Open-BIO

Opening bio-based markets via standards, labelling and procurement

Work package 4

Functionality Testing

Deliverable N° D4.6:

Overview report on functionality testing

Mechanical, chemical, thermal and additional functionality testing

Public

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1 Publishable summary

This is a the public deliverable (D 4.6: Overview report on functionality testing) that summarizes D4.5 (Standardization report on bio-based product testing).

Within the Open-Bio project 7 product categories were adressed;

1. Packaging Films
2. Disposable cups & plates
3. WPC decking
4. Pre manufactured components/insulation
5. Mulch films
6. Adhesives and binders
7. Bio-solvents

Based on the above product categories, commercially available bio-based products have been selected for functionality testing. Key mechanical properties, key chemical/thermal parameters and additional functionalities as defined in the combined deliverable D4.2, D4.3 and D4.4 were evaluated and discussed in D4.5.

Work on Packaging Films and Mulch films was organised and performed by AUA, with the contribution of Novamont and OWS. WFBR organised, and performed work on disposable cups & plates, WPC decking, adhesives and binders and solvents with help of ISA. Work on premanufactured component/insulation materials was limited to a desk study performed by WFBR.

The most important findings (highlights) for each product category are listed below.

1.1 Packaging films;

Main recommendation is to add properties at frozen conditions and very wet conditions to the MDS (material data sheet) since the properties of most bio-based films deteriorate under these conditions. Another addition could be the CO_2 transmission rate values as this is a critical property for Modified Atmosphere packaging of food products. Reproducibility of Elmendorf tear propagation and the WVTR (water vapour transmission rate) between two labs appeared to be poor. Information of the applicability of the test method and further working instructions are recommended.

1.2 Disposable cups and plates;

Main hurdle is that there are no standards to proof the functionality of a disposable cup that could be referred to when new products are marketed. Therefore evaluation was done by using adaptations of standards developed either for paper and board or for plastics. Also, Due to the limited dimensions of the cups, mechanical testing had to be adapted to the dimensions of the available product, usually much smaller than the ones indicated in the standards. The different testing conditions after use (coffee, and several times pouring hot water) show that cups can deal with high temperature liquids, the functionality of the coating in paperboard based cups is sufficient to reuse the cup up to three times.

1.3 WPC-decking

WPC-decking replaces solid wood in outdoor applications and testing is directed towards properties were WPC decking behaves differently from solid wood. There is a harmonized European standard for WPC-decking (EN 15534) and various properties listed in this standard studied in detail. It is recommended to use more demanding conditions both for testing high temperature creep (longer creep periods and higher loads) and low temperature impact (increase dropping height). More over an approach to develop a cleanability test method has been drafted.

1.4 Premanufactured components/isolation

Work on the product category was limited to a desk study because of the complexity of the material (insulation should be measured in a construction) and the highly expensive and very specific type of testing. Based on the desk study there are several recommendations. The advantages of biofibre based insulation mats cannot be marketed effectively due to standard test methods which are not based on practically most relevant testing conditions. It is proposed that the testing method specifies analysis of the thermal conductivity performance of insulation materials at typical winter conditions, and at typical summer conditions, e.g. at 60°C and 60 or 80% RH. using direct measurements (rather than calculations). The advantages sound insulation properties could be demonstrated by testing of sound absorption at different frequencies according to EAD 040005-00-12.01 (section 2.2.8). Just analysing the insulation material alone (not in a construction) will give a good first indication of its performance.

1.5 Mulching films

The biodegradable mulch films are susceptible for ageing resulting in a decrease of mechanical properties. This may not hinder the functionality in the application but still it is recommended to include a section on the artificial weathering exposure of bio-based films also in the draft standard prEN 17033. Moreover, it is advised to measure the relative light transmission over the whole visible spectrum (380-780 nm) and not over the only a portion of the visible radiation spectrum, 500-600 nm like t suggested by the prEN 17033 draft.

1.6 Adhesives and binders.

A bio-based paint and a traditional oil based paint with the same color and for the same application were tested for mechanical and physical properties. A general observation is that most test require very specific (and expensive) test set-ups. In most of the tests the bio-based paint results are comparable to the oil-based paint. Scrub tests show that bio-based paint has good adhesion to the glass plate and scrubs less than the oil-based paint. The bio-based paint is more water sensitive and it absorbs water. Still it is not clear if this hinders commercial use of the paint.

