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Fibre labelling
Polylactide – Cargill Dow

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Analysis conducted on behalf of DG ENTERPRISE

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1. Executive summary

In January 2002 Cargill Dow presented an application to the European Commission for the establishment of a new generic name for their new fibre in accordance with Directive 96/74/EC. The Commission therefore convened two meetings of the technical working group for Directive 96/74/EC on textile names, comprising governmental experts representing each Member State. The meetings were held in Brussels on 12th April and 25th September 2002. The application was found to be justified by the group of experts who recommended an amendment to the list of fibre names in Annex I of Directive 96/74/EC. The name proposed by the Commission for their new fibre is polylactide and it will be thus indicated for the purpose of this report.

The group also decided that the Commission would take the necessary steps to prepare for the validation of the proposed test method for the quantification of polylactide in binary blends, taking into consideration the similar method 6 in Directive 96/73/EC. The Directorate General Joint Research Centre (JRC) was then asked to conduct experimental work to check the validity and suitability of the quantitative method for the analysis of binary mixtures containing polylactide, and to verify the qualitative method for the identification of this new fibre. The determination of the value of agreed allowance for polylactide was also part of this work.

As the result of a consensus, method 6 in Directive 96/73/EC was chosen as the quantitative method for testing on the basis of strong similarities with the method proposed by the applicant and the fact that method 6 is already validated.

The whole set of analyses performed by the JRC confirmed that method 6 is suitable for the quantification of polylactide in binary mixtures with polyester, wool, cotton, viscose, acrylic, nylon and silk. The results showed that method 6 gave a good repeatability, demonstrated by the low values of the relative standard deviation (RSD) used to measure the dispersion of the distribution of test results in one laboratory, both for binary mixtures containing polylactide and for samples of pure fibres. Analyses performed by two other European enforcement laboratories confirmed that there were no relevant systematic errors due to laboratory bias. Results of some tests on yarns of different thickness showed that the conditions of method 6 were strong enough to completely dissolve polylactide, even in the case of thick yarns.

Microscopic analysis confirmed that complete dissolution occurred during the technical work as a whole.

The qualitative method proposed by the petitioner, based on FT-IR analysis of polylactide film, was found to be suitable for the scope of identifying the new fibre, whereas simple microscopic analysis could not always give an unequivocal identification.

The measured agreed allowance for polylactide ranged from 0.22 to 0.32, depending on the type of sample (e.g. yarn versus staple fibre).

On the basis of preliminary experimental results presented at a meeting organised by the JRC in Ispra on 3rd July 2003, the group of experts from enforcement laboratories representing the Member States agreed on the applicability of method 6 and on the suitability of the qualitative method. The experts accepted the value of agreed allowance for polylactide proposed by the petitioner (1.50), even if the experimentally measured value was different, as it was in line with the values of agreed allowances listed in Directive 96/74/EC for other fibres that are all higher than the experimental ones.

The global results presented in this report confirm these preliminary conclusions.

2. Introduction

In January 2002, the European Commission's Directorate General Enterprise received an application from Cargill Dow for the establishment of a new generic fibre name under Directive 96/74/EC on textile names [1]. The name proposed by the Commission for their new fibre is polylactide and it will be thus indicated for the purpose of this report.[1] The proposed definition of polylactide states that it is a manufactured fibre in which the fibre-forming substance is composed of at least 85% (by mass) of lactic acid ester units derived from naturally occurring sugars, with a melting temperature of at least 135 ºC.

Two meetings of the "Technical Expert Working Group on Textile Labelling" composed of Member States' governmental experts associated with the "Committee for Directives relating to Textile Names and Labelling" were held on 12th April 2002 and 25th September 2002 to evaluate the dossier presented by the applicant. The evaluation was based on the following agreed set of criteria:

- the new fibre should be radically different from other fibres by chemical composition and/or by manufacturing route and production process;
- fibre characteristics can be taken into account but need to be examined on a case by case basis;
- the new fibre should be detectable and distinguishable from other fibres by standardised test methods;
- consumer relevance should be shown by active commercial use of the fibre;
- a new name is only justified if the fibre cannot be classified into existing groups.

The group of experts was of the opinion that the petition was justified and that experimental work was needed to verify the applicability of proposed analytical methods for identifying and quantifying polylactide in blends (see Annex I). An amendment to Directive 96/74/EC on textile names could subsequently be prepared. It was then decided that a validated test method, enabling market surveillance authorities in Member States to determine the fibre composition of textile products containing the new fibre, should be established at European level and that the Commission would take the necessary steps to prepare for the validation of the quantitative method proposed by the applicant.

[1] This fibre is commonly reported with the abbreviation PLA in scientific literature and in the USA.

As a result of discussions between the Joint Research Centre (JRC) and DG Enterprise concerning the analytical work involved, it was established that the role of the JRC would be to verify the validity and suitability of the quantitative method for the analysis of the composition of binary mixtures containing polylactide and to coordinate the subsequent ring trial, as well as to verify the qualitative method for the identification of this new fibre.

3. Verification of the applicability of method 6 in Directive 96/73/EC for the quantification of polylactide in relevant binary mixtures

3.1 Background

The original working scheme involved an in-house verification and a comparison of Cargill Dow's method with method 6 in Directive 96/74/EC since they are similar, both in terms of procedure as well as in terms of applicability. During the "Technical Expert Working Group on Textile Labelling" meeting on 25th September 2002, an interesting proposal was made to demonstrate the comparability to method 6 by a smaller number of labs (e.g. 2 labs), with the purpose to provide preliminary data quickly and give enough confidence to the expert group and DG Enterprise to start the acceptation procedure for the polylactide label. Indeed, the quantification method proposed by Cargill Dow (see analytical method 2 in Annex I) is suitable for the compositional analysis of binary mixtures containing polylactide and natural fibres such as cotton and wool. It is a gravimetric method based on dissolving polylactide in dichloromethane and on subsequently weighing the residue. This procedure is very similar to method 6 in Directive 96/73/EC [2] (which is already validated, it is based on the same principle and solvent and it is suitable for the analysis of binary mixtures containing triacetate). The main differences consist in a lower consumption of solvent in the case of Cargill Dow's procedure and the fact that, contrary to the validated methods in Directive 96/73/EC, Cargill Dow's method does not base the calculation of composition on dry mass.

Following the visit of Cargill Dow representatives to the JRC on 15th January 2003 and to the Central Laboratory of the Belgian Ministry of Economic Affairs, where results of some experimentations on method 6 were reported, the conclusion that

method 6 could be tested directly for labelling quantification purposes was reached as, in principle, there was no need to introduce a new specific method requiring validation if method 6 could be used "as it is".

The JRC then carried out work to verify the validity and applicability of method 6 for quantifying polylactide in foreseeable binary mixtures. Relevant samples were selected in collaboration with the applicant, taking into account the market for polylactide and the possible range of compositions in blends. Polylactide's market is mainly foreseen to be in replacement of polyester in a mixture with other natural fibres, in particular wool and cotton, due to the nature of the new fibre which derives from natural sources. For this reason, binary mixtures of polylactide with wool, cotton and polyester in various percentages were initially analysed. The results of the in-house investigation into the applicability of method 6 were presented to experts from enforcement laboratories representing Member States at a meeting organised by the JRC in Ispra on 3th July 2003. The set of results was evaluated positively and, on that basis, the group of experts reached a consensus on the approach to be followed. It was agreed that there was no need to perform a time-consuming ring trial to validate the method proposed by Cargill Dow due to the applicability of method 6 (which was validated for binary mixtures of triacetate with wool, animal hair, silk, cotton, cupro, modal, viscose, acrylic, nylon, polyester and glass fibre), but it was decided to verify the applicability of the method for blends of polylactide in samples containing nylon, silk, acrylic and viscose.

3.2 Procedure

The JRC analysed all the samples received from Cargill Dow (listed in Table 1) using method 6 in Directive 96/73/EC.

Samples were pre-treated using the conditions reported in Directive 96/73/EC in order to eliminate non-fibrous matter that can be extracted with light petroleum ether and water. The procedure foresees a one-hour extraction in Soxhlet with light petroleum ether, followed by a one-hour extraction in water at room temperature and a one-hour extraction in water at 65 ± 5 ºC, using a liquor:specimen ratio of 100:1. Samples were then air-dried.

JRC code	Customer code	Composition	Arrival date	Note	Sample type	Color
001	322-99-1	100 polylactide 268 denier	15/12/2002		knitted sock	white
002	322-99-2	50 polylactide - 50 cotton	15/12/2002	Intimate staple blend, nominal %	knitted sock	off-white
003	322-99-4	100 polyester	15/12/2002		knitted sock	white
004	322-99-5	50.2 polylactide - 49.8 polyester	15/12/2002	% based on denier	knitted sock	white
005	322-100-2	100 cotton	15/12/2002		knitted sock	white
006	479-22-01	100 wool	15/12/2002		knitted sock	green
007	479-22-04	63.7 polylactide - 36.3 wool	15/12/2002	% based on denier	knitted sock	green
008	479-30-03	100 polylactide 268 denier	30/01/2003		knitted sock - fine	white
009	479-30-02	100 polylactide 654 denier	30/01/2003		knitted sock - coarse	white
010	322-99-1	100 polylactide 268 denier	19/03/2003		knitted sock	white
011	322-100-2	100 cotton	19/03/2003		knitted sock	white
012	479-22-04	63.7 polylactide - 36.3 wool	19/03/2003	% based on denier	knitted sock	green
013	479-22-01	100 wool	19/03/2003		knitted sock	green
014	322-99-5	50.2 polylactide - 49.8 polyester	19/03/2003	% based on denier	knitted sock	white
015	322-99-4	100 polyester	19/03/2003		knitted sock	white
016	322-99-2	50 polylactide - 50 cotton	19/03/2003	Intimate staple blend, nominal %	knitted sock	off-white
017	527-11-02	56.6 polylactide - 43.4 cotton	08/04/2003	% based on denier	knitted sock	white
025	479-39-01	40.2 polylactide - 59.8 cotton	18/06/2003	% based on denier	knitted sock	white
026	479-39-02	72.6 polylactide - 27.4 cotton	18/06/2003	% based on denier	knitted sock	white
027	479-39-03	55.8 polylactide - 44.2 wool	18/06/2003	% based on denier	knitted sock	off-white
028	479-39-04	38.3 polylactide - 61.7 wool	18/06/2003	% based on denier	knitted sock	off-white
029	479-39-05	38.1 polylactide - 61.9 polyester	18/06/2003	% based on denier	knitted sock	white
030	479-39-06	71.2 polylactide - 28.8 polyester	18/06/2003	% based on denier	knitted sock	white
031	538-02-01	100 polylactide	26/06/2003	scoured off prior to dyeing	staple yarn	ecru
032	538-02-02	100 polylactide	26/06/2003	washed off for analysis	staple fiber	white
033	479-39-13	55.4 polylactide - 44.6 viscose	05/08/2003	% based on denier	knitted sock	white
034	479-39-14	100 viscose	05/08/2003	% based on denier	knitted sock	white
035	479-39-15	55.6 polylactide - 44.4 acrylic	05/08/2003	% based on denier	knitted sock	white
036	479-39-16	100 acrylic	05/08/2003	% based on denier	knitted sock	white
037	479-39-19	55.8 polylactide - 44.2 nylon	05/08/2003	% based on denier	knitted sock	white
038	479-39-20	100 nylon	05/08/2003	% based on denier	knitted sock	white
041	479-39-07	56.1 polylactide - 43.9 silk	22/10/2003	% based on denier	knitted sock	ecru
042	479-39-08	100 silk	22/10/2003	% based on denier	knitted sock	ecru

Table 1: Samples received from Cargill Dow.

Note 1: Samples 002, 003, 004, 005, 006 and 007 are respectively the same as samples 016, 015, 014, 011, 013 and 012. Samples 001, 008 and 010 are the same.

Note 2: Denier is a weight-per-unit measure of any linear material, numerically equal to the weight in grams of 9000 meters of the material.

Pre-treated samples were cut into specimens of about 1 g, dissected as much as possible, dried, weighed and analysed using method 6 in Directive 96/73/EC. 100 ml of dichloromethane per gram of specimen were used to dissolve polylactide at room temperature under mild shaking conditions for half an hour. After decanting the liquid through a weighed filter crucible, 60 ml of dichloromethane were added to the

residue, manually shaken and filtered through the filter crucible. The residue was then quantitatively transferred to the filter crucible and some more dichloromethane was allowed to drain under gravity. Lastly, the residue was treated with boiling water to eliminate the solvent, dried and weighed. Before weighing, samples were dried in a ventilated oven at 105 ± 3 ºC, for 14 to 16 hours to reach a constant weight, then put in a desiccator for at least 2 hours to cool down, so that a dry mass was always measured and taken into account for subsequent calculations. All weighing operations were performed using an analytical balance with an uncertainty value of ± 0.0001 g.

After weighing, the residues were analysed by microscopy to verify the complete dissolution of polylactide. As an example, Fig.1 in Annex II shows the photo of a blend of polylactide and cotton. There are also some photos of cotton, which is the corresponding insoluble residue in dichloromethane (Figs. 2-4). These pictures demonstrate that polylactide was completely dissolved and that the residue was composed exclusively of cotton. Two photos of clean residues, composed of viscose and acrylic respectively, are also shown (Figs. 5-6).

The percentages of insoluble component on a clean, dry mass basis, disregarding loss of fibre mass during pre-treatment, were calculated using the following formula:

$$P_1\ \% \ = \ \frac{100\ r\ d}{m}$$

where:

P_1 is the percentage of clean, dry insoluble component

m is the dry mass of the specimen after pre-treatment

r is the dry mass of the residue

d is the correction factor for loss of mass of the insoluble component in the reagent during the analysis. For method 6 the value of d is 1.00, except in the case of polyester, for which the value of d is 1.01.

Calculations of percentage of insoluble component on clean, dry mass basis, with adjustment by conventional factors (agreed allowances) and, where appropriate, correction factors for loss of mass during pre-treatment were performed using the following formula:

$$P_{1A}\ \% \ = \ \frac{100 \ P_1 \left(1+\frac{a_1+b_1}{100}\right)}{P_1 \left(1+\frac{a_1+b_1}{100}\right)+(100-P_1)\left(1+\frac{a_2+b_2}{100}\right)}$$

where:

P_{1A} is the percentage of insoluble component, adjusted by agreed allowances and for loss of mass during pre-treatment

P_1 is the percentage of clean, dry insoluble component as calculated from the previous formula

a_1 is the agreed allowance for the insoluble component (listed in Annex II to the Directive on textile names)

a_2 is the agreed allowance for the soluble component (listed in Annex II to the Directive on textile names)

b_1 is the percentage loss of insoluble component caused by the pre-treatment

b_2 is the percentage loss of soluble component caused by the pre-treatment

The percentage of the soluble component (P_{2A}%) was obtained by the difference.

