning mill in this example.

The questions posed in the introduction about the value of recycling and, particularly in relation to the employment of modern cleaners, cannot be answered with a good deal of certainty and as is often the case, the truth lies somewhere in the middle. Within given technological restrictions, it is thus worthwhile reducing the rate of fibre separation at all separation points. It is also worthwhile recovering the content of fibres left in the waste with recycling machines.

In conclusion, therefore, recycling of waste fibres is not only environmentally desirable but also economically attractive.

RECYCLING IN THE FAR EAST - A STUDY OF COTTON WASTE

Nasim A Minhas

Introduction

The subject of recycling is a vast one and its importance is gaining ground every day. This can be attributed to several main reasons:

- The world's population, which is increasing according to a geometrical progression and is producing an excessive amount of waste of all kinds.
- The problems of environmental pollution which can arise in the disposal of such waste.
- The growing shortage of raw materials and the consequent increase in the significance of so-called secondary raw materials (ie. recycled wastes).
- A widespread concern (especially amongst the very young and the very old) for proper conservation of the Earth's resources and for a frugal approach to consumption.
- Economic constraints which necessitate making savings in every process and every business.

Recycling and conservation is now a factor in every field of our lives. Such heightened awareness, along with the changing economic factors, is a dominant force in bringing about change in industry and commerce.

The concept and practice of waste processing in textiles is very old, and has been in existence in most European countries (especially Italy, Britain and Belgium) for more than a century, but it was always done "behind the scenes". Products made from recycled material were considered to be inferior and the word 'shoddy' (open waste) was used as a term of abuse and to denote poor quality. It was almost taboo in developed societies to use cloth made from recycled materials. But since the late 1960's attitudes have changed and people are now proud to use recycled material, and indeed they are becoming quite fashionable. For example, the firm Esprit established in Britain a few years ago is making garments from 100% reprocessed waste and achieving premium prices for them.

In Europe and North America the economics of collection and sorting of various wastes has been influenced by very high wage structures and by the huge reduction in size of the domestic textile industries (in many cases to as little as 20% of their original size). However, these factors do not apply in Asia and the potential for profitable textile recycling is high.

The Start of a Business

The concept of recycling has always been fascinating. During my early career, in the decade from 1953, I worked in woollen mills in Pakistan where waste material was being imported from Italy and Britain for use in blends for blankets and other fabrics. A great variety of different fabrics came to us in this way, for example pastel stockings for blazers, open serge cuttings from overcoatings, and so on. In those days the use of man-made fibres such as polyester and acrylics was not very common, even in Europe. So the blends were mostly of wool or some mixture of wool with viscose or nylon. The Pakistan industry was spending a lot of foreign exchange on imports of such material from abroad.

One Sunday in 1964, while driving through Anarkili, one of the main shopping areas in central Lahore where many tailors were working, outside each of their little shops I saw heaps of fabric cuttings - off cuts - lying on the pavement awaiting the refuse collectors. As I saw the sweeper loading them on to a cart en route to the central dump, I told my children that if all this waste were collected, sorted and opened we could earn good money. They laughed and told me that it would be degrading to collect such waste, and what would everyone say when they heard of such a venture! But in my mind a ball had started rolling, and it became an obsession with me to start such a business. So, a few months later in 1965, I gathered enough strength to put my idea into a practical project: Farooq Waste Trading Company was launched! I engaged a few workers which would collect these cuttings from the shops and bring them to my warehouse for sorting and opening. I imported a single cylinder opener (rag machine) from Japan and from then on I never looked back!

Initially cuttings from tailors were the main source, but after a few years damaged second-hand clothing, especially woollen knitwear, was added to my inventory. The sweaters and pullovers were opened by hand to recover yarn, which was later used in making rugs, carpets and blankets directly. The other secondhand clothes such as coats and skirts were put through the rag opener and subsequently mixed with virgin fibres for making blankets and various other fabrics. Materials which were previously thrown away become a source of income for me and for hundreds more. Tailor cuttings from military ordinance depots and railway garment factories were added to our inventory. It was heartening to see how many people found jobs in this new vocation and new industry, especially women, who at that time had few employment opportunities. Some of my work people even set up in business on their own account in later years. Precious foreign exchange was conserved by using local waste instead of imported waste.

So far I have talked about my own business and naturally about the conditions that pertain in Pakistan. Having being asked to talk in a wider Asian context, however, I have taken some time to research the prospect in the other SARAC nations: that is Indian, Pakistan, Nepal, Maldives, Bhutan, and Sri Lanka. In each of these countries such ventures can be started on a national level under what I may call the CRCS principle, being short for 'Collect, Recycle, Conserve, and Save'.

Cotton Waste Recycling - A Case Study

It is always envisaged that cotton will become scarcer and dearer due to population growth and occasional crop failures because of natural calamities; indeed for the last three years, the latter has happened. In Asian cotton-growing areas, particularly China, India and Pakistan, the crop was badly affected by virus attack. In the following year, because growers did not realise good returns on their cotton crop, cotton was planted in reduced land areas which resulted in acute shortage of cotton supplies. Taking Pakistan's example, the 1994 cotton crop was reduced to only seven million bales (in 1992 the size of the crop was 12.00 million bales). Cotton prices in 1993 averaged at Rs. 1600 per 40 kgs whereas in 1994 the prices jumped to Rs 2400 per 40 kgs. Similar conditions prevailed in India and China. Thus the available cotton supplies to manufacturing industries were drastically reduced. The actual shortfall was in fact 1.5 million bales. In this scenario recycling of cotton wastes has become very important.

Cotton Waste types: First of all, it would be useful to give details of various wastes available in cotton ginning and spinning units and these are as follows:-

- (i) Fibre waste from Ginning Plants;
- (ii) Blow-room gutter waste;
- (iii) Blow-room droppings;
- (iv) Card dropping/lickerin waste;
- (v) Card fly;
- (vi) A.C. gutter waste;
- (vii) A.C. filter waste;
- (viii) Ring sweepings;
- (ix) Lap and web waste from pickers/cards;
- (x) Roving waste;
- (xi) Cotton comber waste/noils;
- (xii) pneumafil/suction waste; and
- (xiii) Yarn waste No.1 quality/oily yarn waste.

Processing Machinery: Many machinery manufacturers from Europe notably Trutzscler and Laroche use modified versions of the step cleaner as the first machine which is comprised of a filling chute with feed table and condenser on top. The feed rate is preselected on a timing relay. The stepcleaner has six beaters with strong strikers which open the waste by interaction with the grids. Depending on condition of the material the cleaning time is adjusted from 3 - 60 seconds as the condenser draws the material from the top beater and after dedusting throws it into the filling chute back into the step cleaner. After cleaning, the cleaned material is delivered to a bale press or chamber from which it is taken to a second cleaning operation.

Temafa use the "clean star" system as their first cleaning machine but due to low production rates this system, although very effective, has not been very popular.

Chinese and other Asian manufacturers use intermittent "Willey" or "Willow" machines as the first cleaning stage. These machines are manufactured and used in Pakistan at a fraction

of the cost of the step cleaner described above.

The second cleaning machine consists of a cylinder with sawtooth metallic wire in fixed bearings and a spring loaded fluted cylinder in movable bearings. A fixed tray feeding system feeds the material to a sawtooth drum which extracts the impurities from waste in conjunction with a moving knife and grid. This machine comes in single and double cylinder versions depending on requirements. Cleaned material from this machine is ready for use in subsequent processes.