1.7 Solvents

For bio-based solvents that aim to replace existent, well known petro-based solvents, a set of characteristics to determine its functionality can be crucial in its successful introduction in the market. Still, solvents are applied in a broad range of markets with their own specific standard test methods and requirements. As a start Hansen solubility parameters can be helpful to use as a first guide on applicability. There are several ways of characterizing thermal properties of solvents. TGA curves give a first impression on the volatility of the solvents and the temperatures of complete and 50% evaporation. This is certainly a valuable method that is highly accessible for most laboratories.

2 Introduction

The objective of work package 4 “Functionality Testing” is to demonstrate the functionality of novel Bio-based Products. The work is a follow-up on KBBPPS results regarding products of major importance and functionality barriers

This is the public deliverable (D 4.6: Overview report on functionality testing) that summarizes D4.5 (Standardization report on bio-based product testing).

As reported within D4.1 (Product category selection) 7 key product categories were selected for testing;

1. Packaging Films
2. Disposable cups & plates
3. WPC decking
4. Pre manufactured components/insulation
5. Mulch films
6. Adhesives and binders
7. Bio-solvents

Key mechanical properties, key chemical/thermal parameters and additional functionalities that ensure the fucntionality of the bio-based project over its entire product life are defined and described in in the combined deliverable D4.2 (mechanical testing), D4.3 (chemical/thermal testing) and D4.4 (other functionalities). This implies testing before and after use or before and after ageing. Additionally, specific characteristics of the bio-based products that differ from existing petrochemical products were addressed.

In each product category commercially available bio-based products have been selected for functionality testing. The outcome of these test are described in the D4.5.

Work on Packaging Films and Mulch films was organised and performed by AUA, with the contribution of Novamont and OWS. WFBR organised, and performed work on disposable cups & plates, WPC decking, adhesives and binders and solvents with help of ISA. Work on premanufactured component/insulation materials was limited to a desk study performed by WFBR.

Summary of the test results and recommendations is presented in the following chapters.

3 Packaging films

3.1 Introduction

The key packaging materials used worldwide according are paper and board and plastics. The food sector is by far the largest consumer of packaging material. Since paper and board are well-known and extensively studied materials, focus within this project will be placed in the food packaging category of products on innovative bio-based plastic packaging. Among the key requirements for the materials used for food packaging are: protection, tampering resistance and special physical, chemical or biological needs to ensure safe and high quality products for the consumer.

3.2 Approach

Food packaging films selected for testing within Open-Bio include 4 typical examples of bio-based compostable food packaging films that have a commercial significance (PLA and PLA blends). A BOPP film was also tested as conventional fossil-oil based reference packaging film product. Standard testing methods were used for testing the most important mechanical properties (tensile properties, tear propagation, penetration resistance, seal strength) and additional physical properties (radiometric properties, gas transport properties and wetting tension) of the plastic food packaging films.

3.3 Results and Recommendations

The tensile properties of the packaging films under standard testing conditions are in agreement with the corresponding values given by the manufacturers (MDS). PLA blends based films have lower tensile strength and higher elongation at break values as compared to PLA and BOPP films. The tensile strength of the films is not affected by low temperature (-18ºC) and water soaking. The elongation at break is reduced under frozen conditions for all bio-based films. The elongation at break of some PLA blends is also reduced under water soaking conditions. A good agreement is confirmed between the laboratories that participated in these tests.

It is recommended to include in the specifications for packaging films two additional conditions in testing the tensile properties of bio-based and conventional packaging films: testing under low temperature (-18ºC) and testing the film soaked in water.

The tear propagation resistance values measured following the Elmendorf method by the participating laboratories and the MDS value reported for a PLA blend based film show significant differences. The measurement of the tear propagation resistance of these very thin films by the Elmendorf tear tester was found to be difficult with high deviations and questionable reliability. The standard itself confirms high standard deviations and discrepancies been reported. The Elmendorf method results should be considered with caution and possibly evaluated as “not good or misleading”. The use of the Elmendorf method is in general not recommended for thin food packaging films.