As specified in Directive 96/73/EC, correction factors b_1 and b_2 could be ignored as the normal pre-treatment by extraction with light petroleum ether and water was applied. The agreed allowances used in the calculations are reported in Table 2. The agreed allowance for polylactide proposed by the applicant, equal to 1.50, was used.

	Agreed allowance
Cotton	8.50
Wool	18.25
Polyester	1.50
Viscose	13.00
Acrylic	2.00
Nylon	6.25
Silk	11.00
Polylactide	1.50

Table 2: Agreed allowances used in the calculations.

3.3 Results

All the samples tested at the JRC were analysed using method 6 in Directive 96/73/EC. Twenty replicate specimens of binary mixture samples were analysed and

the homogeneity of the samples was verified. In addition, pure samples of wool, cotton, polyester etc. were processed for the purpose of completeness and, in this case, ten replicates were tested.

For the purpose of comparison, ten replicates of some relevant blends, in particular sample **012** (63.7% polylactide – 36.3% wool), sample **002** (50% polylactide – 50% cotton), sample **004** (50.2% polylactide – 49.8% polyester) and the corresponding pure fibres (wool, cotton, polyester) were analysed by Cargill Dow using their own method. The same blends were also tested by two independent laboratories (Central Laboratory of the Belgian Ministry of Economic Affairs and Italian National Council of Research, Institute for the Study of Macromolecules) using method 6, in the same way as the JRC, in order to verify the non-existence of a significant laboratory bias.

During the visit to Ispra, Cargill Dow and JRC decided that additional tests should be performed on pure polylactide yarns of different thickness to verify that the conditions of method 6 were stringent enough to dissolve even thick yarns of the new fibre.

The composition of the samples was generally indicated by the petitioner on the basis of the number of denier of the yarns used to knit samples. However, this measurement lacks precision and the stated compositions should not be regarded as a reference value. Furthermore for sample **002** (50% polylactide – 50% cotton) the indicated percentage is only approximate as this is not a mixture made of pure polylactide and cotton yarns, it is an intimate staple blend, i.e. staple fibres of pure polylactide and cotton were mixed together to produce one yarn. In this case the composition is not known exactly because of manufacturing processes and also the homogeneity of the sample may not be as good as the other samples.

The data were collected and subjected to statistical evaluation. The procedure followed guidelines ISO 5725 [3] and IUPAC harmonised protocol (1995) [4]. Firstly, a test was made to compare the work of two different operators. The tests were conducted at a 95% confidence level, in one case assuming a homogeneous variance, not in the other. The values were lower than t-critical, showing that there were no differences between operators. The results were also examined for evidence of individual systematic error using Dixon's test, as laid down in ISO 5725, in order to determine the presence of outliers. Six outliers were found out of more than four hundreds measurements and they were confirmed using some other test statistics [5], summarised in Table 3.

	Upper outlier	Lower outlier	Comments
"t-like" tests statistics	$T1 = \frac{x_n - \bar{x}}{s_x}$	$T1 = \frac{\bar{x} - x_1}{s_x}$	Test for single outlier, sometimes called T_n test
	$T2 = \frac{\sum (x_i - \bar{x})}{s_x}$	$T2 = \frac{\sum (\bar{x} - x_i)}{s_x}$	Block test for k upper or lower outliers
	$T3 = \frac{x_n - x_1}{s_x}$		Block test for one upper and one lower outlier
"Dixon-like" statistics	$T4 = \frac{x_n - x_{n-1}}{x_n - x_1}$	$T4 = \frac{x_2 - x_1}{x_n - x_1}$	Tests for a single outlier, sometimes called the Q test
	$T5 = \frac{x_n - x_{n-2}}{x_n - x_2}$	$T5 = \frac{x_3 - x_1}{x_{n-1} - x_1}$	Tests for a single outlier, a form of the Q-test that provides some protection from masking

Table 3: Test statistics used to confirm the presence of outliers.

The valid results were then subjected to statistical evaluation. The average and standard deviation (SD) of each set of data were calculated as well as the relative standard deviation (RSD). The RSD was used to measure the dispersion of the distribution of test results in one laboratory: the lower the value of RSD the better the repeatability of the method. The confidence intervals (uncertainties) were calculated at 95% and 98% of probability, using the following formula:

$$\mu = x_m \pm \frac{t\ s}{\sqrt{N}}$$

where:

t is the value listed in the Student's t-distribution for a certain number of degrees of freedom and level of probability

s is the estimated standard deviation

μ is the true value

x_m is the average of experimental values

N is the number of measurements.

The results of the analysis of the composition are reported in Annex III. All the measurements were performed at the JRC using method 6, except where otherwise specified. An overview of the relevant results, with uncertainties calculated for a confidence level of 95%, is reported in Tables 4-5.

The results showed that method 6 in Directive 96/73/EC led to a good repeatability, as proved by the low values of RSD and uncertainty. RSD was in the range 0.3 – 1.3

and uncertainty 0.1 – 0.3 for binary mixtures, depending on the composition. The repeatability was even better for samples of pure fibre: RSD was in the range 0.1 - 0.2 whereas uncertainty was lower or equal to 0.1. The results showed that the precision of method 6 for analysing binary mixtures containing polylactide is at least as good as for analysing binary blends containing triacetate with which it was validated. In fact, Directive 96/73/EC states that on a homogeneous mixture of textile materials containing triacetate, the confidence limits of results obtained by method 6 are not greater than ± 1 for a confidence level of 95% and the same is true in the case of polylactide.

The results of analyses are in agreement with the composition indicated by the applicant, considering that a manufacturing tolerance of 3% shall be permitted between the stated fibre percentages and the percentages obtained from analysis, in relation to the total weight of fibres shown on the label (as foreseen by article 6, comma 4b in Directive 96/74/EC on textile names).

The data obtained using method 6 at the JRC, at the Central Laboratory of the Belgian Ministry of Economic Affairs and at the Italian National Council of Research, Institute for the Study of Macromolecules, were generally concordant, with the only exception of sample **002**, thus confirming that no relevant systematic errors due to laboratory bias were present.

JRC code	Stated composition	Replicates	Polylactide %	RSD (other fibre)	RSD (polylactide)
030	71.2 polylactide - 28.8 polyester	19	71.3 ± 0.1	0.9	0.4
004 = 014	50.2 polylactide - 49.8 polyester	21	49.6 ± 0.1	0.6	0.7
029	38.1 polylactide - 61.9 polyester	22	38.1 ± 0.1	0.5	0.8
007 = 012	63.7 polylactide - 36.3 wool	21	62.2 ± 0.2	1.2	0.7
027	55.8 polylactide - 44.2 wool	20	53.1 ± 0.1	0.5	0.5
028	38.3 polylactide - 61.7 wool	20	37.2 ± 0.2	0.6	1.1
026	72.6 polylactide - 27.4 cotton	20	72.1 ± 0.1	0.7	0.3
017	56.6 polylactide - 43.4 cotton	26	55.9 ± 0.2	0.9	0.7
002 = 016	50 polylactide - 50 cotton	16	53.1 ± 0.2	0.9	0.8
025	40.2 polylactide - 59.8 cotton	22	38.9 ± 0.2	0.7	1.1
033	55.4 polylactide - 44.6 viscose	21	54.2 ± 0.3	1.3	1.1
035	55.6 polylactide - 44.4 acrylic	21	56.2 ± 0.3	1.3	1.0
037	55.8 polylactide - 44.2 nylon	21	55.4 ± 0.1	0.7	0.6
041	56.1 polylactide - 43.9 silk	20	56.1 ± 0.1	0.5	0.4

Table 4: Analysis of composition of binary mixtures performed by the JRC with method 6.

JRC code	Stated composition	Replicates	Fibre %	RSD
003 = 015	100 polyester	14	100.2 ± 0.1	0.2
006 = 013	100 wool	13	99.8 ± 0.1	0.1
005 = 011	100 cotton	12	99.84 ± 0.04	0.06
034	100 viscose	10	99.9 ± 0.1	0.1
036	100 acrylic	10	99.8 ± 0.1	0.1
038	100 nylon	9	99.85 ± 0.03	0.04
042	100 silk	10	99.6 ± 0.1	0.2
001 = 010 = 008	100 polylactide 268 denier	10	99.91 ± 0.02	0.04
009	100 polylactide 654 denier	10	99.93 ± 0.03	0.03

Table 5: Analysis of composition of pure fibres performed by the JRC with method 6.

The results obtained by Cargill Dow using their own method, reported in Annex III and summarised in Tables 6-7, showed a good RSD as well. However, the comparison of measurements performed on same samples shows that the absolute values obtained with the two methods differ slightly, most probably due to the fact that Cargill Dow's method is not based on dry mass calculations. In some cases, the method proposed by Cargill Dow led to incomplete dissolution of polylactide, which was solved by an additional step using methanol at room temperature.

JRC code	Stated composition	Replicates	Polylactide %	RSD (other fibre)	RSD (polylactide)
004 = 014	50.2 polylactide - 49.8 polyester	10	50.4 ± 0.2	0.7	0.7
007 = 012	63.7 polylactide - 36.3 wool	10	63.1 ± 0.3	1.2	0.7
002 = 016	50 polylactide - 50 cotton	10	53.7 ± 0.2	0.6	0.5

Table 6: Analysis of composition of binary mixtures performed by Cargill Dow with their own method.

JRC code	Stated composition	Replicates	Fibre %	RSD
003 = 015	100 polyester	10	99.7 ± 0.1	0.1
006 = 013	100 wool	10	101.3 ± 0.1	0.1
005 = 011	100 cotton	10	98.4 ± 0.2	0.3

Table 7: Analysis of composition of pure fibres performed by Cargill Dow with their own method.

4. Verification of the applicability of the method proposed by Cargill Dow for the identification of polylactide

The samples received from Cargill Dow were analysed by microscopy for a preliminary characterisation. An Olympus microscope model IX70 was used and the analysis was performed using transmitted light. Glycerol triacetate was used as contrast reagent.

The photos of the different pure fibres can be seen in Annex II (Figs. 7-14). A sample of pure polylactide at magnifying power 250 and 500 respectively is shown in Fig. 7. These pictures show that the new fibre cannot be unequivocally identified by microscopic analysis, as it can be confused with nylon, for example.

In contrast, the qualitative method proposed by the petitioner was successfully applied as an unambiguous way to identify polylactide (see analytical method 1 in Annex I). The method is based on Fourier transform infrared (FT-IR) spectroscopy and the identification is made by comparing the obtained transmission spectrum with the spectrum of a standard. Recognition of the fibre is easy due to the characteristic fingerprint of the molecule. A quality match of 75% or greater is required, as a criterion of judgement, to confirm the presence of polylactide in the sample.

Samples were cut into specimens of about 1 g, dissected as much as possible, and treated with 10 + 10 ml of dichloromethane to dissolve polylactide. One drop of dichloromethane extract was transferred to a clean KBr plate in order to evaporate the solvent and obtain a film. The transmission FT-IR spectrum was then acquired using a Perkin Elmer instrument (FT-IR spectrometer spectrum 2000), see Fig. 15 in Annex IV.

FT-IR spectra of all samples were also acquired using ATR Attenuated Total Reflectance mode. Samples were analysed as they were received, without any preparation. The spectra of pure fibres and some binary mixtures are reported in Annex IV.

The results suggested that FT-IR allows the unequivocal identification of pure fibres directly on knitted samples using the Attenuated Total Reflectance mode. In the case of binary mixtures, ATR spectra can only suggest the composition, so a procedure like the one proposed by Cargill Dow, with the preparation of a pure film, is necessary for identification purposes.

5. Experimental determination of the agreed allowance for polylactide

A number of experiments were performed on different samples of pure polylactide in order to experimentally evaluate the agreed allowance of this new fibre. Since a specific method for this type of measurement is not available, the following

procedure, which takes into account terms and definitions of UNI 9213 [6] and UNI 8048 [7], was applied.

Weighing bottles were dried for 1 hour in a ventilated oven at 105 ± 3 ºC, then cooled in a dessicator and weighed. One sample of about 2 g of polylactide (pre-treated or untreated, staple yarn or staple fibre) was placed in each weighing bottle and dried for 4 hours in a ventilated oven at 105 ± 3 ºC, then cooled in a dessicator and weighed. The samples were then conditioned for 72 hours at 20 ± 2 ºC and 65 ± 2 % relative humidity and weighed immediately after the conditioning period. The following formulas were used to calculate the agreed allowance.

Water mass = wet sample mass – dried sample mass

Agreed allowance = (water mass / dried sample mass) * 100

Four replicate specimens were analysed for each sample. Sample **009**, both pre-treated and untreated, was also analysed by the laboratory of the Italian National Council of Research to verify the non-existence of a relevant laboratory bias.

Results, reported in Annex V, showed that the experimental agreed allowance ranged from 0.22 to 0.32 depending of the type of sample (e.g. yarn versus staple fibre). These results were discussed by the experts from enforcement laboratories representing Member States, during the meeting organised by the JRC in Ispra on 3th July 2003, in order to reach a consensus on the value of agreed allowance to be proposed for the amendment of Directive 96/74/EC. Even if the experimental value obtained for the humidity regain of polylactide is far from the value of 1.50 proposed by the applicant, it was accepted because it is in line with the values of agreed allowances listed in the Directive that are all higher than the experimental ones. For example, the established value for polyester is 1.50, even if the experimental value of humidity regain for this fibre does not usually exceed 0.40. In addition, for the purpose of the legislation, the agreed allowance is just a conventional factor, only used in calculations to take into account the humidity regain of the fibre, and does not have any other implication.

6. Conclusions

Test results confirm that method 6 in Directive 96/73/EC is suitable for the quantification of polylactide in relevant binary mixtures. The applicability of this method was verified for binary mixtures of polylactide with polyester, wool, cotton, viscose, acrylic, nylon and silk. The results showed that the method led to a good repeatability, calculated as relative standard deviations, both for binary mixtures and for pure fibres. As method 6 has already been validated at European level, it may become the official method to quantify polylactide in binary mixtures with other fibres.

Concerning the identification method, tests performed at the JRC confirmed that the method proposed by the petitioner is suitable for the identification of polylactide.

The experimental value of the agreed allowance for polylactide ranged from 0.22 to 0.32 depending on the type of sample analysed. However, after consultation with experts from Member States' enforcement laboratories, it was agreed that the value of agreed allowance proposed by the applicant (1.50) could be accepted because it is in line with the other values listed in Directive 96/74/EC.

7. References

[1] Directive 96/74/EC of the European Parliament and of the Council of 16 December 1996 on textile names *(Official Journal L032 of 3.2.1997 p. 0038-0055),* http://europa.eu.int/comm/enterprise/textile/intlmarket.htm.