In China, India and Pakistan local versions of this machine are made with single, double or three cylinders at very low price.

The third machine in this series is the roving waste opener which is used for opening rovings. The cylinder of this machine has 36 wooden lags with round-pointed pins for opening action. As the lags are made of laminated wool these can be repinned many times.

The fourth machine is for opening hardwastes and fabric wastes and is called a combined tearing machine depending on the nature of the material; 2 to 6 cylinders with corresponding pinning are used.

Machinery costs: The price factor of such machines is most important, especially when these machines are to be installed in developing Asian Countries. For example, in Pakistan large textile mills may be able to afford machines at European quoted prices but for individual entrepreneurs this is a prohibitive factor. Roughly the prices are as follows:-

- (i) A one step cleaner type opener with one cylinder waste opener along with filtration system would cost around £150,000. This is out of range of most of the waste dealers/processors. Hence effective cheap local machines have been developed, which although may not give as good results as the original machines, but still process material to 70-80% of the quality at a fraction of the machinery cost. For instance, instead of using a step cleaner type machine a Willey machine may be used for precleaning.
- (ii) The first step may be followed by local small width 2-3 cylinders opening machines. These will cost delivered from works to a local buyer:-

Willey type opener	£5000
3 Cylinder Novacotonia-type waste opener	£15000

This represents a total investment of £20,000 against £150,000 for a new European manufactured plant.

The Willey will process 100/150 kgs of waste per hour. For the imported single cylinder opener, 75/100 kgs of waste is processed per hour. Maintenance costs of imported machines are very high as metallic wire needs replacement frequently, whereas on local openers small width wooden cylinders are used with locally made metallic wire which is very cheap.

Similarly, a two cylinder tearing machine (eg. Laroche) would cost around £150,000. There is no good substitute for this machine being made locally. These machines again use wooden cylinder type openers with metallic wire and process the material over and over again (6-7 passages) depending on the condition of hard waste and cuttings being fed in.

This results in excessive fibre damage to the material being processed. Experience shows that cotton waste recovered on local Willey and wasteopeners is good enough for subsequent processing as recommended in Table 1 giving 80% of the quality when compared with imported machines. But local tearing machines do not give the required good results. However, recycled wastes are exported in high quantities abroad for various industrial uses.

As illustrated by the above case study, waste processing offers great opportunities for textile processors in Asia often using locally made machinery. The recycled fibres may be used wholly or as blends with virgin fibre to produce a range of products. This paper has attempted to introduce potentially new entrants to elements of the practical side of the waste recycling industry. Currently the industry relies on practical experience and not scientific principles.

Table 1: Waste types, processing routes and possible end-uses

WASTE CATEGORY	Principle machine use for fibre recovery					Combined Tearing	Fibre yield, %	Possible End-Uses				
	None	Willey	Step cleaner type open	Novacotonia	Roving opener			Ring yarns	Rotor yarns	Wadding	Upholstery yarns	Non-woven
Fibre waste from Ginning:												
Ginning Plant	-	+	+	+	-	-	25-50	-	+	+	+	+
Blow room gutter	-	+	+	+	-	-	10-15	-	-	+	+	+
Blowroom dropping	-	+	+	+	-	-	30-40	-	+	+	+	+
Card dropping/lickerin	-	+	+	+	-	-	20-40	-	+	+	+	+
Card fly	-	-	+	+	-	-	50-80	(+)	+	+	+	+
A C Gutter	-	+	+	-	-	-	40-60	-	+	+	+	+
A C Filter	-	+	+	-	-	-	50-80	+	+	+	+	+
Ring sweepings	First	manual	picking then	Willey and	stepcleaner		50-80	-	+	+	+	+
Lap & Web wastes from pickers/cards	+	-	-	-	-	-	100	+	+	+	+	+
Roving Waste	-	-	-	-	+	-	98	+	+	+	+	+
Cotton comber waste/noil	+	-	-	-	-	-	95-99	+	+	+	+	+
Pneumafil/suction waste	+	-	Maybe	-	-	-	98-100	+	+	+	+	+
Yarn waste and oily yarn waste	-	-	-	-	-	+		-	+	(+)	+	+
Cloth cuttings	-	-	-	-	-	+		-	+	(+)	+	+

Acknowledgments with thanks to Trutzschler.

THE PRODUCTION OF HIGH TENACITY TAPES FROM WASTE POLYPROPYLENE

Subhas Ghosh & A Richard Horrocks

Introduction

The more common uses of recycled polymer waste are for products in which tensile, mechanical and physical properties are not critical and so the presence of degradation centres, damage to polymer chains and impurities are of little consequence. Thus many recycled polyolefins, for example, find use in garden products such as plant containers and timber substitutes. Only where the quality and purity of waste can be assured are secondary products capable of maintaining high levels of mechanical, physical and chemical performance. Of special significance to the textile industry is the re-use of poly (ethylene terephthalate) pressurised drink containers as raw materials for melt extrusion into fibres. However, because of the presence of inherent colour within the original bottles, fibres produced may also be coloured and so of less than premium quality.

The area of technical textiles, where aesthetics are of less consequence than in other textile sectors, offers the opportunity to create fibres and tapes having first grade properties if the physics and chemistry of recycled polymer and its conversion are understood. Of particular interest is the field of geotextiles (textiles used in civil engineering) where the annual world market for geotextiles is approaching 1000 million square meters equivalent to about 250,000 tonnes of raw materials of which over 70% comprises polypropylene. During the extrusion of component fibres and tapes, up to 10% by weight of process waste polymer is introduced without noticeable effects on quality. There is a desire by producers⁽¹⁾ to increase the amount of blended recycled polypropylene and even include polyethylene with virgin polymer during the extrusion process. In this way, they can fully re-use their own wastes, supplement them by including wastes from other sources and so replace virgin polypropylene by cheaper, recycled polymer. Sources of non-processor waste may include polypropylene granules from recycled automobile battery housing⁽²⁾ and industrial film waste, for example.

To date no academic study has been undertaken and reported which investigates the effects that added polyolefin waste has on the tensile, physical and chemical properties of orientated polypropylene tapes although La Mantia and co-workers^(3,4) have undertaken research into the consequences of adding heterogeneous waste to polyethylene. This paper reports such a study and analyses the effects that adding large proportions of waste has on the properties of orientated polypropylene tapes. The full details have been published elsewhere^(5,6).

Experimental

Materials: Isplen PP040 manufactured by Repsol, Spain was used as virgin polypropylene. The virgin Isplen PP040 had a nominal melt flow index (MFI) of 3.0 (g/10 min at 230°C,

2.16kg). The polypropylene pellets contained 0.10% of a hindered phenol antioxidant, Irganox 1010, as a heat stabilizer. To produce simulated process waste, Isplen PP040 was re-extruded and pelletized up to ten times. At the end of two, five and ten cycles a mass of the recycled polypropylene was collected for recycling with virgin polymer. Industrial polypropylene film wastes having MFI of 8.6 (g/10 min at 230°C, 2.16 kg) was obtained from Cabot Plastics Limited, UK. This waste contained compacted films from various sources and was granulated prior to extrusion. In the following discussions waste designations A, B, C and D will be given to industrial film and 2x, 5x and 10x re-extruded PP040 polypropylene wastes, respectively.