The penetration resistance tests revealed a typical linear elastic behaviour for BOPP and PLA films with high penetration resistance values while PLA blend based films showed a nonlinear behaviour with a two-peak curves in some cases. The penetration resistance for these films was calculated based on the maximum load and was significantly lower than that of BOPP. To allow for the applicability of existing standards for measuring the penetration resistance of bio-based films it is recommended that “penetration resistance” is defined as the maximum load, whether this occurs in the first or the second peak in cases of two peak curves. Likewise, energy for penetration shall be defined as the energy at maximum load.

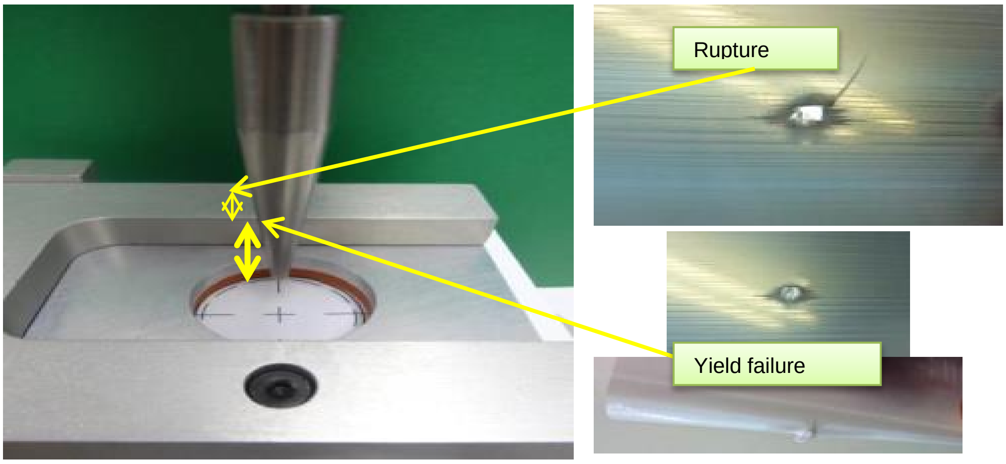

Figure 1 Measuring penetration resistance of packaging films.

The seal strength values of PLA and PLA blends based films are higher than those of the conventional BOPP packaging films. Since most of the commercial thermo-sealing machines are designed for a sealing width of 2 mm, it is recommended to define seal strength as the average maximum force to peel the seal for the given 2 mm sample width, based on a number of at least five measurements.

Radiometric properties: The PLA blends based films exhibit high haze values while BOPP and PLA present high clarity. The values obtained by the two participating labs for transmittance are in good agreement. The inter-laboratory values of haze and the values given by the manufacturers (MDS), generally show a good agreement (except for 1 film). As the total transmittance values measured for bio-based packaging films with high haze values vary with the spectra wavelength, it is recommended to present also, as complementary information, the variation of the measured total transmittance and haze values with the wavelength for the given test materials and film thickness.

Water vapour transmittance: The WVTR values of all bio-based films exceed significantly the corresponding value of the conventional reference BOPP film. A disagreement is observed between the WVTR values obtained by the two laboratories following the same ASTM E96 / E96M – 14 standard testing method, possibly because of the different approaches employed to achieve the required testing conditions. Also, large discrepancies are found between the measured values and those reported by the manufacturers (MDS) employing different standard testing methods and testing conditions. To avoid major discrepancies among the reported WVTR values by various laboratories using the same standard (e.g. ASTM E96 / E96M – 14) it is recommended that the standard provides an approach to be used in achieving the targeted testing conditions. As the WVTR rates depend very much on temperature and possibly on the partial pressure difference of WV across the film, it is also recommended that the same standard testing methods at pre-defined standard testing conditions are followed by the manufacturers (in addition to other conditions tested for specific applications) to measure and report the WVTR values, allowing for the comparison between the reported WVTR values of different products.

O_2 transmission rate values of bio-based films exceed the corresponding values of the conventional reference BOPP film except for PLA film and for the thicker Ecovio film. CO_2 transmission rate values of bio-based films exceed the corresponding values of the conventional reference BOPP film at a higher rate than for O_2 transmission rate except for the PLA film. The values obtained by the two laboratories are in good agreement. No CO_2 transmission rate values are reported in MDS. It is highly recommended to report values CO_2 transmission rate in MDS as this is a critical property for Modified Atmosphere packaging of food products.