[2] Directive 96/73/EC of the European Parliament and of the Council of 16 December 1996 on certain methods for the quantitative analysis of binary textile fibre mixtures *(Official Journal L032 of 3.2.1997 p. 0001-0037),* http://europa.eu.int/comm/enterprise/textile/intlmarket.htm.

[3] ISO 5725 (1994) Accuracy (trueness and precision) of measurement methods and results, Part 1-6.

[4] Horwitz, W. (1995) IUPAC: Protocol for the design, conduct and interpretation of method performance studies, *Pure & Applied Chem.*, **67**, 331-343.

[5] Stevenson, C.L. The statistic of measurements, University of Richmond, Chemistry 300 (2000)

[6] UNI 9213 (1989) Determination of commercial mass.

[7] UNI 8048 (1980) Determination of dry content for chemical and/or physical measurements.

Annex I

Analytical methods proposed by the applicant

ANALYTICAL METHOD 1

Used for Identification of PLA[2] Fiber

1. PRINCIPLE

This method uses Fourier transform infrared (FT-IR) spectroscopy to qualitatively assess the presence of polylactide in cotton, wool, and rayon. The natural fiber is dissolved in dichloromethane. The PLA fraction dissolves in dichloromethane leaving the natural fiber as solid in solution. An aliquot of the dichloromethane extract is transferred to a KBr plate. The dichloromethane is evaporated and the remaining film is analyzed by FT-IR. The transmission spectrum of the film is compared to the transmission spectrum of PLA standard. A spectral quality match of 75% or greater is a positive indication for the presence of PLA in the natural fiber.

2. SCOPE

This method can be used to qualitatively assess the presence of PLA in blends with cotton, wool, and rayon.

3. EQUIPMENT AND REAGENTS

3.1. Instrumentation/Equipment

3.1.1 Fourier Transform Infrared Spectrophotometer, Spectrum One, PerkinElmer, or equivalent.

3.1.2 KBr plate, PerkinElmer, Catalog #L1900034, or equivalent.

3.1.3 Analytical balance: Mettler AE-20, or equivalent.

3.1.4 Eppendorf repeater pipettor: VWR, Catalog #53512-500, or equivalent.

3.1.5 Orbital Shaker: Lab-Line, VWR, Catalog #57018-853, or equivalent.

3.1.6 Mini-Vap Evaporator: VWR, Catalog #21506-184, or equivalent.

3.1.7 Disposible pipets, VWR, Catalog #14670-103, or equivalent.

3.2. Reagents/Materials

3.2.1. Dichloromethane: OmniSolve, EM Science, VWR, Catalog #EM-DX0837-1, or equivalent.

3.2.2. 20 ml scintillation vials with foil lined caps: VWR, Catalog #66022-004, or equivalent.

4. SAFETY NOTES

4.1. Dichloromethane Health (4), Flammability (1), Reactivity (1)

5. PROCEDURE

5.1. Preparation of samples

5.1.1. Accurately add 1.0 +/- 0.1 grams of the PLA blended fiber to a 20 ml scintillation vial and record the exact weight

5.1.2. Accurately add 10.0 ml of dichloromethane to the first 20 ml scintillation vial. Shake the 20 ml scintillation vial for 1.0 hour.

5.1.3. Decant the dichloromethane into a second 20 ml scintillation vial.

5.1.4. Accurately add another 10.0 ml of dichloromethane to the first 20 ml scintillation vial.

5.1.5. Shake the first 20 ml scintillation vial for 30 minutes.

5.1.6. Decant the dichloromethane and combine with the first dichloromethane extract.

5.1.7. Transfer one drop of the dichloromethane extract to a clean KBr plate.

5.1.8. Evaporate the remaining dichloromethane from the KBr plate.

5.1.9. Analyze the KBr plate by FT-IR

5.1.10. Compare the transmission spectrum of the dichloromethane extract to the transmission spectrum of PLA. A quality match of 75% or greater is a positive indication for the presence of PLA in the blended natural fiber.

5.2. Instrument Conditions

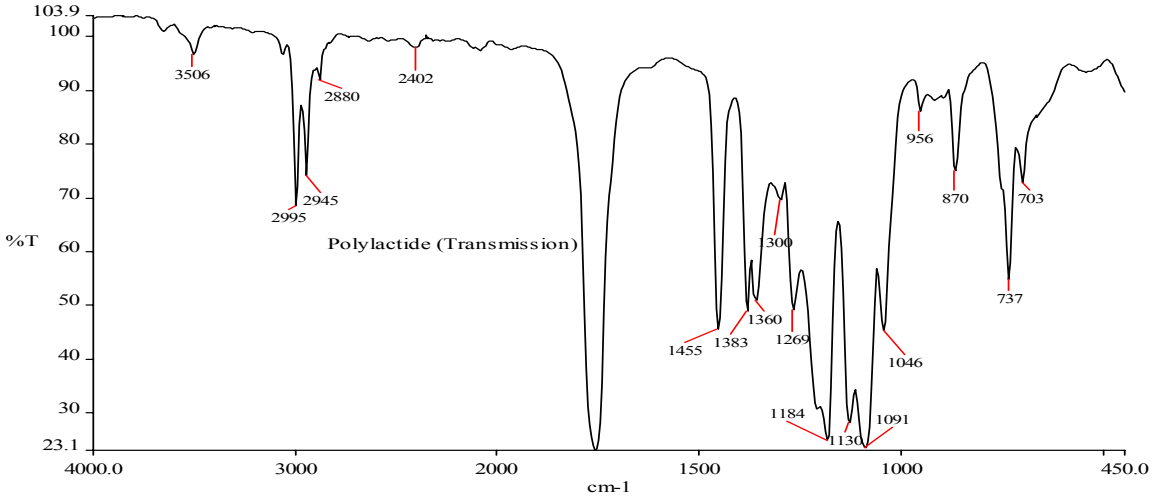
5.2.1. Resolution: 4 cm^{-1}

5.2.2. Number Scans: 4

5.2.3. Scan Range: 350 to 4,000 cm^{-1}

[2] Abbreviation used in Cargill Dow's documentation for polylactide.

Figure 1
PLA FT-IR transmission spectrum



ANALYTICAL METHOD 2

Calculates % PLA in Binary Blends with Natural Fibres such as cotton and wool and for use as second extraction in Ternary Mixtures

1. PRINCIPLE

This method can be used to quantify the concentration of polylactide (PLA) in blends with natural fibers. Solubility difference between PLA and natural fibers in dichloromethane is the basis for this method. An aliquot of dichloromethane is added to a known mass of PLA blend. The PLA fraction of the PLA blend is soluble in methylene chloride while the natural fibers are not. The concentration of PLA is quantitatively measured by comparing the mass of the fraction that did not dissolve in methylene chloride to the mass of the PLA blend sample.

2. SCOPE

This method can be used to quantify the concentration of PLA in blends with natural fibers. This method will not work if the natural fiber is soluble in dichloromethane. The method has not been validated, so the values generated from this method should be considered as approximate and not absolute.

3. EQUIPMENT AND REAGENTS

3.1. Instrumentation/Equipment

3.1.1 Analytical balance: Mettler AE-20, or equivalent.

3.1.2 Eppendorf repeater pipettor: VWR, Catalog #53512-500, or equivalent.

3.1.3 Orbital Shaker: Lab-Line, VWR, Catalog #57018-853, or equivalent.

3.1.4 Mini-Vap Evaporator: VWR, Catalog #21506-184, or equivalent.

3.2. Reagents/Materials

3.2.1. Dichloromethane: OmniSolve, EM Science, VWR, Catalog #EM-DX0837-1, or equivalent.

3.2.2. 20 ml scintillation vials with foil lined caps: VWR, Catalog #66022-004, or equivalent.

4. SAFETY NOTES

4.1.Dichloromethane Health (4), Flammability (1), Reactivity (1)

5. PROCEDURE

5.1. Preparation of samples

5.1.1 Accurately weigh a clean 20 ml scintillation vial. Record the weight.

5.1.2 Accurately add 1.0 +/- 0.1 grams of PLA blend sample to the above 20 ml scintillation vial and record the exact weight.

5.1.3 Accurately add 10.0 ml of dichloromethane to the first 20 ml scintillation vial.

5.1.4 Shake the 20 ml scintillation vial for 1.0 hour.

5.1.5 Decant the dichloromethane.

5.1.6 Transfer the 10 ml dichloromethane extract to a second 20 ml scintillation vial.

5.1.7 Accurately add another 10.0 ml of dichloromethane to the first 20 ml scintillation vial.

5.1.8 Shake the first 20 ml scintillation vial for 30 minutes.

5.1.9 Decant the dichloromethane.

5.1.10 Combine the second dichloromethane extract with the first dichloromethane extract. There should be approximately 20 ml.

5.1.11 Sparge nitrogen, to evaporate remaining dichloromethane, over the solid precipitate in the first 20 ml vial for 15 minutes.

5.1.12 Record the exact weight of the 20 ml scintillation vial with the solid precipitate.

5.1.13 Calculate the weight of the precipitate using equation 1.

5.1.14 Calculate % PLA using equation 2.

5.2. Calculations

5.2.1 Precipitate weight (grams) = ((Weight of Precipitate +20 ml Vial (gram)) – (Weight of 20 ml Vial (gram))

5.2.2. % PLA = $\left\{ \frac{\left(\left(PLA\ Blend\ (grams) \right) - \left(\mathrm{Pr}\ ecipitate\ (grams) \right) \right)}{PLA\ Blend\ (grams)} \right\} X\ 100$

Annex II

Microscopic analysis

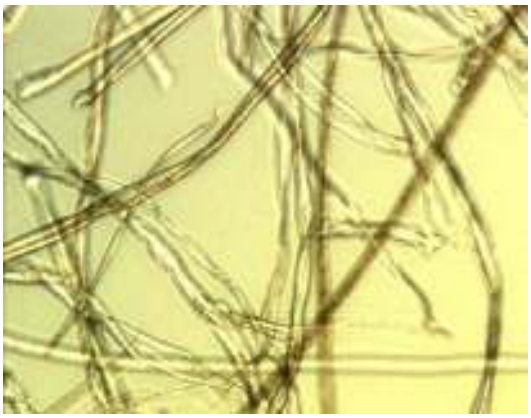


Fig. 1: 40.2% polylactide – 59.8% cotton (sample **025**). 250X.

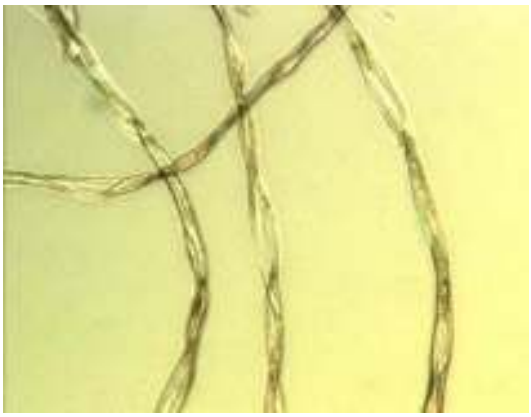


Fig. 2: Residue of cotton after dissolution of polylactide (sample **016**). 250X.

Fig. 3: Residue of cotton after dissolution of polylactide (sample **017**). 250X.

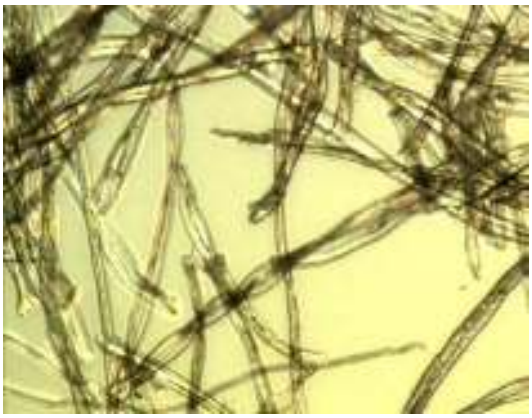


Fig. 4: Residue of cotton after dissolution of polylactide (sample **025**). 250X.

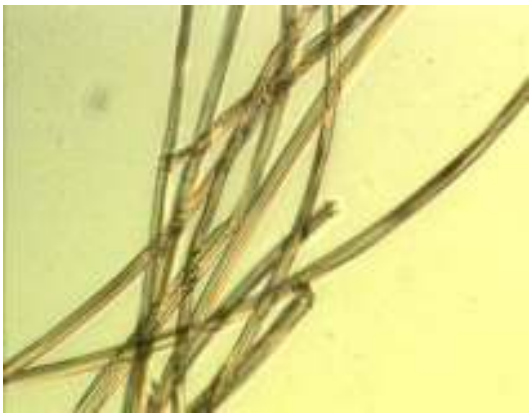


Fig. 5: Residue of viscose after dissolution of polylactide (sample **033**). 250X.

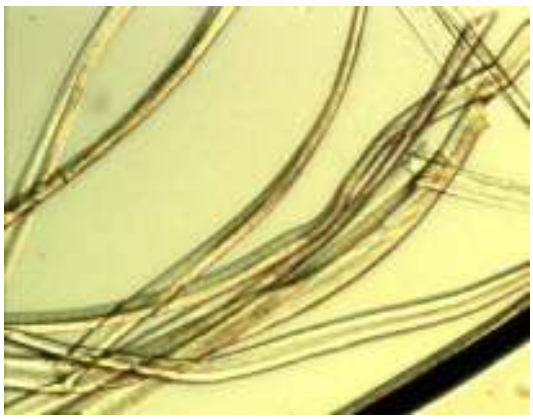


Fig. 6: Residue of acrylic after dissolution of polylactide (sample **035**). 250X.

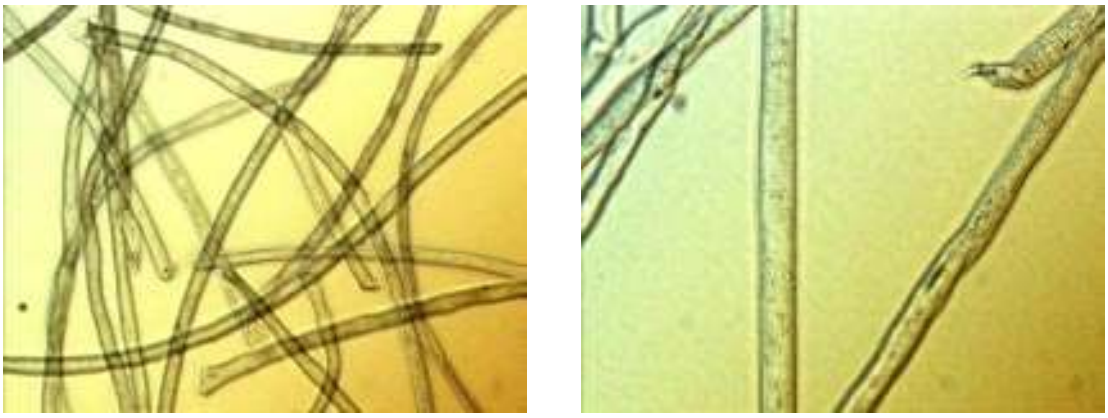


Fig. 7: 100% polylactide (Sample **010**). 250X and 500X respectively.