Stabilizing System: In order to reduce the effects of degradation products present in recycled polymers and thus enhance the durabilities of tapes, an additional additive package comprising process (heat) and U.V. light stabilizers was used in this introduction into each polymer. This masterbatch contained Ciba-Geigy Type B561 stabilizer which comprises Irgafos 168 and Irganox 1010 in the weight proportion of 4:1. Irgafos 168 is a phosphite type processing stabilizer which in combination with the phenolic antioxidant Irganox 1010 provides both processing and long term thermal stability⁽⁷⁾. In addition, Tinuvin 770 DF, a hindered amine light stabilizer (HALS), was also used in the masterbatch in equal proportion by weight with the heat stabilizer.

Tape Production: All tapes were produced from waste-virgin polymer blends on a Mk-1 Lanline Laboratory extruder from Plasticisers Engineering, Ltd., England, as discussed fully elsewhere^(5,6) to give tensile properties similar to those for normal commercial tapes.

Melt Flow Index (MFI), Tape Tensile Property and Physical Characterisation: MFI of the polymer pellets and oriented tapes were determined on a Martin Melt Flow Indexer at 230°C ± 1°C under a load of 2.1kg using BS2782: Part 7: Method 720A: 1979. Five measurements were made on each sample (see Table 1).

Tensile properties were determined by tensile tensiometry and indications of tape crystallinities were measured as intensity ratios of the 974 and 995 cm⁻¹ bands as described fully elsewhere^(5,6).

Results and Discussion

Effect of Recycling History on Polymer Melt Flow Index: Table 1 lists the MFI values of virgin and waste polymers before and after extrusion, with and without additional stabiliser. It is clear that values increase with a number of recycling and that the presence of stabiliser reduces the magnitude of MFI increase during extrusion. Furthermore, the MFI value of the 10x re-extruded Isplen PP040 is close to that of the industrial film waste and may be taken to be an acceptable model waste.

TABLE I. MELT FLOW INDICES OF POLYMER PELLETS AND EXTRUDATES

	MFI, g/10 min, 130°C, 2.16kg		
	Polymer Pellets	Extruded Tapes	
		Without Stabiliser	With Stabiliser
Virgin Isplen PP040	3.24	4.18	4.19
Isplen After 2x Recycled (B)	3.55	4.95	4.68
Isplen After 5x Recycled (C)	5.01	8.45	6.64
Isplen After 10x Recycled (D)	7.05	10.53	8.20
Industrial Film Waste (A)	8.64	11.87	10.39

Effect of Waste Inclusion on Tensile Properties: A general trend of decreasing tape tenacity consequential upon the addition of recycled wastes both in absence and presence of stabilizer has been observed. The relationships between tape tenacity and recycled waste concentration for all four types of recycled materials A,B, C and D are illustrated in Figure 1 respectively.

In all cases, tape strength decreases with increasing recycled material concentration, however, strength losses were greater after the initial 10% additions than after subsequent incremental additions of waste. Reductions in tenacity are relatively small for additions above 50%. A variance analysis of tenacity data indicated that the tape strength loss increased in the waste order B (two passes) < C (five passes) < A (film waste ≤ D (ten passes) i.e. tenacity retention are in the order B > C > A > D as illustrated in Figure 1. These general decreases in strength with increasing waste concentrations and hence increased thermal history of recycled polymer can be attributed to the thermo-oxidative degradation of the recycled polymers demonstrated by their increased MFI values shown in Table I.

The presence of additional stabilizers has significantly improved tape strength retention as seen in Figure 1. The effect of stabilizers in reducing tenacity loss was greatest for both the industrial film waste and ten times recycled Isplen PP040 waste D which is probably a consequence of these two waste materials containing higher levels of degradation products. These would tend to promote higher levels of subsequent oxidative degradation during polymer blend extrusion. The retarding effects of the antioxidants on the degradation of the polymers are evident in lower MFI values (Table I) of recycled materials presence of additional stabilizers relative to their unstabilized analogues.

Breaking strain values of all tape samples were highly variable and did not show a significant relationship with waste concentration, unlike tenacity values. At higher waste concentrations and particularly for waste C, breaking strains of tape samples generally decreased. The improved retention in tenacity for each stabilized waste-containing tape series relative to its unstabilized analogue noted in Figure 1 were matched by similar improvements in breaking strain retention. This is to be expected as a consequence of improved melt stability and hence extrusion uniformity which the presence of melt stabilisation additives confers.

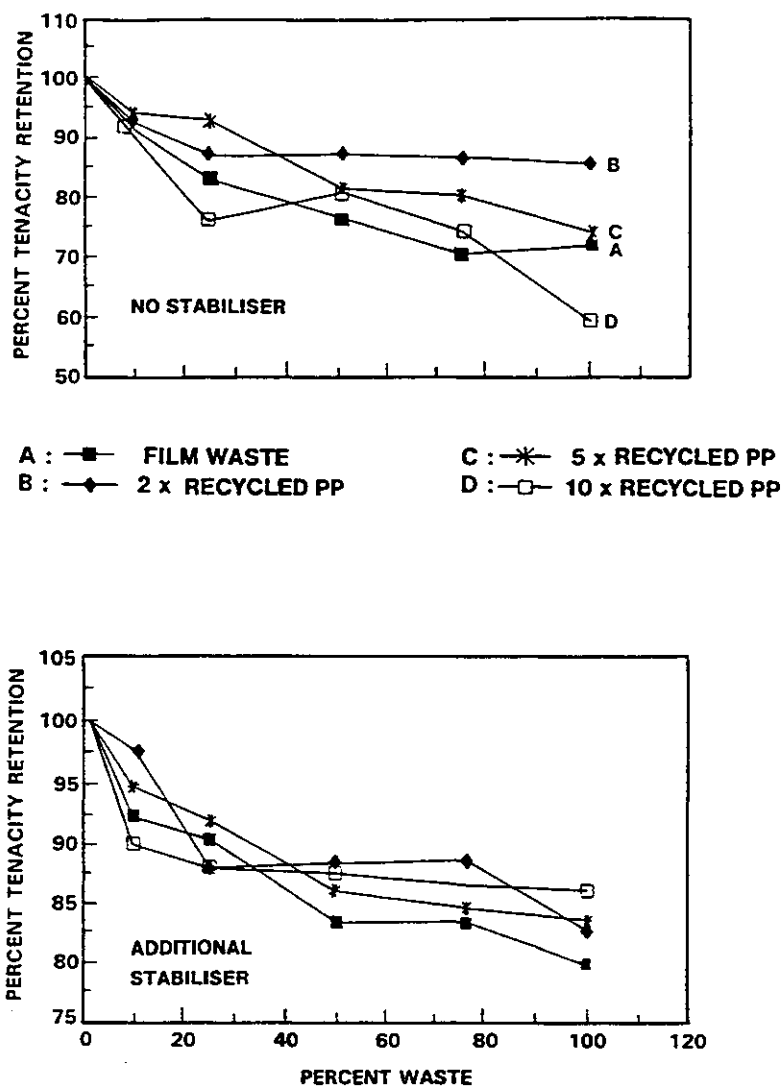


Figure 1: Effect of addition of various polypropylene wastes, without and with additional stabiliser

IR Crystallinity Index: Figures 2 and 3 show the changes in crystallinity index as functions of increasing waste A (industrial) and B (10x re-extruded virgin) concentrations. These U-shaped curves are typical of many polymer property dependencies on concentration of an interactive mixture of two similar components. It is well known⁽⁸⁾ that mixing two similar and miscible polymers produces such a degree of crystallinity versus concentration dependence as a consequence of the presence of a smaller concentration of either component reducing the ordering tendency and hence crystallinity of the major component. In Figure 2 and 3 it is interesting to note that pure polymer, which has the highest order, shows a rapid drop on addition of small (<25%) amounts of waste, passage through a minimum and then a gradual rise to the lower degree of crystallinity of the respective 100% waste orientated tape. Thus the U-shaped curve could be explained in terms of the change from a more highly ordered polycrystalline state (the virgin polypropylene) to one having lower order (the 100% recycled polypropylene). Intermediate mixtures or blends of virgin and waste represent the effect that the minor component has on the major component present which necessitates passing through a minimum state of order. The causes of the change in polycrystalline order between each component is a consequence of differences in thermal oxidative histories which give rise to variation in molecular weight averages and distribution and the presence of oxidative centres within polymer chains.