Wetting tension: Most of bio-based plastics have moderate to low wettability and printability with water based printing colours and adhesives and increased probability of water condensation inside fresh produce packaging. In order to achieve better wettability of the film surfaces, the application of external layers or surface modification by corona or plasma treatment is needed. Water-alcohol mixtures at high percentages of alcohol allow for improved wettability and printability of water/alcohol based inks. Standard testing methods are using a series of given test mixtures of graduated wetting tension which may be toxic/non-applicable to bio-based food packaging films and they could possibly interfere with compostability. Developing a standard testing method for measuring the wetting tension of bio-based packaging films using water-based non-toxic solutions (e.g. used with compatible inks) is recommended.

4 Disposable cups & plates

4.1 Introduction

Disposable cups & plates find their main use in catering, festivals and in vending machines (coffee). When selecting disposables, paperboard based products are preferred by most consumers and companies because of their environmentally friendly appearance and because they can be printed easily and in higher quality. Paperboard needs to be coated (waterproofing) to obtain a water resistant product. Bio-based biodegradable coatings are increasingly used since this offers additional end-of life opportunities after use via organic recycling.

4.2 Approach

The functionality of three different commercially available coated paperboard disposable cups was evaluated. Two cups with a (partially) bio-based coating and one cup with commonly used petro-based coating were selected for testing, this last one was used for comparison. Requirements that are related to the functionality of disposable cups are mechanical properties like stiffness, strength, elongation properties and the performance of the waterproofing coating. There are no standards available to characterize paperboard based cups as a product. Therefore evaluation was done by using adaptations of standards developed either for paper and board or for plastics.

4.3 Results and Recommendations

Properties evaluated can be divided in two categories: mechanical properties before and after use and/or ageing; and the ability of the cups to hold hot/fatty liquids by means of leaking/permeability tests in the walls and in the seals of the cups. Additionally thermal analysis was also done.

Focusing only on the coating material was difficult due to the large influence of paperboard type in for example mechanical properties. In some of the tests, the coating was separated from the paperboard to be evaluated, however this was not successful for all the products evaluated, giving extra information on the bonding of the coating to the paperboard.

Mechanical testing standards are usually designed for paper and board or for plastic. Within the paper and board standards, there are few standards that mention explicitly coated paper, for example NEN-ISO 5630-5, NEN-EN 13676 and NEN-ISO 16532-1, however in the first one it is difficult to distinguish the ageing of paperboard from the ageing of the coating. Due to the limited dimensions of the cups, mechanical testing had to be adapted to the dimensions of the available product, usually much smaller than the ones indicated in the standards. At least 3 to 5 samples per product were evaluated. All measurements were done after conditioning samples according to ISO 187: standard for paper, board and pulps under the conditions 23°C and 50% relative humidity (R.H.)

Evaluated mechanical properties were tensile strength (ISO 527-1 for plastics) in machine and cross machine direction, before and after use/ageing; resistance to bending (ISO 5628 for paper and board) in machine and cross machine direction, before and after ageing; tear strength in machine and cross machine direction (ISO 1974 for paper) and puncture resistance (NEN-EN 14477 for packaging) before and after use.

The different testing conditions after use (coffee, and several times pouring hot water) show that cups can deal with high temperature liquids, the functionality of the coating in paperboard based cups is sufficient to reuse the cup up to three times. Longer storage of liquids in the disposable cups is not recommended due to possible leaking, de-attachment of the coating, etc., here the extreme case of one week was evaluated. Chosen testing after use conditions (coffee, and several times pouring hot water) evaluate the performance of the bio-based coating and can be adopted as a functionality test, keeping in mind that functionality must be enough for a disposable. The puncture resistance and elongation at break are not influenced by the use of the cups.

Mechanical testing is largely influenced by the paperboard properties instead of by the coating material. Most mechanical tests do not show any difference between bio-based coated cups and a tested petro-based coated paperboard cup before and after use. Cups are certainly suitable as disposables and keep their functionality after limited use. From the evaluated mechanical properties tear strength does not give relevant information on the cups functionality.