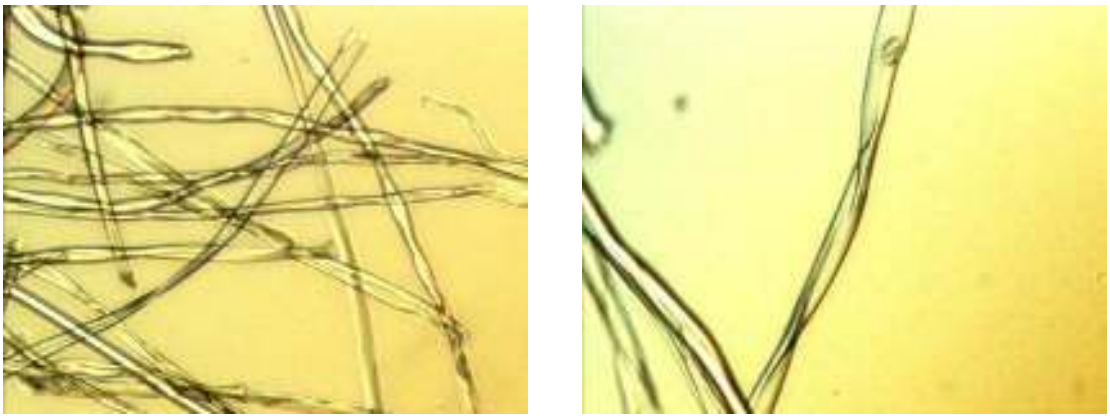


Fig. 8: 100% cotton (sample **005**). 250X and 500X respectively.

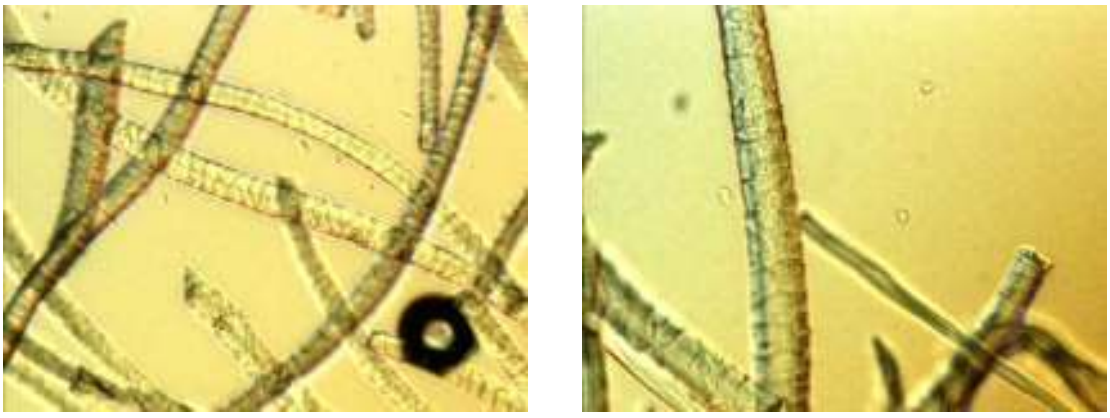


Fig. 9: 100% wool (sample **006**). 250X and 500X respectively.

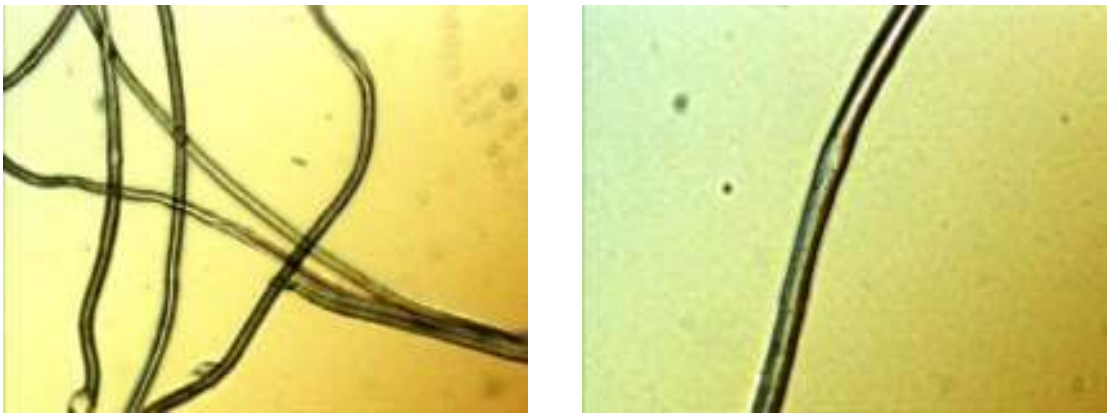


Fig. 10: 100% polyester (sample **015**). 250X and 500X respectively.

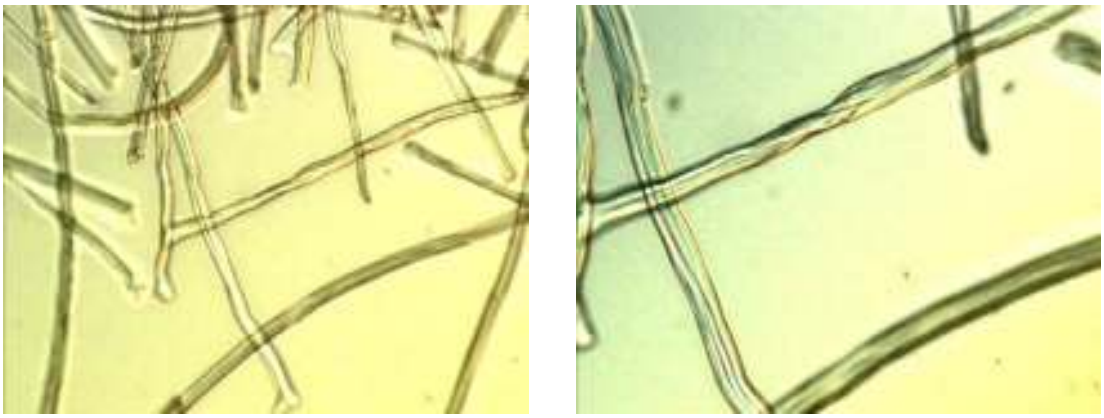


Fig. 11: 100% viscose (sample **034**). 250X and 500X respectively.

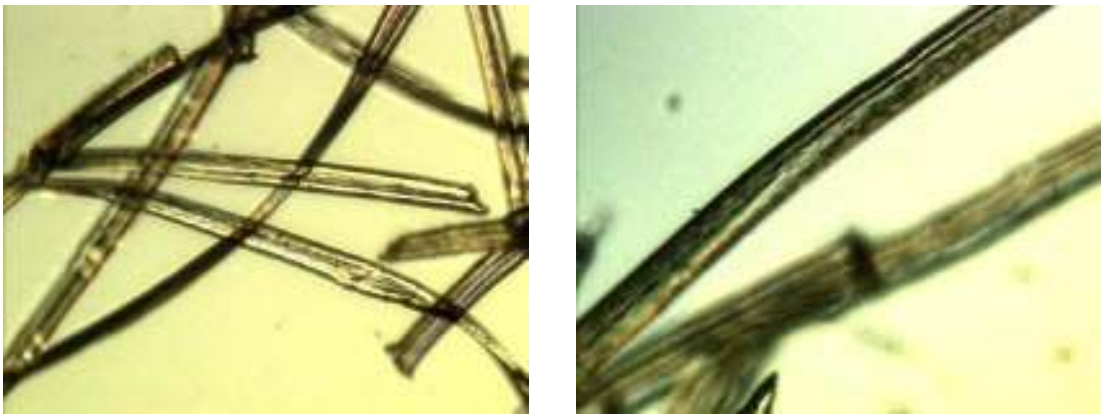


Fig. 12: 100% acrylic (sample **036**). 250X and 500X respectively.

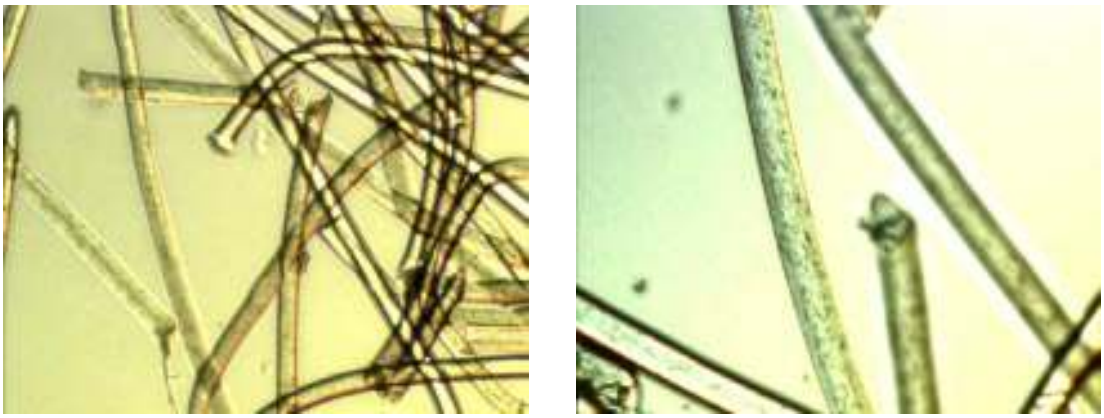


Fig. 13: 100% nylon (sample **038**). 250X and 500X respectively.

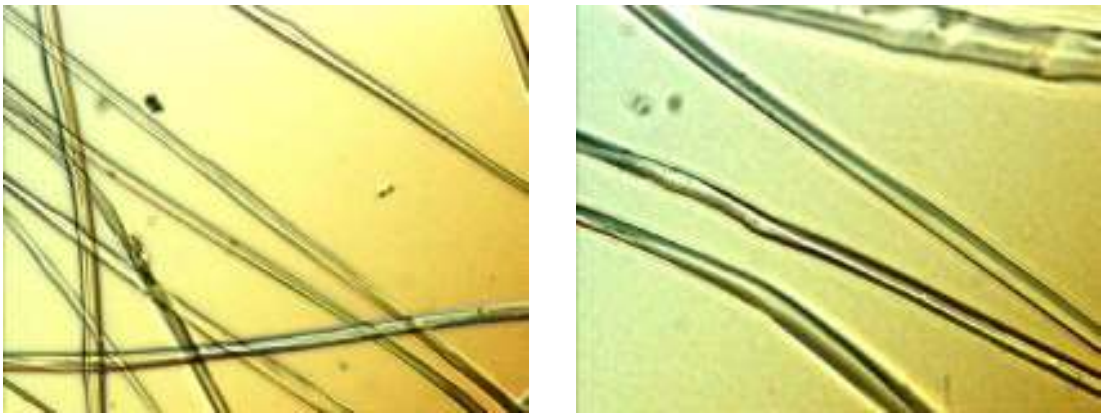


Fig. 14: 100% silk (sample **042**). 250X and 500X respectively.

Annex III

Analysis of composition

71.2 % polylactide – 28.8 % polyester (sample 030)

JRC code	sample mass	polyester mass	polyester	polylactide
	g	g	%	%
030A	1.0684	0.3030	28.6	71.4
030C	1.0029	0.2808	28.3	71.7
030E	1.2462	0.3559	28.8	71.2
030F	1.1452	0.3249	28.7	71.3
030G	1.3919	0.3950	28.7	71.3
030H	1.2219	0.3475	28.7	71.3
030I	1.1708	0.3351	28.9	71.1
030L	1.1352	0.3210	28.6	71.4
030M	1.4163	0.3926	28.0	72.0
030N	1.3625	0.3867	28.7	71.3
030O	1.3721	0.3893	28.7	71.3
030P	1.3486	0.3872	29.0	71.0
030Q	1.5972	0.4534	28.7	71.3
030R	1.6250	0.4576	28.4	71.6
030S	0.9905	0.2812	28.7	71.3
030T	0.9288	0.2673	29.1	70.9
030U	0.7638	0.2187	28.9	71.1
030V	0.9580	0.2749	29.0	71.0
030Z	0.8797	0.2502	28.7	71.3
average			28.7	71.3
SD			0.3	0.3
RSD			0.9	0.4

n-1=18	t (0.05)	t (0.02)
	2.086	2.528
average	71.3	71.3
uncertainty	0.1	0.1

JRC code	sample mass	polyester mass	polyester	polylactide
	g	g	%	%
030-1	1.3501	0.3882	28.8	71.2
030-2	1.0738	0.3064	28.5	71.5
030-3	1.1961	0.3395	28.4	71.6
average			28.6	71.4
SD			0.2	0.2
RSD			0.7	0.3

n-1=2	t (0.05)	t (0.02)
	4.303	6.964
average	71.4	71.4
uncertainty	0.5	0.7

Results obtained by Cargill Dow using their own method.

50.2 % polylactide – 49.8 % polyester (samples 004-014)

JRC code	sample mass	polyester mass	polyester	polylactide
	g	g	%	%
004A	1.9518	0.9750	50.5	49.5
004B	2.3774	1.1908	50.6	49.4
004C	1.7899	0.8976	50.7	49.3
004D	1.9866	0.9950	50.6	49.4
004E	0.8387	0.4190	50.5	49.5
004F	1.4346	0.7206	50.7	49.3
004G	1.2374	0.6264	51.1	48.9
004H	1.1925	0.5993	50.8	49.2
004I	1.0712	0.5386	50.8	49.2
004L	1.2322	0.6204	50.9	49.1
004M	1.2034	0.6021	50.5	49.5
014A	1.0943	0.5429	50.1	49.9
014B	1.0385	0.5142	50.0	50.0
014C	0.9965	0.4955	50.2	49.8
014D	1.1746	0.5852	50.3	49.7
014E	1.2005	0.5926	49.9	50.1
014F	1.0549	0.5246	50.2	49.8
014G	0.9727	0.4814	50.0	50.0
014H	0.9478	0.4718	50.3	49.7
014I	1.1235	0.5596	50.3	49.7
014L	1.1271	0.5601	50.2	49.8
average			**50.4**	**49.6**
SD			**0.3**	**0.3**
RSD			**0.6**	**0.7**

n-1=20	t (0.05)	t (0.02)
	2.086	2.528
average	**49.6**	**49.6**
uncertainty	**0.1**	**0.2**

JRC code	polyester	polylactide
	%	%
004A	50.3	49.7
004B	50.9	49.1
average	**50.6**	**49.4**

Results obtained by the Central Laboratory of Belgian Ministry of Economic Affairs using method 6.