The effects of stabilisers on tape IR crystallinity were not found to be significant according to an analysis of variance. A more comprehensive study on the fine structure may provide a better understanding of the role that stabilizers have on tape morphology.

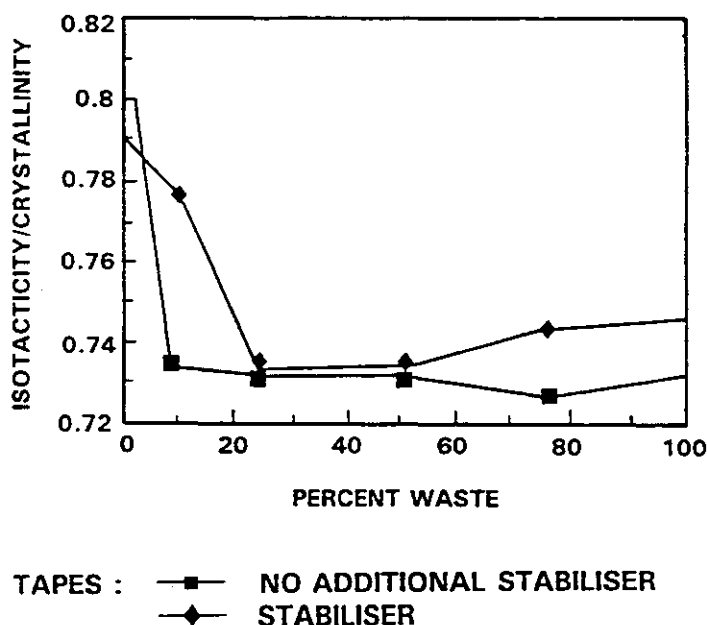


Figure 2: The relationship between crystallinity index and waste concentration when industrial film waste, polymer A was added.

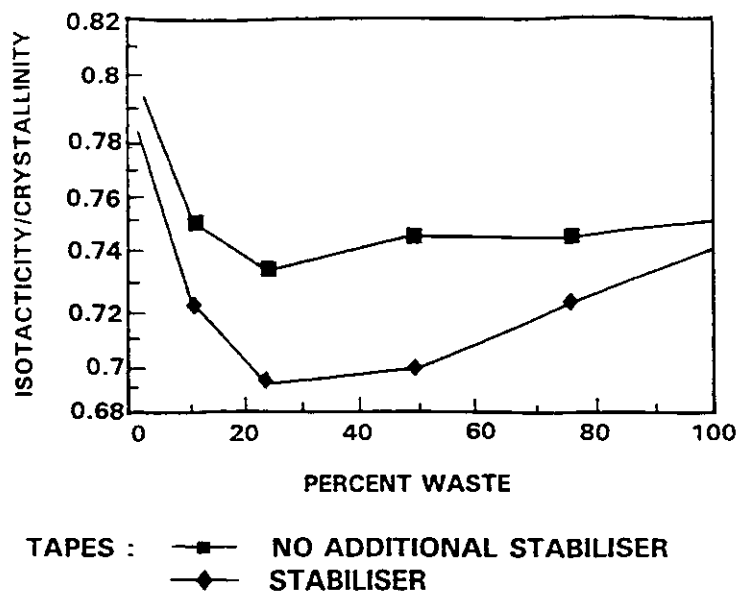


Figure 3: The relationship between crystallinity index and waste concentration when 10x recycled Isplen PP040, polymer D was added.

The sudden drop in crystallinity at low waste concentrations correlated with the similar reductions in tenacity. As it passes through a minimum with increasing waste content, so tape tenacity loss reduces. The lower sensitivity of tenacity at waste concentrations of 50% and above may be partly explained by subsequent increased ordering which offsets the counter effects of increased chain scission. Thus it is possible that the effects of adding waste-containing oxidative centres are to promote oxidative chain scission to the polymer mixture during extrusion which at low levels causes reduction in tape crystallinity and tensile properties i.e., tenacity. At higher waste levels, the increased chain scission enables the effect of the initial crystallinity reductions to be offset as reordering tendencies increase once waste levels exceed 50% by weight.

Summary

It has been shown that while the addition of recycled materials into virgin polymers has significant effects on oriented tape properties, useful tape having acceptable tensile properties (tenacity > 0.5 tex⁻¹) can be produced in absence of added stabilizer using up to 25% w/w degraded recycled wastes from three out of four waste types investigated. Use of additional stabilizers shows a significant improvement in tensile property retention and MFI. A

stabilizing system consisting of a primary hindered phenol e.g., Irganox 1010, and a secondary organic phosphite antioxidant, e.g., Irgafos 168 at an appropriate concentration and mass ratio, is particularly effective in counteracting the otherwise oxidizing effects that added waste has on resulting tape properties. The HALS stabilizers such as Tinuvin 770, normally present to function as a UV stabilizer may also offer some thermal protection whereby the hindered amine is oxidized to form nitroxyl radicals which react with free radicals in the polymer⁽⁷⁾. Waste-sensitized thermal degradation of polypropylene is significantly impeded by the presence of these stabilisers as indicated by reduced MFI values and losses in tenacity. Under these circumstances, upto 75 % by weight waste may be added to the virgin polymer and yield acceptable commercial tape tenacities. It is probable that certain types of sorted, clean consumer waste may also be usefully added to virgin polymer and find acceptability in geotextile products. The issue of durability⁽¹⁰⁾ has not, however, been addressed in this paper and is currently being investigated.

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THE ROLE OF PROCESS STABILISERS IN RECYCLING POLYOLEFINS

H(Heinz) Herbst, K Hoffmann, R Pfaendner and F Sitek

Introduction

For ecological, economical, and political reasons, material recycling is of eminent importance as an integral component in the efforts of plastic wastes in terms of:-

- reduction of raw materials
- reuse
- material recycling
- chemical recycling
- incineration with energy recovery
- landfill reduction

Value after regeneration via thermo-mechanical processes can be achieved with high value products. Coupling qualitatively high value products with old, used plastics is often perceived today as a contradiction.

Plastics and thus also recyclates, are subjected to damage of the polymeric chains during every processing step and long term use by oxidative and/or photooxidative ageing. Damage to the plastics by shear forces, heat or radiation (light), and oxidation often proceed in a cascading or concerted manner. Traces of transition metals, heat, light and predamage (see Figure 1) [1 -3] have an accelerating effect.