Accelerated ageing under the conditions specified in NEN-ISO 5630-5:2009 standard is a suitable method for evaluating storage properties of coated cups, for example during transport or during storage in vending machines. Here tensile strength (ISO 527-1) and resistance to bending (ISO 5628) were evaluated before and after ageing since they are more representative for the functionality of the cups than the folding endurance and tear strength specified in the standard. Exposing disposable cups to elevated temperature at 100°C for 5 days do not give any negative effect on the mechanical properties of the paper-based coated cups neither with bio-based coating nor with petro-based coating.

Permeability and leakage testing was done by evaluating mainly grease and dye through the cups seals. Leakage in seals and walls (pinholes) was done by following an adapted protocol from the one described in NEN-EN 13676 standard. This standard is specific for testing "polymer coated paper and board intended for food contact" so one of the few standards that can be applied to polymer coated cups by adapting the recommended sample size and testing the full cup.

Figure 2 Leakage at the seals for the different coated cups

Seal leakage was observed for all products. It is probably related to the manufacturing process and to a less extent to the actual properties of the bio or petro based coating. NEN-ISO 16532-1 is a suitable test to evaluate grease resistance of paperboard based cups with different polymeric coatings. However, since the tested products are commercially avail-able and probably made suitable for contact with grease, then the tests with palm oil in the middle of the cups do not show any difference after 3 weeks of testing. This indicates that Ecovio and PLA coating materials are comparable to PE coating materials in grease re-sistance. The palm oil did not penetrate any of the tested products indicating that all coatings have good grease resistance independently of the polymer used for the coating.

The evaluation of the seals is in agreement with the results obtained with the dye. Ecovio coated cups seem to allow the pass of oil through the seals earlier than PLA and PE coated cups.

Water vapour permeability is the highest for Ecovio coated cups, followed by PLA coated cups and PE coated cups. It was evaluated by measuring the water vapour transmission rate according to ASTM E96 or by weighting the water vapour accumulated under the cup after exposing it to hot water. In both cases large differences are observed for the different materials. This property is most probably related to the coating material, however higher water vapour transmission does not affect the functionality of the cups to hold hot liquids.

Mechanical properties of cups before and after use/ageing are certainly not a hurdle for widespread introduction of paperboard based cups with bio-based coatings in the market. Permeability tests can be a hurdle for widespread introduction, however it has to be objectively evaluated which values of permeability are sufficient to ensure the functionality of the cup in the market and create market acceptance.

5 WPC decking

5.1 Introduction

WPC's are composite materials produced from wood flour and thermoplastic resins. WPC decking profiles can replace solid wood in outdoor applications were they are promoted because of low maintanance. Commonly decking material is PP filled with wood fibres. Additives like colorants, (UV-) stabilisers and coupling agents are added to obtain the desired properties.

5.2 Approach

Three commercially available WPC decking materials have been selected for testing. Two hollow profiles and one solid profile. Three key parameters have been identified which are currently a technical hurdle for widespread introduction of WPC decking profiles in the European market: creep behaviour at elevated temperature (e.g. 50°C), impact resistance at low temperature (e.g. -20°C) and cleanability. Furthermore, heat build up, though not a property directly related to customer satisfaction, is considered a key parameter as it is a simple test which relates to high temperature creep rate of WPC profiles.

5.3 Summary

Results show that creep deformation of all three profiles meet the requirements at both 20°C and 50°C as specified in EN 15534-4. However, at 50°C and conditions somewhat more demanding than specified in EN 15534-4 though not unrealistic, one grade of the WPC profiles exhibited breaks in 3 out of 4 creep trials. Outdoor trials show that even in the Netherlands the WPC profiles can reach temperatures of around 40-50°C during several days a year. Likely, the temperature of WPC decking in other parts of Europe increases to even higher values. Therefore, it is recommended that creep at elevated temperature is tested at more demanding conditions than presently specified in EN 15534-4.