JRC code	sample mass	polyester mass	polyester	polylactide
	g	g	%	%
014-1	0.9952	0.4924	49.5	50.5
014-2	1.0156	0.5018	49.4	50.6
014-3	0.9996	0.4946	49.5	50.5
014-4	1.0094	0.4985	49.4	50.6
014-5	1.0196	0.5028	49.3	50.7
014-6	1.0245	0.5068	49.5	50.5
014-7	1.0035	0.496	49.4	50.6
014-8	1.0199	0.508	49.8	50.2
014-9	1.0105	0.5093	50.4	49.6
014-10	1.0053	0.4956	49.3	50.7
average			**49.5**	**50.5**
SD			**0.3**	**0.3**
RSD			**0.7**	**0.7**

n-1=9	t (0.05)	t (0.02)
	2.262	2.821
average	**50.5**	**50.5**
uncertainty	**0.2**	**0.3**

Results obtained by Cargill Dow using their own method.

38.1 % polylactide – 61.9 % polyester (sample 029)

JRC code	sample mass	polyester mass	polyester	polylactide
	g	g	%	%
029A	1.1836	0.7218	61.6	38.4
029B	1.1314	0.7024	62.7	37.3
029C	1.5435	0.9360	61.2	38.8
029D	1.6038	0.9815	61.8	38.2
029E	1.0100	0.6202	62.0	38.0
029F	1.1121	0.6822	62.0	38.0
029G	0.8926	0.5503	62.3	37.7
029H	1.1818	0.7255	62.0	38.0
029I	1.0423	0.6375	61.8	38.2
029L	1.1692	0.7158	61.8	38.2
029M	1.3675	0.8367	61.8	38.2
029N	1.3270	0.8137	61.9	38.1
029O	1.2445	0.7619	61.8	38.2
029P	1.3209	0.8162	62.4	37.6
029Q	1.4841	0.9112	62.0	38.0
029R	1.2285	0.7533	61.9	38.1
029S	1.0795	0.6604	61.8	38.2
029T	1.3897	0.8508	61.8	38.2
029U	1.3008	0.7956	61.8	38.2
029V	1.0608	0.6480	61.7	38.3
029Z	1.0633	0.6496	61.7	38.3
029J	1.1538	0.7065	61.8	38.2
average			61.9	38.1
SD			0.3	0.3
RSD			0.5	0.8

n-1=21	t (0.05)	t (0.02)
	2.080	2.518
average	38.1	38.1
uncertainty	0.1	0.2

JRC code	sample mass	polyester mass	polyester	polylactide
	g	g	%	%
029-1	1.2455	0.7819	62.8	37.2
029-2	1.2994	0.8159	62.8	37.2
029-3	1.1978	0.7716	64.4	35.6
average			63.3	36.7
SD			0.9	0.9
RSD			1.5	2.6

n-1=2	t (0.05)	t (0.02)
	4.303	6.964
average	36.7	36.7
uncertainty	2.3	3.8

Results obtained by Cargill Dow using their own method.

63.7 % polylactide – 36.3 % wool (samples 007-012)

JRC code	sample mass	wool mass	wool	polylactide
	g	g	%	%
007A	1.0302	0.3577	38.3	61.7
007B	1.1873	0.4032	37.5	62.5
007C	1.1473	0.3880	37.3	62.7
007D	1.0460	0.3580	37.7	62.3
007E	1.2561	0.4304	37.8	62.2
007F	0.9814	0.3322	37.3	62.7
012A	1.1278	0.3916	38.3	61.7
012B	1.2572	0.4318	37.9	62.1
012C	1.1953	0.4130	38.1	61.9
012D	1.4315	0.4808	37.1	62.9
012E	0.9606	0.3341	38.3	61.7
012F	0.9034	0.3150	38.4	61.6
012G	0.9198	0.3190	38.2	61.8
012H	0.8940	0.3096	38.2	61.8
012I	1.0458	0.3632	38.3	61.7
012L	1.0446	0.3565	37.6	62.4
012N	2.2415	0.7608	37.4	62.6
012O	1.6882	0.5723	37.4	62.6
012P	2.3438	0.7869	37.1	62.9
012Q	1.7144	0.5779	37.2	62.8
012R	1.5089	0.5240	38.3	61.7
average			**37.8**	**62.2**
SD			**0.5**	**0.5**
RSD			**1.2**	**0.7**

n-1=20	t (0.05)	t (0.02)
	2.086	2.528
average	**62.2**	**62.2**
uncertainty	**0.2**	**0.3**

JRC code	wool	polylactide
	%	%
007A	38.0	62.3
007B	37.7	62.0
average	**37.9**	**62.2**

Results obtained by the Central Laboratory of Belgian Ministry of Economic Affairs using method 6.

JRC code	sample mass	wool mass	wool	polylactide
	g	g	%	%
012-1	0.9758	0.3538	36.3	63.7
012-2	1.0039	0.3691	36.8	63.2
012-3	0.9938	0.3662	36.8	63.2
012-4	1.0072	0.3692	36.7	63.3
012-5	0.9996	0.3645	36.5	63.5
012-6	1.0229	0.3720	36.4	63.6
012-7	0.9809	0.3647	37.2	62.8
012-8	0.9781	0.3626	37.1	62.9
012-9	1.0247	0.3850	37.6	62.4
012-10	1.0233	0.3830	37.4	62.6
average			**36.9**	**63.1**
SD			**0.4**	**0.4**
RSD			**1.2**	**0.7**

n-1=9	t (0.05)	t (0.02)
	2.262	2.821
average	**63.1**	**63.1**
uncertainty	**0.3**	**0.4**

Results obtained by Cargill Dow using their own method.

55.8 % polylactide – 44.2 % wool (sample 027)

JRC code	sample mass	wool mass	wool	polylactide
	g	g	%	%
027A	1.0228	0.4401	46.8	53.2
027B	1.1709	0.5067	47.1	52.9
027C	1.2291	0.5315	47.0	53.0
027D	1.4030	0.6063	47.0	53.0
027E	0.9848	0.4248	46.9	53.1
027F	0.8851	0.3833	47.1	52.9
027G	0.9362	0.4026	46.8	53.2
027H	0.7698	0.3318	46.9	53.1
027I	0.9080	0.3945	47.2	52.8
027L	1.2036	0.5153	46.6	53.4
027M	1.2248	0.5340	47.4	52.6
027N	0.9873	0.4272	47.1	52.9
027O	0.8528	0.3669	46.8	53.2
027P	1.1290	0.4845	46.7	53.3
027Q	0.9546	0.4110	46.8	53.2
027R	1.0315	0.4398	46.4	53.6
027S	0.9235	0.3951	46.6	53.4
027T	0.8520	0.3703	47.2	52.8
027U	1.1986	0.5144	46.7	53.3
027V	0.7166	0.3077	46.7	53.3
average			46.9	53.1
SD			0.2	0.2
RSD			0.5	0.5

n-1=19	t (0.05)	t (0.02)
	2.093	2.539
average	53.1	53.1
uncertainty	0.1	0.1

JRC code	sample mass	wool mass	wool	polylactide
	g	g	%	%
027-1	1.0704	0.4685	43.8	56.2
027-2	0.8590	0.3723	43.3	56.7
027-3	0.8031	0.3474	43.3	56.7
average			43.5	56.5
SD			0.3	0.3
RSD			0.6	0.5

n-1=2	t (0.05)	t (0.02)
	4.303	6.964
average	56.5	56.5
uncertainty	0.7	1.1

Results obtained by Cargill Dow using their own method.

38.3 % polylactide – 61.7 % wool (sample 028)

JRC code	sample mass	wool mass	wool	polylactide
	g	g	%	%
028A	1.1211	0.6529	61.9	38.1
028B	1.2193	0.7233	62.9	37.1
028C	1.1270	0.6719	63.2	36.8
028D	1.2461	0.7491	63.7	36.3
028E	1.1237	0.6638	62.7	37.3
028F	1.0516	0.6200	62.6	37.4
028G	1.1223	0.6667	63.0	37.0
028H	1.0239	0.6015	62.4	37.6
028I	1.0186	0.6021	62.7	37.3
028L	1.2206	0.7190	62.5	37.5
028N	2.1271	1.2556	62.7	37.3
028O	1.5677	0.9362	63.3	36.7
028P	1.6589	0.9830	62.9	37.1
028Q	2.2360	1.3100	62.2	37.8
028R	1.6020	0.9453	62.6	37.4
028S	2.1836	1.2919	62.8	37.2
028T	1.6171	0.9557	62.7	37.3
028U	2.4624	1.4435	62.3	37.7
028V	1.7481	1.0380	63.0	37.0
028Z	1.7837	1.0567	62.9	37.1
average			62.8	37.2
SD			0.4	0.4
RSD			0.6	1.1

n-1=19	t (0.05)	t (0.02)
	2.093	2.539
average	37.2	37.2
uncertainty	0.2	0.2

JRC code	sample mass	wool mass	wool	polylactide
	g	g	%	%
028-1	1.2162	0.7309	60.1	39.9
028-2	1.0741	0.6470	60.2	39.8
028-3	1.1826	0.7119	60.2	39.8
average			60.2	39.8
SD			0.1	0.1
RSD			0.1	0.2

n-1=2	t (0.05)	t (0.02)
	4.303	6.964
average	39.8	39.8
uncertainty	0.2	0.3

Results obtained by Cargill Dow using their own method.

72.6 % polylactide – 27.4 % cotton (sample 026)

JRC code	sample mass	cotton mass	cotton	polylactide
	g	g	%	%
026A	1.5921	0.4195	27.7	72.3
026B	1.2937	0.3399	27.6	72.4
026C	1.5417	0.4103	27.9	72.1
026D	2.0675	0.5448	27.7	72.3
026E	0.8382	0.2265	28.4	71.6
026F	0.9172	0.2437	27.9	72.1
026G	0.7987	0.2144	28.2	71.8
026H	1.0387	0.2755	27.8	72.2
026I	1.1240	0.2963	27.7	72.3
026L	1.1678	0.3100	27.9	72.1
026M	1.2921	0.3427	27.8	72.2
026N	1.2476	0.3332	28.0	72.0
026O	0.8923	0.2364	27.8	72.2
026P	0.8753	0.2315	27.8	72.2
026Q	0.9997	0.2662	28.0	72.0
026R	1.1159	0.2964	27.9	72.1
026S	0.8727	0.2318	27.9	72.1
026T	0.9589	0.2580	28.2	71.8
026U	1.3735	0.3652	27.9	72.1
026V	1.2681	0.3384	28.0	72.0
average			27.9	72.1
SD			0.2	0.2
RSD			0.7	0.3

n-1=19	t (0.05)	t (0.02)
	2.093	2.539
average	72.1	72.1
uncertainty	0.1	0.1

JRC code	sample mass	cotton mass	cotton	polylactide
	g	g	%	%
026-1	1.0961	0.2940	26.8	73.2
026-2	1.2389	0.3350	27.0	73.0
026-3	1.1707	0.3155	26.9	73.1
average			26.9	73.1
SD			0.1	0.1
RSD			0.4	0.1

n-1=2	t (0.05)	t (0.02)
	4.303	6.964
average	73.1	73.1
uncertainty	0.3	0.4

Results obtained by Cargill Dow using their own method.

56.5 % polylactide – 43.5 % cotton (sample 017)

JRC code	sample mass	cotton mass	cotton	polylactide
	g	g	%	%
017A	0.8882	0.3763	44.0	56.0
017B	0.8824	0.3914	44.0	56.0
017C	1.5084	0.6418	44.1	55.9
017D	0.7833	0.3328	44.1	55.9
017E	1.2248	0.5289	43.8	56.2
017F	1.3054	0.5610	43.6	56.4
017G	0.9765	0.4198	43.6	56.4
017H	1.2365	0.5310	44.2	55.8
017I	1.2314	0.5192	43.8	56.2
017L	1.2168	0.5315	44.0	56.0
017M	0.8932	0.3686	42.9	57.1
017N	1.0503	0.4424	43.8	56.2
017O	0.9426	0.4013	44.2	55.8
017P	0.9931	0.4185	43.8	56.2
017Q	1.1098	0.4704	44.0	56.0
017R	1.1549	0.4959	44.6	55.4
017S	1.4004	0.5998	44.5	55.5
017T	1.7878	0.7646	44.4	55.6
017U	1.5165	0.6527	44.7	55.3
017V	1.2698	0.5464	44.7	55.3
017Z	1.3786	0.5815	43.8	56.2
017J	1.5786	0.6647	43.7	56.3
017K	1.3614	0.5798	44.2	55.8
017Y	1.3672	0.5875	44.6	55.4
017W	1.4200	0.6066	44.4	55.6
017Q	1.3140	0.5548	43.9	56.1
average			**44.1**	**55.9**
SD			**0.4**	**0.4**
RSD			**0.9**	**0.7**

n-1=25	t (0.05)	t (0.02)
	2.060	2.485
average	**55.9**	**55.9**
uncertainty	**0.2**	**0.2**

50 % polylactide – 50 % cotton (samples 002-016)

(intimate staple blend, nominal percentage)

JRC code	sample mass	cotton mass	cotton	polylactide
	g	g	%	%
002A	1.1897	0.5323	46.4	53.6
002B	1.1160	0.4985	46.3	53.7
002C	0.8765	0.3886	46.0	54.0
002D	0.9425	0.4207	46.3	53.7
002E	1.0868	0.4897	46.7	53.3
002F	0.8881	0.4011	46.8	53.2
016A	0.9463	0.4289	47.0	53.0
016B	0.6973	0.3179	47.2	52.8
016C	1.0343	0.4712	47.2	52.8
016D	1.0860	0.4949	47.2	52.8
016E	1.0456	0.4754	47.1	52.9
016F	0.9932	0.4527	47.2	52.8
016G	1.0398	0.4721	47.1	52.9
016H	0.9358	0.4258	47.2	52.8
016I	0.8779	0.3991	47.1	52.9
016L	1.0692	0.4849	47.0	53.0
average			**46.9**	**53.1**
SD			**0.4**	**0.4**
RSD			**0.9**	**0.8**

n-1=15	t (0.05)	t (0.02)
	2.131	2.602
average	**53.1**	**53.1**
uncertainty	**0.2**	**0.3**

JRC code	cotton	polylactide
	%	%
002A	45.6	54.4
002B	45.9	54.1
average	**45.8**	**54.3**

Results obtained by the Italian National Council of Research, Institute for the Study of Macromolecules using method 6.

JRC code	cotton	polylactide
	%	%
002A	49.2	50.8
002B	49.0	51.0
average	49.1	50.9

Results obtained by the Central Laboratory of Belgian Ministry of Economic Affairs using method 6.