Oxidation products such as hydroperoxides, carbonyl groups and double bonds are chromophores which, among others, interact with light. They enhance photooxidation and consequently are starting points for progressive degradation (see Figure 2).

The oxidation process and the oxidation products [4, 5] can be blocked effectively and efficiently by stabilizers (eg. antioxidants and processing stabilizers such as phosphates).

These processes are usually polymer-specific and lead to molecular weight degradation by chain scission or crosslinking of the polymeric chains. In both instances the consequence is deterioration of mechanical properties up to total uselessness of the products. Ageing processes are, furthermore, the reason for macroscopic surface damage such as discoloration, bleaching, crack formation, etc., that may also contribute to the product's failure.

Qualitatively high value products, particularly from used plastics, are only possible if the above-mentioned deleterious influences are opposed in good time. They have to be neutralized effectively during the product's life cycle or be eliminated or inhibited by means of "repair" mechanisms.

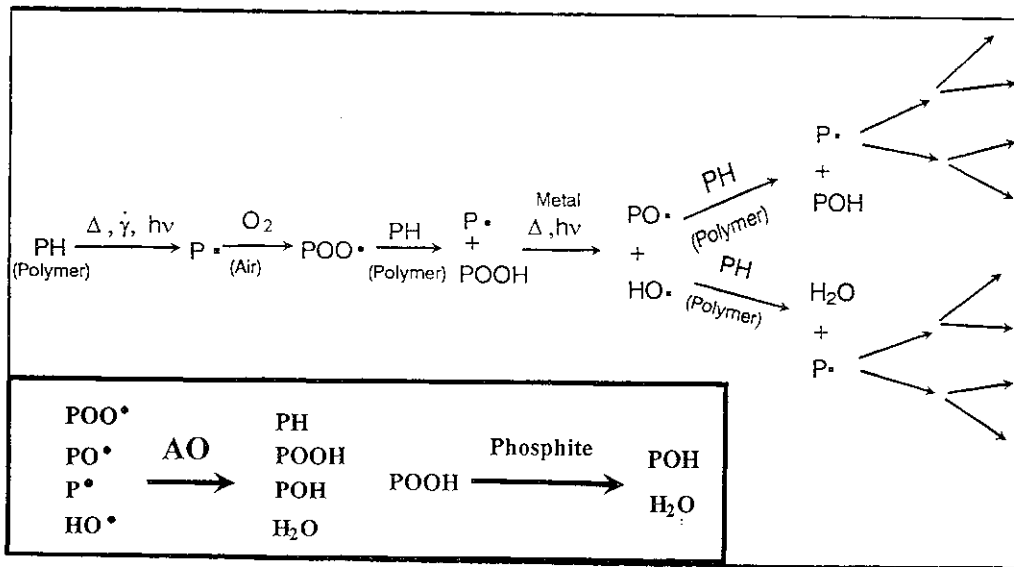


Figure 1: Oxidation and stabilisation of plastics

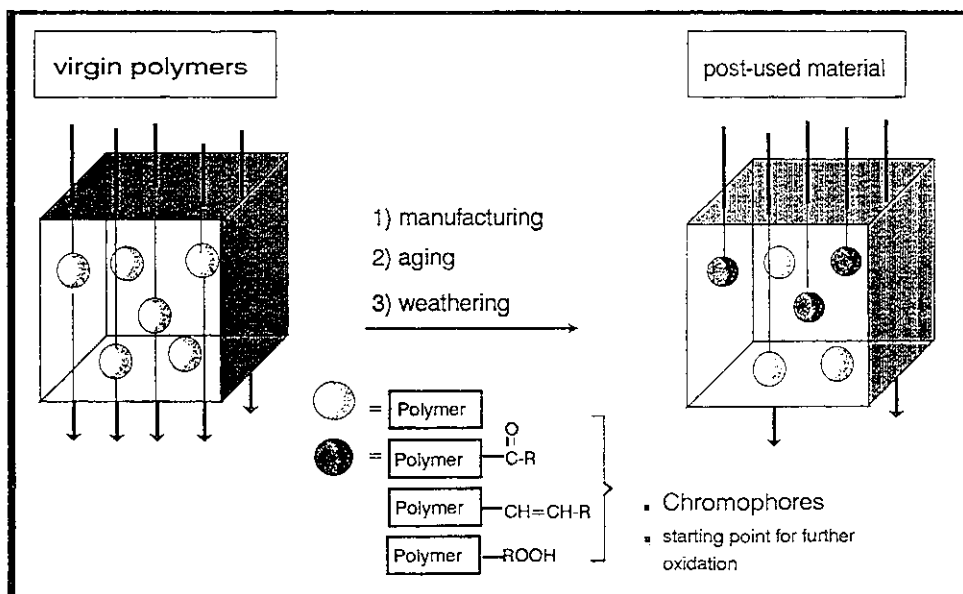


Figure 2: Photo-oxidation of plastics

Reasons for recyclates usage are based on eco-marketing considerations, sometimes environmental and legislative measures or simple price advantage. Another market segment for recyclates is the substitution of classic materials such as hardwood, concrete, etc. It is precisely their plastics-specific properties from which technological advantages can be derived which lead to low system costs as long as comparable life-times can be achieved in the products containing recyclates.

The following selected examples demonstrate quality improvement and quality assurance achievable by means of additives whose contribution to high-value plastics articles is of eminent importance.

High Density Polyethylene

A considerable fraction of plastic domestic refuse of interest for many applications comprises HDPE blow-moulded articles. Although relatively good segregation from the total waste flow is possible, impurities such as polypropylene (PP) closures and bottles from other materials (PVC, PP, PET) cannot be completely excluded. For example, long term thermal stability of a 95 : 5 polyolefin mixture (HDPE : PP) is affected by the PP content. Injection-moulded test specimens after one and five extrusions, if stored in a circulating air oven at 120°C until brittleness shows, in the absence of stabilizers, failure of the bending test after 18 and 14 days respectively as Table 1 shows. Restabilization with 0.2% Irganox B 225, a typical stabilizer for virgin material, improves long term thermal stability to 37 and 25 days. Still better stability is obtained with Recyclostab 451 - a Ciba Geigy stabilizer developed especially for recyclates - 116 and 115 days respectively were achieved. Noteworthy also is that the difference between the first and fifth extrusions is diminished in terms of differences in enhanced embrittlement times.

Table 1: Effect of additional stabilisers on thermal stability of P/PP mixtures.