The reason for the failure of one of the WPC profile grades was its low failure strain when compared to typical biofibre polymer composites. Because EN 15534-4 specifies a maximum creep deflection in mm, which can be met by decreasing the support span distance, the standard does not focus attention to the strain in % in the outer profile layers which determined whether a material fails or not. And when decreasing the span, the material strain in % increases at the same level of deflection in mm. So the standard tends to distract attention from the key failure parameter being failure strain. To overcome this issue, a requirement level for creep strain (%) relative to static failure strain (%) rather than a requirement level for creep deflection in mm could be specified in quality standards.

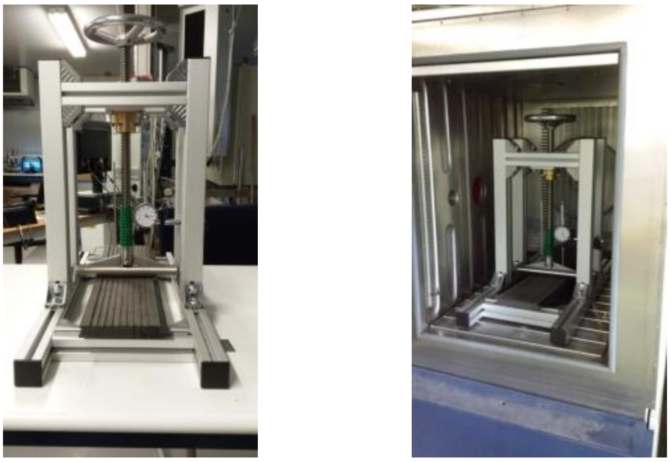

Figure 3 Analysis of creep behaviour of WPC decking profiles at 20 and 50°C 50%RV

All three WPC profiles meet the specifications set for dart impact resistance in EN 15534-4, both at +20°C as well as at -20°C. However, it is questionable whether the impact mass of 1 kg dropped from 70 cm height sufficiently cover accidents reasonably occurring throughout many years of anticipated WPC service life. Impact tests using an increased dropping height of 150 cm, not uncommon for everyday life, show that one of the WPC profile grades leaves residual indentation and crack lengths larger than specified maximum in EN 15534-4. It is recommended that dart impact performance is tested at different and more demanding impact energy levels than presently specified in EN 15534-4. Eventually, different classes for impact performance may be developed.

Heat build up testing shows that both WPC design and colour affect increase of WPC surface temperature resulting from a white IR lamp, showing temperature increases of 45–60°C. The limited number of data collected in this research present a hint that outdoor creep deformation may be correlated to heat build up, however, more trials would be needed in order to allow firm conclusions.

Initial staining and cleanability trials have been performed. Further contamination and cleanability tests on WPC materials have to be performed in order to develop a standard test method for cleanability which is reproducible and which has practical meaning. An approach to develop such a standard cleanability test method has been drafted.

6 Insulation materials

6.1 Introduction

The category pre-manufactured components/insulation focuses on insulation materials were specific barriers were found for novel bio-based insulation materials. Common insulation materials are mineral fibre, expanded polystyrene and polyurethane foams. Examples of bio-based insulation materials are cork, cotton, recycled tissue/clothes, hemp, flax, coco, wool, wood fibers, cellulose, etc .

6.2 Approach

The key technical properties of pre manufactured biofibre insulation materials include:

- Thermal conductivity, relative to mineral wool based insulation, over the relevant relative humidity range.
- Heat storage capacity which is not included in everyday practice and design.
- Sound insulation, which is considered to be better for biofibre than for mineral fibre, however, a clear comparative study on sound absorption seems missing.

As testing of these properties of pre-maufactured components containing bio-based insulation materails would be complex and none of the Open-Bio project partners has got facilities to test these properties, it was decided that no tests will be performed. Instead, the background of the issues has been mapped, and a proposal for dealing with the issues have been developed.

6.3 Results and Recommendations

The advantages of biofibre based insulation mats cannot be marketed effectively due to standard test methods which are not based on practically most relevant testing conditions, but for instance at 10°C and dry conditions and at 23°C and 50% relative humidity (RH). In Germany, testing is required at 80% RH which basically never occurs when insulation materials are installed properly. Furthermore, more or less extreme effects which may occur, like dew water penetration in the insulation mats, are not addressed in testing standards, while the water sorptive and transporting characteristics of biofibre are claimed to provide an advantage for biofibres over mineral wool. Marketing of biofibre insulation materials will benefit from comparative data for biofibre and mineral wool based insulation materials at specific conditions like dew water penetration. Also, the way in which the thermal conductivity of any type of insulation material is measured and calculated seems unnecessarily complex which poses a disadvantage for SMEs. Changing the standards for testing and calculation of thermal conductivity, however, will also affect the rules for engineering.