JRC code	sample mass	cotton mass	cotton	polylactide
	g	g	%	%
016-1	1.0037	0.4689	46.7	53.3
016-2	0.999	0.4604	46.1	53.9
016-3	1.0096	0.4689	46.4	53.6
016-4	0.9973	0.4588	46.0	54.0
016-5	1.0102	0.4639	45.9	54.1
016-6	0.9982	0.4651	46.6	53.4
016-7	1.0269	0.4777	46.5	53.5
016-8	1.0132	0.4718	46.6	53.4
016-9	1.012	0.468	46.2	53.8
016-10	0.9962	0.4593	46.1	53.9
average			46.3	53.7
SD			0.3	0.3
RSD			0.6	0.5

n-1=9	t (0.05)	t (0.02)
	2.262	2.821
average	53.7	53.7
uncertainty	0.2	0.3

Results obtained by Cargill Dow using their own method.

40.2 % polylactide – 59.8 % cotton (sample 025)

JRC code	sample mass	cotton mass	cotton	polylactide
	g	g	%	%
025A	1.2736	0.7642	61.6	38.4
025B	1.0770	0.6448	61.5	38.5
025C	1.3892	0.8363	61.8	38.2
025D	1.5371	0.9169	61.2	38.8
025E	0.9210	0.5455	60.8	39.2
025F	0.9521	0.5720	61.7	38.3
025G	1.4971	0.8783	60.3	39.7
025H	2.0176	1.1961	60.9	39.1
025I	1.5471	0.9199	61.1	38.9
025L	1.3666	0.8161	61.3	38.7
025M	1.8539	1.1107	61.5	38.5
025N	1.4406	0.8598	61.3	38.7
025O	2.2861	1.3510	60.7	39.3
025P	1.4604	0.8774	61.7	38.3
025Q	2.2180	1.3090	60.6	39.4
025R	1.8280	1.0978	61.6	38.4
025S	1.2466	0.7384	60.8	39.2
025T	1.3573	0.8024	60.7	39.3
025U	1.0582	0.6273	60.9	39.1
025V	1.1991	0.7133	61.1	38.9
025Z	0.9954	0.5912	61.0	39.0
025J	1.1377	0.6752	60.9	39.1
average			61.1	38.9
SD			0.4	0.4
RSD			0.7	1.1

n-1=21	t (0.05)	t (0.02)
	2.080	2.518
average	38.9	38.9
uncertainty	0.2	0.2

JRC code	sample mass	cotton mass	cotton	polylactide
	g	g	%	%
025-1	1.0197	0.6037	59.2	40.8
025-2	1.1454	0.6879	60.1	39.9
025-3	0.9808	0.5969	60.9	39.1
average			60.0	40.0
SD			0.8	0.8
RSD			1.4	2.1

n-1=2	t (0.05)	t (0.02)
	4.303	6.964
average	40.0	40.0
uncertainty	2.1	3.3

Results obtained by Cargill Dow using their own method.

55.4 % polylactide – 44.6 % viscose (sample 033)

JRC code	sample mass	viscose mass	viscose	polylactide
	g	g	%	%
033A	1.2292	0.5399	46.6	53.4
033B	1.0661	0.4688	46.6	53.4
033C	1.3241	0.5799	46.5	53.5
033D	1.3846	0.6009	46.1	53.9
033E	1.1269	0.4899	46.1	53.9
033F	1.0738	0.4703	46.5	53.5
033G	1.1980	0.5136	45.5	54.5
033H	1.2791	0.5495	45.6	54.4
033I	1.3578	0.5903	46.1	53.9
033L	1.2298	0.5304	45.8	54.2
033M	1.4378	0.6379	47.0	53.0
033N	1.7117	0.7344	45.6	54.4
033O	1.4859	0.6461	46.1	53.9
033P	1.3888	0.5930	45.3	54.7
033Q	1.8781	0.8138	46.0	54.0
033R	0.8738	0.3697	44.9	55.1
033S	0.9964	0.4233	45.1	54.9
033T	0.9689	0.4161	45.6	54.4
033U	0.8267	0.3500	45.0	55.0
033V	0.8952	0.3799	45.1	54.9
033Z	0.8502	0.3622	45.2	54.8
average			45.8	54.2
SD			0.6	0.6
RSD			1.3	1.1

n-1=20	t (0.05)	t (0.02)
	2.086	2.528
average	54.2	54.2
uncertainty	0.3	0.3

JRC code	sample mass	viscose mass	viscose	polylactide
	g	g	%	%
033-1	1.3848	0.6069	43.8	56.2
033-2	1.2133	0.5329	43.9	56.1
033-3	1.2134	0.5258	43.3	56.7
average			43.7	56.3
SD			0.3	0.3
RSD			0.7	0.6

n-1=2	t (0.05)	t (0.02)
	4.303	6.964
average	56.3	56.3
uncertainty	0.8	1.3

Results obtained by Cargill Dow using their own method.

55.6 % polylactide – 44.4 % acrylic (sample 035)

JRC code	sample mass	acrylic mass	acrylic	polylactide
	g	g	%	%
035A	0.9452	0.4151	44.0	56.0
035B	0.9511	0.4166	43.9	56.1
035C	0.7712	0.3284	42.7	57.3
035D	0.8159	0.3507	43.1	56.9
035E	0.7398	0.3193	43.3	56.7
035F	0.7599	0.3300	43.5	56.5
035G	0.9880	0.4255	43.2	56.8
035H	1.0360	0.4470	43.3	56.7
035I	1.2960	0.5683	44.0	56.0
035L	1.0481	0.4572	43.7	56.3
035M	1.8146	0.8050	44.5	55.5
035N	1.8098	0.7820	43.3	56.7
035O	1.5668	0.6917	44.3	55.7
035P	1.9458	0.8783	45.3	54.7
035Q	1.5873	0.6893	43.5	56.5
035R	0.8293	0.3665	44.3	55.7
035S	0.7910	0.3493	44.3	55.7
035T	0.7829	0.3457	44.3	55.7
035U	0.8203	0.3598	44.0	56.0
035V	0.8945	0.3907	43.8	56.2
035Z	0.8524	0.3720	43.8	56.2
average			43.8	56.2
SD			0.6	0.6
RSD			1.3	1.0

n-1=20	t (0.05)	t (0.02)
	2.086	2.528
average	56.2	56.2
uncertainty	0.3	0.3

JRC code	sample mass	acrylic mass	acrylic	polylactide
	g	g	%	%
035-1	1.7828	0.8374	47.0	53.0
035-2	1.6248	0.7377	45.4	54.6
035-3	1.4996	0.6760	45.1	54.9
average			45.8	54.2
SD			1.0	1.0
RSD			2.2	1.9

n-1=2	t (0.05)	t (0.02)
	4.303	6.964
average	54.2	54.2
uncertainty	2.5	4.1

Results obtained by Cargill Dow using their own method.

55.8 % polylactide – 44.2 % nylon (sample 037)

JRC code	sample mass	nylon mass	nylon	polylactide
	g	g	%	%
037A	2.1693	0.9321	44.1	55.9
035B	1.8412	0.7950	44.3	55.7
037C	2.1246	0.9343	45.1	54.9
037D	1.9108	0.8379	45.0	55.0
037E	2.3751	1.0290	44.5	55.5
037F	2.7275	1.2008	45.2	54.8
037G	2.2492	0.9820	44.8	55.2
037H	1.8926	0.8221	44.6	55.4
037I	2.0650	0.9004	44.7	55.3
037L	2.2563	0.9939	45.2	54.8
037M	1.7407	0.7528	44.4	55.6
037N	1.6901	0.7340	44.6	55.4
037O	2.0456	0.8803	44.2	55.8
037P	2.4172	1.0578	44.9	55.1
037Q	2.1487	0.9352	44.7	55.3
037R	0.9364	0.4065	44.5	55.5
037S	1.0152	0.4389	44.4	55.6
037T	0.9495	0.4128	44.6	55.4
037U	1.0461	0.4560	44.7	55.3
037V	0.8593	0.3729	44.5	55.5
037Z	0.8849	0.3847	44.6	55.4
average			44.6	55.4
SD			0.3	0.3
RSD			0.7	0.6

n-1=20	t (0.05)	t (0.02)
	2.086	2.528
average	55.4	55.4
uncertainty	0.1	0.2

JRC code	sample mass	nylon mass	nylon	polylactide
	g	g	%	%
037-1	1.3490	0.6157	45.6	54.4
037-2	1.3059	0.5905	45.2	54.8
037-3	1.3547	0.6148	45.4	54.6
average			45.4	54.6
SD			0.2	0.2
RSD			0.5	0.4

n-1=2	t (0.05)	t (0.02)
	4.303	6.964
average	54.6	54.6
uncertainty	0.5	0.9

Results obtained by Cargill Dow using their own method.

56.1 % polylactide – 43.9 % silk (sample 041)

JRC code	sample mass	silk mass	silk	polylactide
	g	g	%	%
041A	2.7896	1.1656	44.0	56.0
041B	2.5813	1.0752	43.8	56.2
041C	2.6227	1.0887	43.7	56.3
041D	2.0463	0.8542	43.9	56.1
041E	3.2268	1.3470	43.9	56.1
041F	2.4982	1.0453	44.0	56.0
041G	2.1965	0.9151	43.9	56.1
041H	3.6365	1.5291	44.2	55.8
041I	1.6373	0.6898	44.3	55.7
041L	2.5817	1.0831	44.1	55.9
041M	0.7625	0.3198	44.1	55.9
041N	1.1372	0.4747	43.9	56.1
041O	1.2388	0.5135	43.6	56.4
041P	0.9558	0.3958	43.6	56.4
041Q	0.8146	0.3383	43.7	56.3
041R	1.2295	0.5120	43.8	56.2
041S	1.1075	0.4616	43.9	56.1
041T	1.0756	0.4465	43.7	56.3
041U	1.2613	0.5273	44.0	56.0
041V	1.1116	0.4671	44.2	55.8
average			43.9	56.1
SD			0.2	0.2
RSD			0.5	0.4

n-1=19	t (0.05)	t (0.02)
	2.093	2.539
average	56.1	56.1
uncertainty	0.1	0.1

JRC code	sample mass	silk mass	silk	polylactide
	g	g	%	%
041-1	1.0452	0.4468	42.7	57.3
041-2	0.8536	0.3631	42.5	57.5
041-3	1.0075	0.4294	42.6	57.4
average			42.6	57.4
SD			0.1	0.1
RSD			0.2	0.2

n-1=2	t (0.05)	t (0.02)
	4.303	6.964
average	57.4	57.4
uncertainty	0.3	0.4

Results obtained by Cargill Dow using their own method.

100 % polyester (samples 003-015)

JRC code	sample mass	polyester mass	polyester
	g	g	%
003A	0.9310	0.9232	100.2
003B	0.9299	0.9216	100.1
015A	1.0192	1.0090	100.0
015B	1.1435	1.1320	100.0
015C	1.1946	1.1835	100.1
015D	1.1767	1.1655	100.0
015E	1.1761	1.1641	100.0
015F	0.9157	0.9064	100.0
015G	0.9547	0.9498	100.5
015H	1.0944	1.0886	100.5
015I	1.1067	1.1012	100.5
015L	1.4729	1.4654	100.5
015M	1.2749	1.2676	100.4
015L	0.8811	0.8771	100.5
average			100.2
SD			0.2
RSD			0.2

n-1=13	t (0.05)	t (0.02)
	2.160	2.650
average	100.2	100.2
uncertainty	0.1	0.2

JRC code	sample mass	polyester mass	polyester
	g	g	%
015-1	0.9972	0.9929	99.6
015-2	0.9989	0.9954	99.6
015-3	1.0046	1.0014	99.7
015-4	1.0271	1.0254	99.8
015-5	1.0188	1.0156	99.7
015-6	1.0115	1.0107	99.9
015-7	0.996	0.9933	99.7
015-8	1.0088	1.0061	99.7
015-9	1.0138	1.012	99.8
015-10	1.0029	1.0002	99.7
average			99.7
SD			0.1
RSD			0.1

n-1=9	t (0.05)	t (0.02)
	2.262	2.821
average	99.7	99.7
uncertainty	0.1	0.1

Results obtained by Cargill Dow using their own method.

100 % wool (samples 006-013)

JRC code	sample mass	wool mass	wool
	g	g	%
006A	0.9548	0.9521	99.8
006B	1.3100	1.3055	99.7
013A	1.0662	1.0646	99.9
013B	1.1384	1.1358	99.8
013C	1.1282	1.1234	99.6
013D	1.3599	1.3562	99.8
013E	0.8612	0.8573	99.6
013M	0.9250	0.9226	99.8
013N	0.9999	0.9985	99.9
013O	1.0286	1.0284	100.0
013P	1.1144	1.1119	99.8
013Q	0.9323	0.9300	99.8
013R	0.9991	0.9974	99.9
average			99.8
SD			0.1
RSD			0.1

n-1=12	t (0.05)	t (0.02)
	2.179	2.681
average	99.8	99.8
uncertainty	0.1	0.1

JRC code	sample mass	wool mass	wool
	g	g	%
013-1	0.9722	0.9851	101.3
013-2	0.9792	0.9904	101.1
013-3	1.0430	1.0571	101.4
013-4	0.9538	0.9653	101.2
013-5	1.0387	1.0513	101.2
013-6	0.9538	0.9669	101.4
013-7	0.9928	1.0058	101.3
013-8	0.9950	1.0067	101.2
013-9	1.0036	1.0171	101.3
013-10	1.0095	1.0227	101.3
Average			101.3
SD			0.1
RSD			0.1

n-1=9	t (0.05)	t (0.02)
	2.262	2.821
average	101.3	101.3
uncertainty	0.1	0.1

Results obtained by Cargill Dow using their own method.

100 % cotton (samples 005-011)

JRC code	sample mass	cotton mass	cotton
	g	g	%
005A	1.2893	1.2857	99.74
005B	1.3863	1.3826	99.75
011A	1.0908	1.0892	99.86
011B	1.0441	1.0422	99.83
011C	1.0843	1.0820	99.80
011D	1.3556	1.3524	99.78
011M	1.2912	1.2892	99.86
011N	1.1193	1.1186	99.94
011O	1.2207	1.2194	99.90
011P	0.9624	0.9615	99.91
011Q	1.0109	1.0094	99.86
011R	1.3619	1.3602	99.88
average			99.84
SD			0.06
RSD			0.06

n-1=11	t (0.05)	t (0.02)
	2.201	2.718
average	99.84	99.8
uncertainty	0.04	0.1

JRC code	sample mass	cotton mass	cotton
	g	g	%
011-1	0.9995	0.9805	98.1
011-2	1.0136	0.9957	98.2
011-3	1.0091	0.9909	98.2
011-4	0.9949	0.9763	98.1
011-5	1.0034	0.987	98.4
011-6	1.0121	1.0036	99.2
011-7	1.0242	1.0102	98.6
011-8	1.016	1.0002	98.4
011-9	1.0233	1.0053	98.2
011-10	1.0011	0.982	98.1
average			98.4
SD			0.3
RSD			0.3

n-1=9	t (0.05)	t (0.02)
	2.262	2.821
average	98.4	98.4
uncertainty	0.2	0.3

Results obtained by Cargill Dow using their own method.