Sample P/PP	Stabiliser	Days to embrittlement, one extrusion	120°C five extrusions
95:5	None	18	14
	0.2% Irganox B225	37	25
	0.2% Recyclostab 451	116	115
90:10	"	94	84
90:20	"	74	57
80:20 (+ 0.20% PVC)	"	50	35

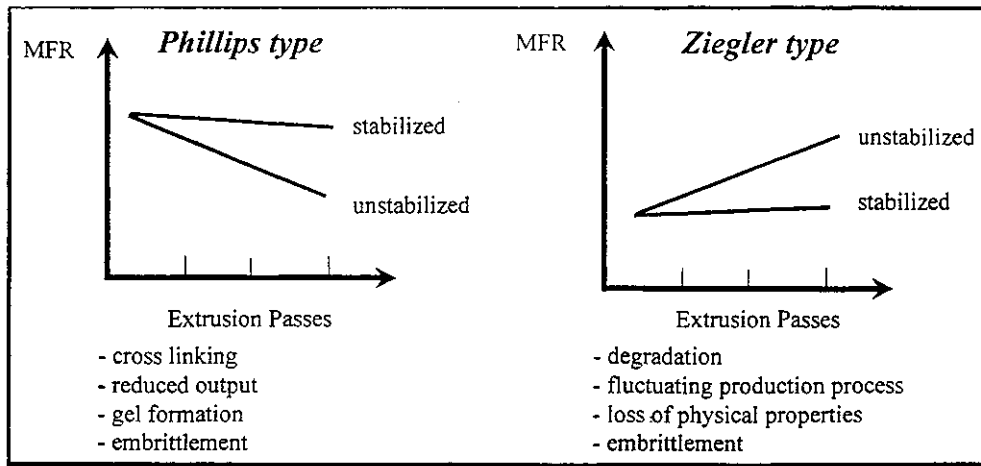


Figure 3: High density polyethylene processing behaviour

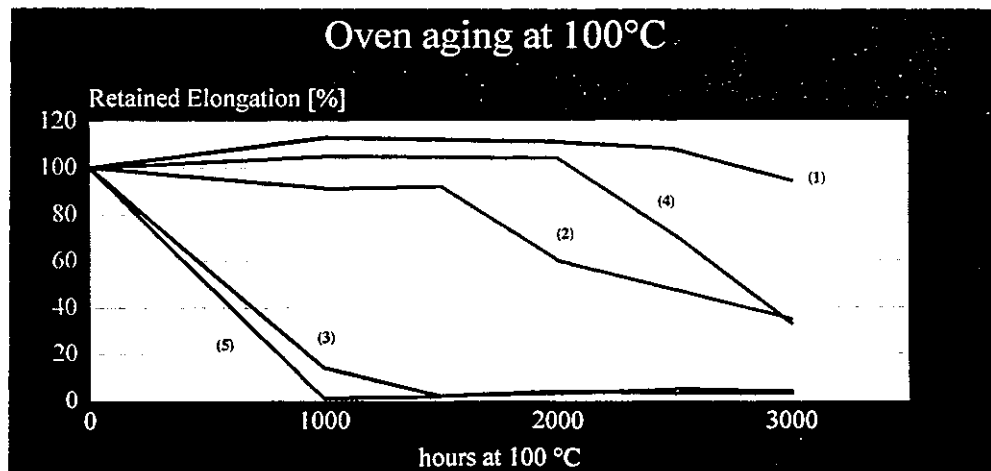


Figure 4: Irrigation pipe from LLDPE/LDPE recyclate oven-aged at 100°C;
Key- (1) 100% virgin stabilised;(2) Recyclate, 2.5%CB/0.3% Tinuvin 622;(3) Recyclate without restabilisation;(4) Recyclate, 2.5%CB/0.3% Tinuvin 622/0.1% Irganox 1010;(5) Recyclate, 2.5%CB (CB = carbon black).

The effect of increasing polypropylene content or traces of polyvinyl chloride is demonstrated in Table 1. At the same restabilization with 0.2% Recyclostab 451 and oven-ageing at 120°C, time to embrittlement of the test specimens is reduced dramatically with increasing polypropylene content. Moreover, the difference between first and fifth extrusions is again evident. These experiments indicate that, within certain limits, constant composition of the recyclate is an important quality criterion.

The molecular weight-stabilising effect of a combination of phenolic antioxidants and phosphates has been demonstrated by multiple extrusions of various polyethylene recyclates as bottles [6, 7]. Thus for example, a thorough study with HDPE (blow moulded) has shown that the addition of a stabilizer (phenolic antioxidant and phosphite) leads to uniform wall thickness and consequently, permits increased proportion of recyclate in blends with virgin material [7].

The behaviour of polyethylene during processing and ageing depends on the catalyst type used for its production (see Figure 3). HDPE of the classical Ziegler type exhibits molecular weight degradation, while Phillips-catalysed HDPE leads to crosslinking up to gel formation and thus molecular weight increase [8].

In working with HDPE recyclates it should always be borne in mind that one may be dealing with a blend of various types of fluctuating composition. Therefore, restabilization systems have to cope with both degradation mechanisms and in this way ensure uniform processing.

Multiple processing of Phillips - type HDPE leads to reduction of the melt flow, i.e. to crosslinking and in the worst case, to gel formation. Restabilization with the combination Irganox 1010 and Irgafos 168 in 1 : 1 ratio permits maintaining the melt flow rate or index value after five extrusions. To achieve this effect, at least 0.1% of each component is necessary, although in the recyclate a stabilizer residue concentration of approximately 0.09% phenolic antioxidant and 0.03% phosphite was still found. The same result was achieved with 0.2% Recyclostab 411 developed specifically for recyclates.

Results of multiple extrusions of a Ziegler-type HDPE which tends to molecular weight degradation for the same application shows that a combination of Irganox 1010 and Irgafos 168 (1 : 1) is inferior to Recyclostab 411 with regard to molecular weight conservation during multiple processing.

Pipe applications: Carbon black-pigmented irrigation pipes for vineyards in Cap Province, South Africa manufactured with LLDPE/LDPE recyclate over the course of one year showed crack formation and breakage which did not occur with pipes made of virgin material. Artificial weathering experiments revealed quickly that pigmenting with 2.5% carbon black alone does not impart sufficient protection against light. Maintenance of physical properties during 9000 hrs artificial weathering, corresponding to natural weathering in South Africa of 2 - 3 years (approx. 160 kLy/year), requires the use of additional HALS light stabilizers such as Tinuvin 622. In addition, the thermal stress on black pipes in such applications should not be underestimated. Elevated temperatures accelerate thermal ageing and sometimes carbon black can have a negative effect on thermo-oxidation of polyolefins [9, 10]. Adequate lifetime can, therefore, be achieved only if the restabilization formulation

contains along with light stabilizers also thermal stabilizers such as e.g. the phenolic antioxidant Irganox 1010 (see Figure 4).

A second example presents results obtained in a project concerned with optimization of raw materials for LDPE/HDPE recycle-containing pipes.

Oven ageing of LDPE/HDPE recycle at 110°C exhibits crack formation after only 52 days without restabilization. This effect is observed after 97 days if the material is restabilized with 0.033% Irganox 1010, 0.067% Irgafos 168 and 0.1% HALS, Chimassorb 944. Ageing resistance can be further enhanced by the use of Recyclostab 411 (0.1%) and Recyclostab 421 (0.1%) stabilizers developed specifically for recycles, to 106 days, and 116 days respectively, or to 125 days with 0.2% Recyclostab 421 + 0.1% Chimassorb 944. Complementary to these ageing studies, tests are being carried out at present involving storage in water at 80°C and natural weathering in South Africa.

Repair concept

As already discussed stabilizers enable processing and permit long term applications of virgin materials by providing protection against oxidation and photo-chemical ageing processes. It follows that almost all virgin plastics contain tailor-made stabilization packages focused on the specific processing and the specific application [1, 2].

Although the fundamental importance of restabilization of recycles has been stressed by various authors in many publications [6, 11 -21], this is frequently neglected. It is often forgotten that stabilizers are added for one-time processing and first use conditions only.