It is proposed that the testing method specifies analysis of the thermal conductivity performance of insulation materials at typical winter conditions, e.g. at -10°C and 30% RH, and at typical summer conditions, e.g. at 60°C and 60 or 80% RH. The thermal conductivity should be measured directly at the relevant conditions, so without calculations using alternately logarithms and exponents. Such a method would provide a transparent and direct method to compare the thermal conductivity values of different types of insulation materials. Additional test methods would need to be developed, and comparative experimental data would need to be collected, in order to account for specific characteristics not covered so far in standard test standards: a method which analyses the effect of dew water penetration on insulation performance and a method to determine the effect of ageing on thermal conductivity.

Biofibres have a higher heat capacity than mineral wool, which allows for improved protection against summer heat by using biofibres. However, as insulation materials can be covered at both sides with a wide range of materials, and as outside temperature may vary a lot as well, protection against heat from outside has many variables, and consequently communication to consumers is complex. Moreover, calculations tend to focus on extreme conditions and are based on further assumptions.

It is proposed that a relevant set of experimental data will be collected to form the basis for communication to consumers. This set of data should cover different types of insulation material, covering panel, roofing tiles, and different temperatures of the tiles related to air temperature and sun irradiation. Subsequently, existing calculation tools can be used by installers and contractors to address further detailed customer information needs.

Impact sound reduction tests have to be performed for the entire construction, so including the covering, which may be a wood fibre based panel on one side and roofing tiles on the other side. The high costs for such tests limit the possibilities for the manufacturers of biofibre based insulation materials, usually being SMEs. The contribution of insulation materials in between the covering panels and roofing tiles, however, is mainly based on sound absorption (sound reflection mainly being accounted for by the roofing tiles).

It is proposed that the testing of sound absorption at different frequencies according to EAD 040005-00-12.01 (section 2.2.8) by just the insulation material alone will give a good first indication of its performance. Both biofibre and mineral wool based insulation materials need to be tested in a comparative study. Additionally, testing of sound absorption by typical covering panels will provide information on which types of panels make a good match with biofibre insulation materials: A good match is expected to be achieved for covering panels absorbing at frequencies which show highest transmission by the biofibre insulation material.

7 Mulch films

7.1 Introduction

The bio-based biodegradable in the soil mulching films provide a solution for the mismanagement of the plastic mulching (and other agricultural) film waste. They do not need to be removed from the field but also the thinner they are, the faster they biodegrade. This results in significant saving in material (thinner films) and in the elimination of the disposal cost.

7.2 Approach

Products selected for testing are two typical examples of bio-based biodegradable mulching films that have a commercial significance: starch and PLA blend based mulching films. A Linear Low Density Polyethylene (LLDPE) 3-layer mulching film was used as conventional fossil-oil based reference mulching film product. Standard testing methods were used for the mechanical properties (tensile properties, weathering effect on tensile properties, tear propagation, impact resistance) and additional physical properties (radiometric properties, water vapour transmittance) of the plastic mulching films.

7.3 Results and Recommendations

The tensile strength values of the mulching films under standard testing conditions are in agreement with the corresponding values given by the manufacturers (MDS). Tensile strength of the starch-based film decreases under low temperature (-18ºC) (MD/TD) and when soaked in water (MD). The elongation at break of all bio-based and the conventional materials is reduced under frozen conditions. The elongation at break of the starch-based film is also reduced under water conditions (MD). The bio-based and the reference films satisfy the prEN 17033 draft and EN13655 standard requirements, respectively, except for the elongation at break of the PLA blend based film in MD. A good agreement is confirmed between the two laboratories that participated in these tests. Tensile strength and elongation at break of the bio-based and reference mulching films may decrease significantly under low temperature (-18ºC) and when the films are soaked in water. It is recommended to include in the prEN 17033 draft and the EN13655 standard these two additional conditions in testing the tensile properties of bio-based and conventional mulching films, respectively.