100 % viscose (sample 034)

JRC code	sample mass	viscose mass	viscose
	g	g	%
034A	1.2243	1.2247	100.0
034B	0.9196	0.9189	99.9
034C	1.4869	1.4871	100.0
034D	0.9923	0.9925	100.0
034E	0.9544	0.9526	99.8
034F	0.9496	0.9485	99.9
034G	0.9628	0.9631	100.0
034H	1.0696	1.0670	99.8
034I	1.0484	1.0454	99.7
034L	1.0138	1.0125	99.9
average			99.9
SD			0.1
RSD			0.1

n-1=9	t (0.05)	t (0.02)
	2.262	2.821
average	99.9	99.9
uncertainty	0.1	0.1

JRC code	sample mass	viscose mass	viscose
	g	g	%
034-1	1.5970	1.5379	96.3
034-2	1.3006	1.2650	97.3
034-3	1.2226	1.1889	97.2
average			96.9
SD			0.6
RSD			0.6

n-1=2	t (0.05)	t (0.02)
	4.303	6.964
average	96.9	96.9
uncertainty	1.4	2.2

Results obtained by Cargill Dow using their own method.

100 % acrylic (sample 036)

JRC code	sample mass	acrylic mass	acrylic
	g	g	%
036A	1.1700	1.1677	99.8
036B	1.2948	1.2913	99.7
036C	1.0701	1.0694	99.9
036D	1.1862	1.1840	99.8
036E	0.9913	0.9898	99.8
036F	1.1249	1.1225	99.8
036G	1.0324	1.0318	99.9
036H	1.1681	1.1649	99.7
036I	1.0858	1.0839	99.8
036L	1.2352	1.2318	99.7
average			99.8
SD			0.1
RSD			0.1

n-1=9	t (0.05)	t (0.02)
	2.262	2.821
average	99.8	99.8
uncertainty	0.1	0.1

JRC code	sample mass	acrylic mass	acrylic
	g	g	%
036-1	0.9362	0.9332	99.7
036-2	1.1597	1.1512	99.3
036-3	1.1126	1.1061	99.4
average			99.5
SD			0.2
RSD			0.2

n-1=2	t (0.05)	t (0.02)
	4.303	6.964
average	99.5	99.5
uncertainty	0.5	0.8

Results obtained by Cargill Dow using their own method.

100 % nylon (sample 038)

JRC code	sample mass	nylon mass	nylon
	g	g	%
038A	1.1310	1.1288	99.81
038B	1.2049	1.2021	99.78
038C	1.0485	1.0468	99.85
038D	1.2211	1.2197	99.89
038E	1.3559	1.3540	99.87
038F	1.1555	1.1540	99.88
038G	1.4490	1.4467	99.85
038H	1.2394	1.2368	99.80
038I	1.4706	1.4689	99.89
average			99.85
SD			0.04
RSD			0.04

n-1=8	t (0.05)	t (0.02)
	2.306	2.896
average	99.85	99.85
uncertainty	0.03	0.04

JRC code	sample mass	nylon mass	nylon
	g	g	%
038-1	1.2600	1.2505	99.2
038-2	1.4078	1.4052	99.8
038-3	1.3077	1.3018	99.5
average			99.5
SD			0.3
RSD			0.3

n-1=2	t (0.05)	t (0.02)
	4.303	6.964
average	99.5	99.5
uncertainty	0.7	1.1

Results obtained by Cargill Dow using their own method.

100 % silk (sample 042)

JRC code	sample mass	silk mass	silk
	g	g	%
042A	1.1808	1.1742	99.5
042B	0.6666	0.6609	99.2
042C	0.9148	0.9110	99.6
042D	0.9007	0.8970	99.6
042E	0.9426	0.9365	99.4
042F	1.0873	1.0812	99.5
042G	0.6856	0.6829	99.6
042H	1.0507	1.0489	99.8
042I	1.1051	1.1031	99.8
042L	1.1426	1.1362	99.5
average			99.6
SD			0.2
RSD			0.2

n-1=9	t (0.05)	t (0.02)
	2.262	2.821
average	99.6	99.6
uncertainty	0.1	0.2

JRC code	sample mass	silk mass	silk
	g	g	%
042-1	0.9484	0.9250	97.53
042-2	1.0590	1.0325	97.50
042-3	1.1503	1.1224	97.57
average			97.53
SD			0.04
RSD			0.04

n-1=2	t (0.05)	t (0.02)
	4.303	6.964
average	97.53	97.53
uncertainty	0.10	0.15

Results obtained by Cargill Dow using their own method.

100 % polylactide – 268 denier (samples 001-008-010)

JRC code	sample mass	residue mass	polylactide
	g	g	%
001A	1.2069	0.0012	99.90
001B	1.3410	0.0009	99.93
001C	1.9664	0.0027	99.86
001D	1.3609	0.0018	99.87
010A	1.0636	0.0010	99.91
010B	1.0212	0.0007	99.93
010C	1.1820	0.0008	99.93
010D	1.1541	0.0008	99.93
010E	1.0062	0.0000	100.00
010F	1.2932	0.0015	99.89
008A	1.0689	0.0003	99.97
008B	1.0692	0.0003	99.97
008C	1.0295	0.0017	99.84
008D	1.0991	0.0012	99.89
008E	0.9950	0.0014	99.86
008F	1.0661	0.0016	99.85
008G	1.4948	0.0009	99.94
008H	0.9984	0.0007	99.93
008I	1.0298	0.0009	99.91
008L	0.8703	0.0008	99.91
average			**99.91**
SD			**0.04**
RSD			**0.04**

n-1=19	t (0.05)	t (0.02)
	2.093	2.539
average	**99.91**	**99.91**
uncertainty	**0.02**	**0.02**

100 % polylactide – 654 denier (sample 009)

JRC code	sample mass	residue mass	polylactide
	g	g	%
009A	1.1969	0.0002	99.98
009B	1.2259	0.0000	100.00
009C	1.1143	0.0011	99.90
009D	1.4292	0.0018	99.88
009E	1.3891	0.0012	99.91
009F	1.1325	0.0009	99.92
009G	1.1140	0.0009	99.92
009H	1.0783	0.0007	99.94
009I	1.2186	0.0010	99.92
009L	1.3285	0.0012	99.91
009M	1.1761	0.0009	99.92
009N	0.9656	0.0005	99.95
009M	0.8464	0.0015	99.82
009N	0.9485	0.0012	99.87
009O	0.7065	0.0012	99.83
009P	1.0740	0.0030	99.72
009Q	0.9223	0.0024	99.74
009R	0.8907	0.0010	99.89
009S	0.9235	0.0015	99.84
009T	1.0072	0.0012	99.88
009U	1.0054	0.0011	99.89
009V	0.9751	0.0013	99.87
average			**99.89**
SD			**0.07**
RSD			**0.07**

n-1=21	t (0.05)	t (0.02)
	2.080	2.518
average	**99.89**	**99.89**
uncertainty	**0.03**	**0.04**

Annex IV

Spectroscopic analysis

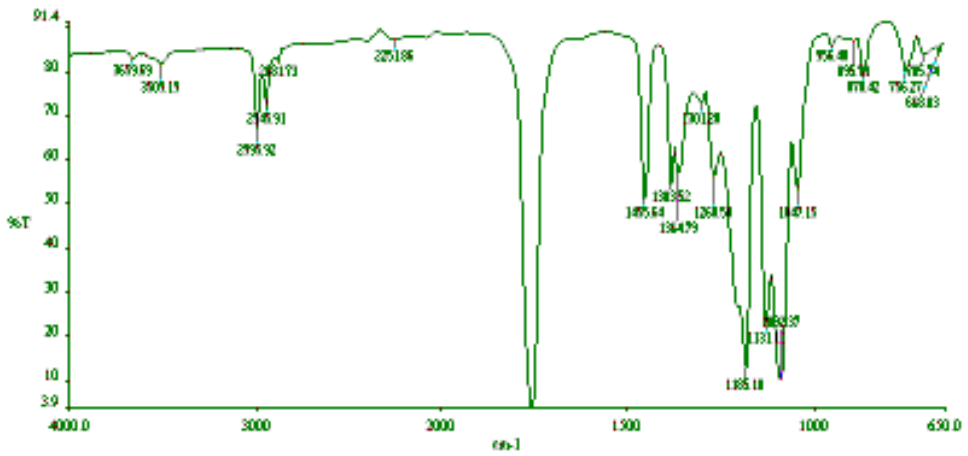


Fig 15: FT-IR spectrum of a film of pure polylactide (sample **010**). Cargill Dow's procedure.

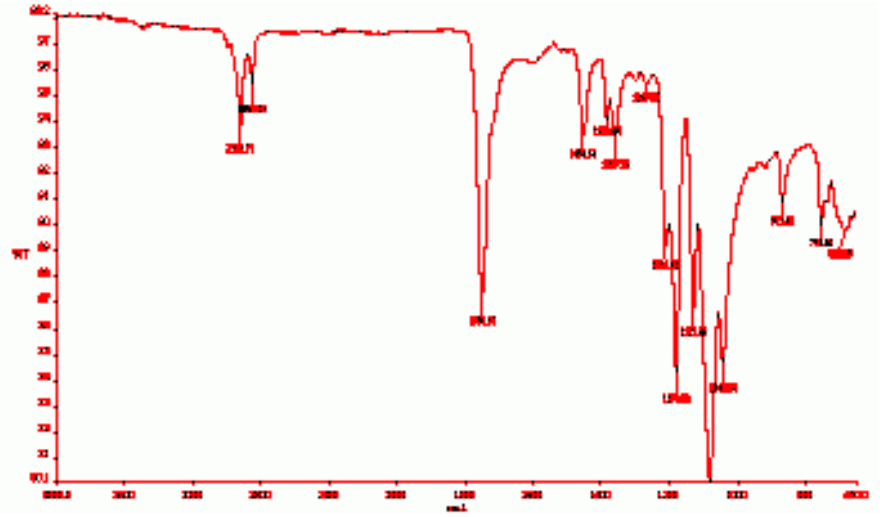


Fig 16: FT-IR spectrum (ATR) of pure polylactide (sample **010**).

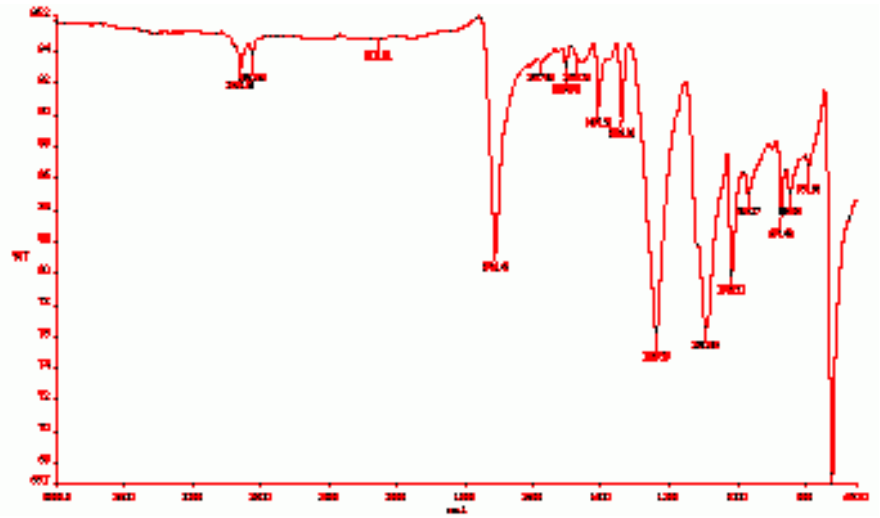


Fig 17: FT-IR spectrum (ATR) of pure polyester (sample **015**).

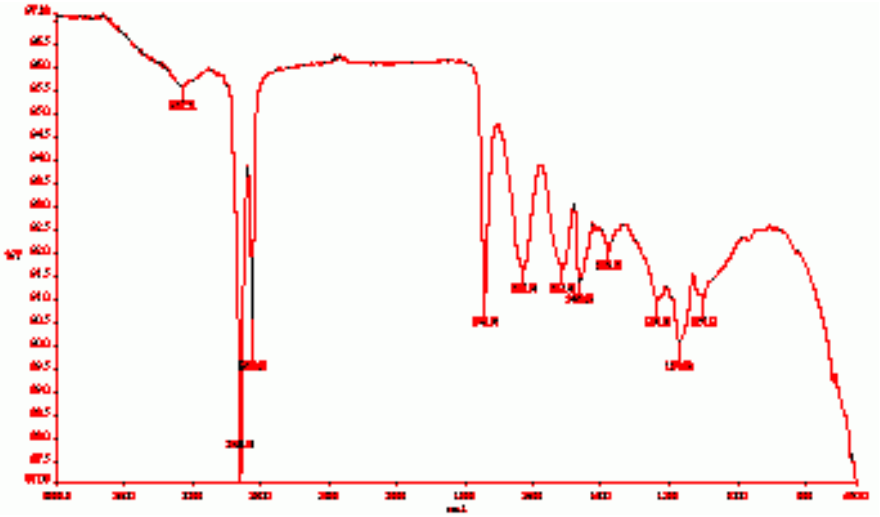


Fig 18: FT-IR spectrum (ATR) of pure wool (sample **013**).

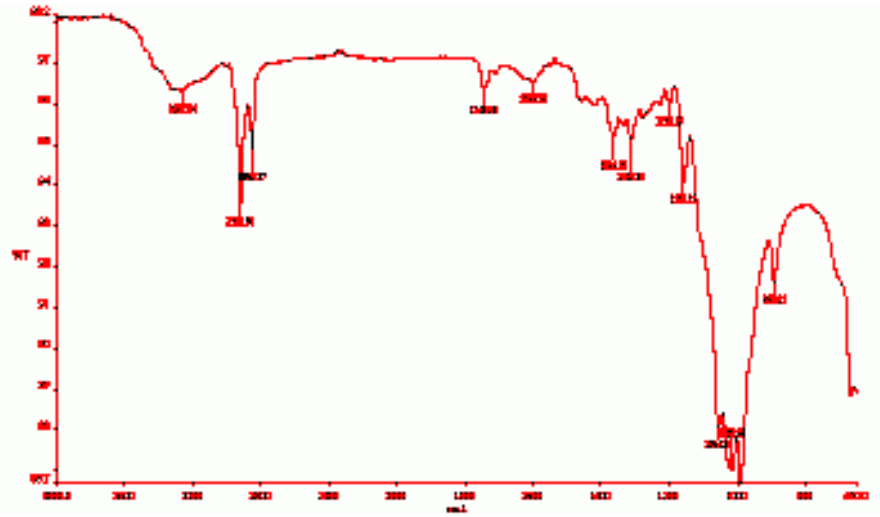


Fig 19: FT-IR spectrum (ATR) of pure cotton (sample **011**).