Decisive considerations become necessary if recycles from a short-term application (e.g. packaging) are to enter long-term applications. Such materials contain from their original use insufficient stabilizer residues (eg antioxidants) and little or no light protection for the second application. In addition to the usual effect of stabilizers against degradation in virgin plastics, the deleterious influence of previous damage and impurities (as initiating sites) in recycles has to be compensated or eliminated. It is, therefore, not surprising that stabilizer combinations are already on the market aimed at the particular characteristics of recycles [22,23]. For the future is the development of a broad range of special additives and additive blends for recycles [11, 24].

Heavily damaged materials would require considerable improvement of their physical properties if they are to be used in high-value secondary applications. In this context, reactive additives will gain particular importance by offering the possibility of repair or elimination of damaged sites or the bonding of damaged polymeric chains. Such "repair"-molecules are already mentioned in the literature [25 - 30] in connection with recycles and virgin plastics.

PP/EPDM Automobile bumpers as recycles

Bumpers are important materials for recycling because they can be recovered easily when disassembling used cars [31]. This is old material whose surface has been damaged in a

relatively severe way because of outdoor use. Contaminants are frequently paint residues which, as is well known, have a deleterious effect on mechanical properties such as notched impact strength [32]. The influence of paint residue on molecular weight degradation is relatively small during processing, however, thermal ageing of painted bumper production waste leads to extremely high deterioration.

This extreme loss of long term properties can no longer be compensated by stabilization alone. Possibly certain improvement can be achieved by the addition of a thiosynergist [13]. However, the values of new material after ageing cannot be reached any longer. Stabilization of a paint-containing recyclate has to compensate the negative effect of the paint which is very often polyurethane-based. Surprisingly, with a new stabilizer combination, containing a reactive additive, molecular weight deterioration can be compensated and long term thermal stability improved. By this extrusion process, not only processing and long term properties are raised, but also mechanical properties such as e.g. elongation and impact tensile strength, so that the resulting material becomes comparable to virgin product.

Conclusions

Data in the literature and experiments outlined above already allow the conclusion that reuse of recyclates is possible in demanding applications, provided the correspondingly necessary stabilization has been applied. This is equally valid for single plastic recyclates, recyclate blends and also for mixed plastics.

Furthermore, it should be noted that because of initiator-(faulty) sites, old material degrades faster and differently and, therefore, in many instances, specially designed stabilizer systems are required. Although there are at present no sufficient scientific investigations concerning the oxidative and photo-oxidative behaviour of recyclates, already a number of applications have been realized in a variety of industries (e.g. packaging, automotive, construction). Hence, in summary, the following can be stated regarding restabilization of recyclates:

- Restabilization is absolutely necessary for the use of recyclates in high value applications.
- Restabilization has to take into account previous damage, subsequent application and residual stabilizer content and has to be specific for a given polymer.
- Sufficient amount of processing stabilizers is needed to reduce as much as possible damage to the recyclate during processing. To this end, combinations of phenolic antioxidants and phosphites are primarily recommended.
- Light stabilization is highly recommended for outdoor applications. The products of choice are HALS compounds or combinations of HALS compounds with UV absorbers.

Furthermore, the repair concept by use of reactive additives has to be taken into consideration with heavily degraded polymers and detrimental impurities.

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RECYCLING CARBON FIBRE-PEEK COMPOSITES

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Introduction

Modern polymer based composites date from the turn of this century, these being based on thermosetting polymers, such as phenolic resins, coupled with natural-fibre reinforcements such as paper. The early 1950's saw the introduction of *glass reinforced plastics*, this resulting from the concurrent development of thermosetting polymers, for example unsaturated polyester resins, and the technology for the successful production of suitable glass fibre. However, these materials are not easily recycled due to the, often, highly-cross-linked structure.

Thermoplastic based composites were not introduced into the market until the 1960's, these principally being of the short-fibre variety, the reinforcement overcoming some of the mechanical limitations of the materials. However, whilst these materials have the benefits of recyclability, processing of short fibre composites can lead to products with anisotropic properties due to the orientation of the fibres generated by the flow processes occurring during fabrication.

This presentation concerns the recycling of carbon fibre-PEEK composites. PEEK^{1,2}, poly(ether ether ketone), is a semi-crystalline thermoplastic engineering polymer. It exhibits a glass transition temperature around 143°C and the crystalline melting point is around 334°C, the degree of crystallinity typically being around 30%. The polymer exhibits good chemical and thermal resistance.

Carbon fibre-PEEK composites are available in two forms, a short fibre reinforced material and a uniaxial continuous carbon fibre-PEEK pre-preg. The short fibre reinforced material can be processed by the routes usually used for thermoplastics such as injection moulding.

The uniaxial continuous carbon fibre-PEEK pre-preg, marketed commercially under the name APC-2 (*aromatic-polymer composite*), is typically utilised by a hand lay-up process followed by compression moulding, sheets of pre-preg being cut to the shape required to fit the mould. The fibre orientation in the product is controlled by changing the relative axis of the successive layers of pre-preg. This type of process generates quantities of offcuts and this presentation considers the recycling of this waste into a short fibre composite, suitable for injection moulding. McGrath and co-workers^{3,4} and Belbin *et al*^{5,6} have also considered the recycling of APC-2, the route used by the latter being similar to that investigated in this work.

Experimental

The material used for this work was in the form of a roll of the composite rather than actual offcuts. The reprocessing route consisted of three stages:

- (1) Size reduction of the offcuts.
- (2) Compounding of the size-reduced offcuts with more PEEK.
- (3) Fabrication of components, in this case, suitable test pieces.

Size Reduction: The offcuts from the moulding of the APC-2 can vary in size but will typically contain fibres very much larger than those normally found in a composite that can be processed by injection moulding. Typical fibre lengths in a short fibre-reinforced composite are of the order of 3mm.

The initial stage of size reduction involved feeding the strip of composite from the roll into an office paper shredder in order to generate composite flakes, typically 5mm square.

In order to produce a successful injection-moulding compound the fibre content of the recycled material needed to be reduced from that found in APC-2, typically around 60%, to 20%-30%. This required the addition of further PEEK resin (LNP LC1006), this resin being chosen on the basis of its suitability for the injection moulding process. As will be shown later, the molar mass of this resin was substantially lower than that found in the pre-preg material. Attempts to use the flake directly, along with a quantity of unfilled PEEK resin, to produce injection moulded components did not prove successful due to the poor dispersion of the fibre in the components although this method would represent the simplest recycling route. The difficulties experienced arose due to:

- (i) The relatively low shearing forces present during the injection moulding process.
- (ii) The rheological differences in the original resin, found in the pre-preg, and that added to reduce the fibre content of the composite.

As a consequence methods were investigated whereby the size of the flakes could be further reduced in order that this recycling method could be utilised. However attempts to reduce the size of the flakes by simple mechanical means proved unsuccessful. The methods evaluated included cryogenic grinding, the use of an internal mixer (Midget Banbury) and ball milling.

Compounding: As a result of the difficulties found with the direct injection moulding of the flake/PEEK mixture, the mixture was compounded using a Betol BT30S co-rotating twin-screw extruder, the ratio of flake to additional PEEK being such that the product contained approximately 25% by weight fibre.

The extruder was equipped with 30mm diameter screws of length-to-diameter ratio of 22:1. The processing conditions are given in Table 1. The extruder was thoroughly cleaned, using a fluidised bed, prior to the compounding operations as any polymeric residues from previous compounding operations were likely to degrade at the elevated temperatures used to process the PEEK, this resulting in contamination of the product.