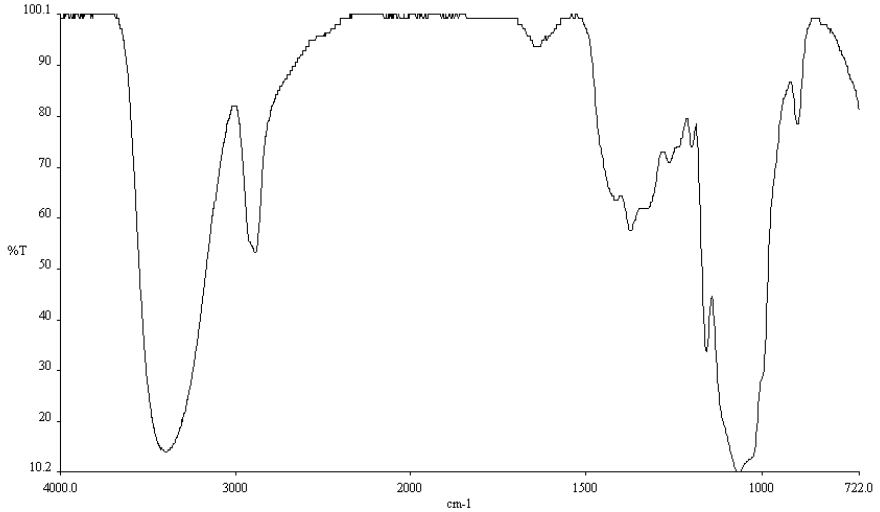


Fig 20: FT-IR spectrum (ATR) of pure viscose (sample **034**).

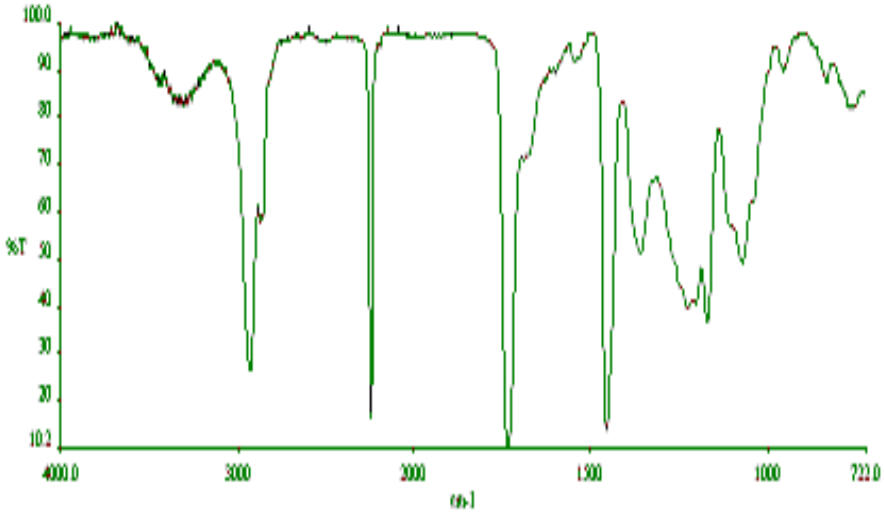


Fig 21: FT-IR spectrum (ATR) of pure acrylic (sample **036**).

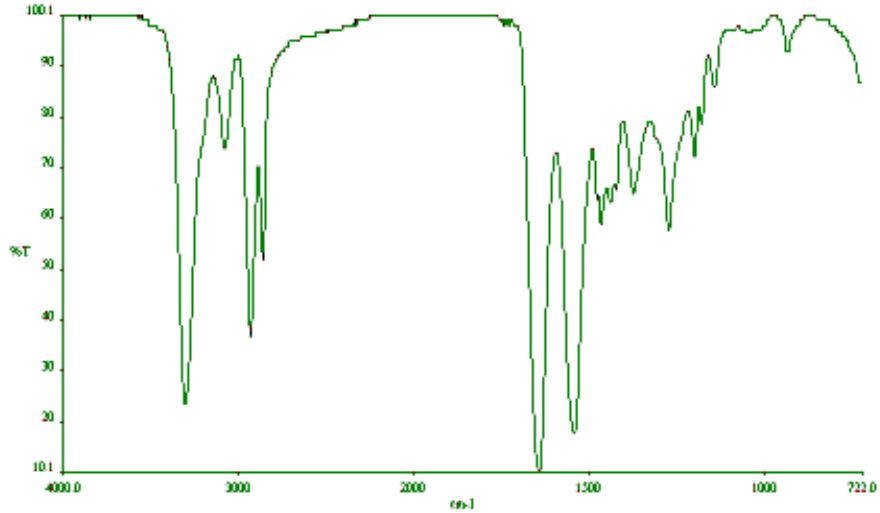


Fig 22: FT-IR spectrum (ATR) of pure nylon (sample **038**).

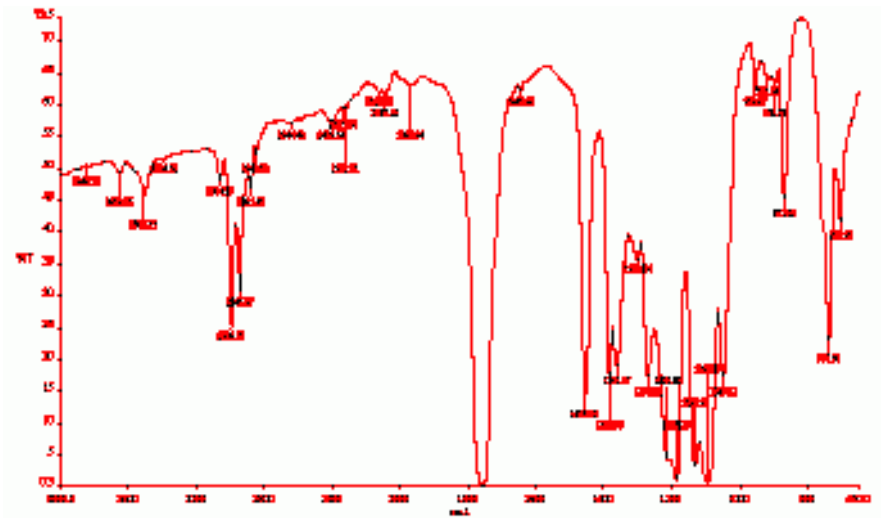


Fig 23: FT-IR spectrum (ATR) of pure silk (sample **042**).

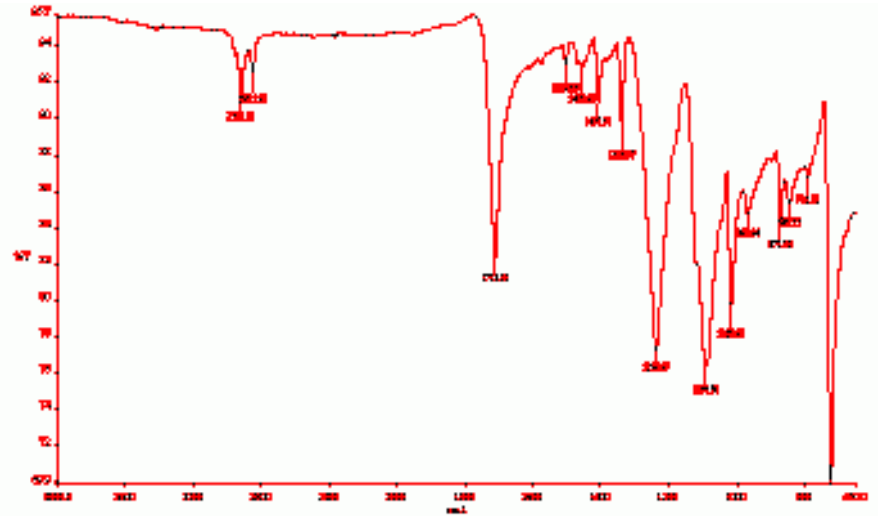


Fig 24: FT-IR spectrum (ATR) of sample **014** (50.2% polylactide – 49.8% polyester).

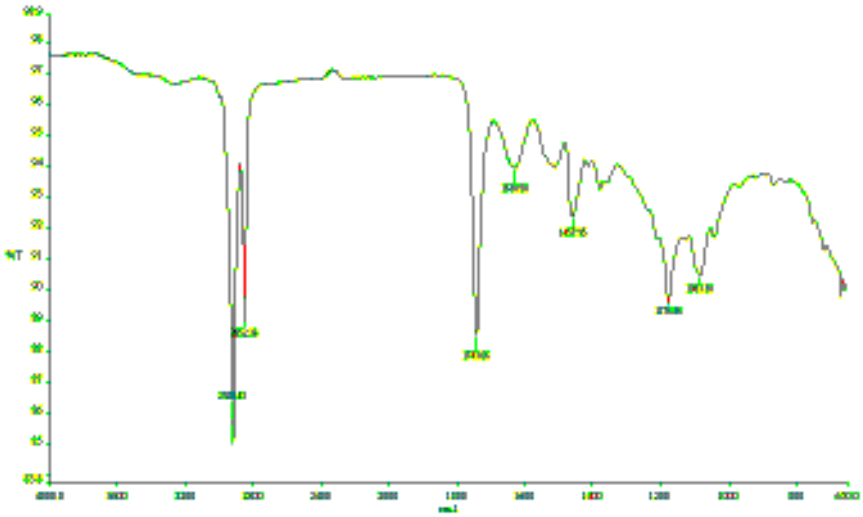


Fig 25: FT-IR spectrum (ATR) of sample **012** (63.7% polylactide – 36.3% wool).

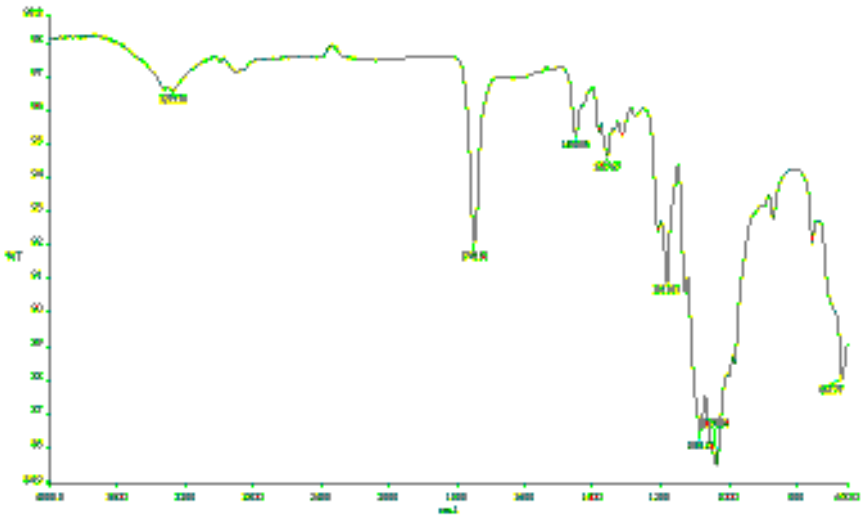


Fig 26: FT-IR spectrum (ATR) of sample 016 (50% polylactide – 50% cotton).

Annex V

Determination of agreed allowance

JRC code	dried sample mass	wet sample mass	water mass	agreed allowance
	g	g	g	%
008A	2.1402	2.1469	0.0067	0.31
008B	2.0076	2.0142	0.0066	0.33
008C	1.7962	1.8018	0.0056	0.31
008D	1.7215	1.7268	0.0053	0.31
average				0.32
SD				0.01
RSD				2.97

n-1=3	t (0.05)	t (0.02)
	3.1824	4.5407
average	0.32	0.32
uncertainty	0.02	0.02

Table 8: Pre-treated sample – 100% polylactide – yarn – 268 denier.

JRC code	dried sample mass	wet sample mass	water mass	agreed allowance
	g	g	g	%
010A	1.5789	1.5825	0.0036	0.23
010B	2.2466	2.2523	0.0057	0.25
010C	1.7798	1.7842	0.0044	0.25
010D	1.9168	1.9214	0.0046	0.24
average				0.24
SD				0.01
RSD				5.50

n-1=3	t (0.05)	t (0.02)
	3.1824	4.5407
average	0.24	0.24
uncertainty	0.02	0.03

Table 9: Untreated sample – 100% polylactide – yarn – 268 denier.

JRC code	dried sample mass	wet sample mass	water mass	agreed allowance
	g	g	g	%
018A	2.2456	2.2525	0.0069	0.31
018B	2.0372	2.0433	0.0061	0.30
018C	2.265	2.2721	0.0071	0.31
018D	2.8461	2.855	0.0089	0.31
average				0.31
SD				0.01
RSD				2.29

n-1=3	t (0.05)	t (0.02)
	3.1824	4.5407
average	0.31	0.31
uncertainty	0.01	0.02

Table 10: Untreated sample – 100% polylactide – staple yarn.

JRC code	dried sample mass	wet sample mass	water mass	agreed allowance
	g	g	g	%
019A	2.6953	2.7034	0.0081	0.30
019B	2.2647	2.2713	0.0066	0.29
019C	2.063	2.0686	0.0056	0.27
019D	2.7482	2.7557	0.0075	0.27
average				0.29
SD				0.01
RSD				5.17

n-1=3	t (0.05)	t (0.02)
	3.1824	4.5407
average	0.29	0.29
uncertainty	0.02	0.03

Table 11: Untreated sample – 100% polylactide – staple fibre.

JRC code	agreed allowance
	%
009A	0.23
009B	0.25
009C	0.29
009D	0.25
average	0.26
SD	0.02
RSD	8.55

n-1=3	t (0.05)	t (0.02)
	3.1824	4.5407
average	0.26	0.26
uncertainty	0.03	0.05

Table 12: Untreated sample – 100% polylactide – yarn – 654 denier – CNR results.

JRC code	agreed allowance
	%
009E	0.21
009F	0.22
average	0.22

Table 13: Pre-treated sample – 100% polylactide – yarn – 654 denier – CNR results.

Mission of the JRC

The mission of the Joint Research Centre is to provide customer-driven scientific and technical support for the conception, development, implementation and monitoring of EU policies. As a service of the European Commission, the JRC functions as a reference centre of science and technology for the Union. Close to the policy-making process, it serves the common interest of the Member States, while being independent of special interests, whether private or national.