Extruder Temperature Settings	
Die	380 °C
Front	370 °C
Centre	360 °C
Rear	350 °C
Extruder Screw Speed	70 rpm

Table 1: Extrusion processing conditions.

Parameter	Commercial	Recycled
Temperature Settings / °C		
Nozzle	399	399
Front	399	399
Centre	390	390
Rear	370	370
Mould	157	157
Cycle Timing / s		
Injection time	1	1
Hold-on time	10	10
Cooling time	40	40
Melt Pressure / bar		
Injection pressure	990	690
Hold-on pressure	950	600
Back pressure	150	20

Table 2: Injection moulding conditions.

Moulding: Test pieces were produced from the compounded recycled material and a commercial short fibre reinforced carbon fibre-PEEK composite, based on LNP LC1006, containing 30% by weight of fibre. Test specimens were manufactured using a Negri-Bossi NB60 microprocessor-controlled injection moulding machine, the operating conditions being given in Table 2. As was the case with the twin-screw extruder, the injection moulding machine needed to be cleaned thoroughly before moulding.

$$E = \frac{PL_0^3}{4CD^3Y}$$

The tensile and flexural moduli of the moulded products were determined, the samples being conditioned at 23°C and 50% relative humidity prior to testing. The flexural modulus was calculated using the expression⁷ below:

where P = applied force (N)

L₀ = Distance between supports = 50mm

C = specimen width (mm)

D = specimen thickness (mm)

Y = specimen deflection (mm) under load P

Fracture surfaces were viewed using a Philips 525 scanning electron microscope (SEM).

The effect of processing on the molar mass distribution of the polymer and the fibre length distribution of the reinforcement was determined. Determination of the viscosity average molar mass was done by solution viscometry using an Ubbelohde viscometer, the PEEK being dissolved in concentrated sulphuric acid, any fibres present being filtered off. The fibres, thus collected, were weighed to allow the determination of the fibre content of the composite, and their lengths measured so that the fibre length distributions occurring in the composites could be determined. Fibre length measurements were carried out by photographing the fibres under a Vickers optical microscope. Viewed using a video camera, the image produced was analysed using a Magiscan image analysis system. Approximately 500 fibres were measured for each sample.

The thermal characteristics of the recycled and commercial materials were determined using differential scanning calorimetry (DSC), on a DuPont 2000 analyst system. The melting temperature of the materials, T_m, was taken as the temperature at the peak of the melting endotherm. Degree of crystallisation was determined by measuring the heat of fusion of the sample and comparing this with the heat of fusion for totally crystalline PEEK⁸, this being taken as 130 J g⁻¹.

Material	Condition	$(M_v)_s$
PEEK LN LC1006	As-received	15 200
APC-2	As-received	35 800
Recycled material	before injection moulding	23 200
	after injection moulding	22 400
Commercial material	before injection moulding	25 700
	after injection moulding	24 200

Table 3: Viscosity-average molar masses, $(M_v)_s$, of PEEK samples.

Material	$T_m / ^\circ\text{C}$	Crystallinity / %
PEEK	345	37
APC-2	342	27
Recycled material*	343	36
Commercial material*	338	26

Table 4: Thermal characteristics of the PEEK materials.
(* - after moulding)

Material	Stage of Processing	Weight-average fibre length (μm)	Number-average fibre length (μm)
Recycled composite	before moulding	271	200
	after moulding	212	155
Commercial composite	before moulding	186	145
	after moulding	151	109

Table 5: Fibre length distributions in the recycled and commercial composites

Results and Discussion

The viscosity average molar masses of the PEEK components of the composites are shown in Table 3 this indicating, as would be expected^{9,10}, that little degradation occurred as a result of processing and shows the similarity between the molar masses of the recycled and commercial materials.

Table 4 shows the thermal characteristics of the recycled and commercial materials, slight variations being observed in the T_m of the PEEK. However, large differences are observed in the degree of crystallisation in the various materials. The degree of crystallinity can have a significant effect on the mechanical performance of PEEK^{10,11}, and thus the recycled material would be expected to have superior performance to the commercial material.

The results regarding the fibre length distributions are shown in Table 5. It can be seen that the average fibre length in the recycled material is longer than that found in the commercial material. In both cases, a significant amount of fibre breakdown occurs on injection moulding. The greater reduction in average fibre length found in the case of the commercial material probably results from the increased fibre loading, this leading to a greater likelihood of fibre-fibre interactions during processing¹².

Material	Young's Modulus / GPa	Ultimate Tensile Stress / MPa	Flexural Modulus /GPa
Recycled material ($w_f=25\%$)	26 ± 2	240 ± 13	16.9 ± 0.2
Commercial material ($w_f=30\%$)	20 ± 2	202 ± 6	16.9 ± 0.3

Table 6: Mechanical properties of the recycled and commercial composites

The results from the mechanical tests carried out are given in Table 6. It can be seen that the tensile properties of the recycled composite are around 25% better whereas the flexural properties are similar. The difference in tensile properties is perhaps a little surprising due to the higher fibre loadings in the commercial material, although the average fibre length in the recycled material is higher as is the degree of crystallinity. The results obtained for the commercial material were consistent with those supplied by the manufacturer^{3,4}.

It is apparent from the SEM micrographs of fracture surfaces that failure in the commercial material was accompanied by substantial fibre pull-out whereas in the case of the recycled material the majority of fibres failed at the crack face. This suggests that the matrix fibre bonding was stronger in the recycled materials, this serving to improve the mechanical properties. This may in part result from the manufacturing process for the pre-preg. Some bundles of fibre were evident at the failure surfaces in the recycled materials, indicating that the mixing of the APC-2 composite with the PEEK resin may not have been complete.

Economics of Recycling

The cost of recycling APC-2 in the manner described above was determined, based on:

- (1) The scrap APC-2 is supplied to the recycling scheme free of charge and is readily available.
- (2) Compounding is carried out using a twin-screw extruder, capital cost £150 000, running costs £20 h⁻¹ which is run on a 40 h week.
- (3) Operator costs are £10 h⁻¹.
- (4) The capital cost of the extruder is to be recovered over a two year period.
- (5) The cost of the PEEK resin used to reduce the fibre concentration in the composite is £46 000 T⁻¹.
- (6) The output rate from the plant is 250 kg h⁻¹, full production being assumed.

This yields a cost of under £23 000 T⁻¹, which is very much less than the price of the equivalent commercial compound, this being of the order of £49000-56000T⁻¹. The price of the recycled material is relatively insensitive to all factors excluding the price of the PEEK polymer used in the formulation.

However, whilst the economics of the process seem advantageous, it should be remembered that the quantity of offcuts produced is limited and probably widely distributed. Hence there may be a problem obtaining sufficient material to set up a commercial scale reprocessing facility. It has also been assumed that the offcuts are supplied free of charge.

The modelled recycling system would generate 500 T of composite annually which represents a substantial proportion of the estimated market for the material, and would require 250 T of scrap APC-2. It is not known whether the latter quantity is realistic. However, if smaller quantities were available, the costs would be such that recycling would still be beneficial.

Conclusions

The results indicate that APC-2 composite can be reprocessed to produce short fibre composite suitable for the use in the injection moulding process. The properties of the recycled material are shown to be superior to that of an equivalent commercial grade of material. The economics of the recycling process indicate that the route is commercially viable, the main problem being the collection of sufficient scrap APC-2 to make the recycling process worthwhile.

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