The evolution of the tensile properties of the mulching films with the exposure time at the QUV accelerated weathering tester shows a decrease of the tensile strength values of the bio-based films that approach asymptotically the stress at yield value in TD in different periods of exposure. The evolution of the elongation at break of the mulching films with the exposure time is drastically reduced over the first 2 cycles, especially in MD, reaching the criterion of ageing (reduction to 50% of the initial εbr value) in analogous but much shorter time, to those recorded for the tensile strength. The conventional LLDPE film εbr value experiences only a small reduction but this film is not biodegradable.

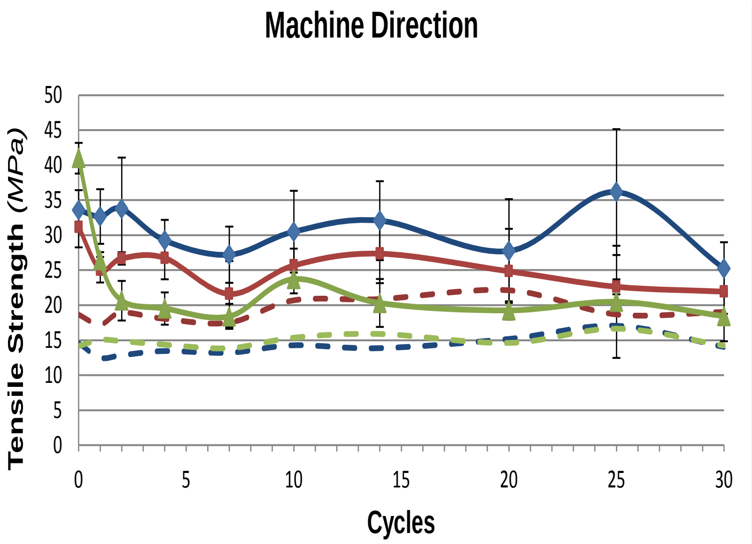

Figure 4. Evolution of tensile properties of bio-based and reference mulching films at the QUV accelerated weathering tester.

The EN13655 standard classifies the duration of a conventional mulching film based on testing the decrease of the tensile strain at break under artificial weathering. It is recommended to include a section on the artificial weathering exposure of bio-based films also in the draft standard prEN 17033.

Tear propagation resistance: The mulching films were tested by the Elmendorf method. The tear propagation resistance of the thicker LLDPE film is shown to be higher as compared to the resistance of the two bio-based films. This is confirmed by the reported MDS values. The measurement of the tear propagation resistance of these very thin films by the Elmendorf tear tester was found to be difficult with high deviations and questionable reliability. The standard itself confirms that high standard deviations and discrepancies have been reported with the tear propagation resistance measurements. It is recommended that the Elmendorf method results should be considered with caution and possibly evaluated as “not good or misleading”. The use of the Elmendorf method is in general not recommended for thin mulching films.

The penetration resistance measured shows one-peak behaviour for LLDPE films and two-peak curves in the case of the bio-based mulching films. The penetration resistance values of the thin bio-based films, calculated based on the maximum load, are shown to be comparable, but lower than that of the thicker LLDPE film. It is proposed that “penetration resistance” is defined as the maximum load, whether this occurs in the first or the second peak in cases of two peak curves. Likewise, Energy for penetration shall be defined as the Energy at maximum load.

The impact resistance of the mulching films appears to be much higher for the thin bio-based films as compared to the conventional LLDPE film resistance. The bio-based films satisfy the impact resistance requirements of the prEN 17033 draft standard specifications.

Radiometric: The PAR transmittance values of the bio-based and reference mulching films satisfy the relative light transmission requirement set by the prEN 17033 draft (≤3%) and the EN13655 standard (≤1%), respectively (there is a mistake in the units in the EN13655 standard, indicating 0.01%). The relative light transmission is suggested by prEN 17033 draft and the EN13655 standard to be measured by a lux-meter over a portion of the visible radiation spectrum, 500-600 nm, and not over the whole visible spectrum (380-780 nm), or the most important for plants (and weeds) PAR spectrum (400-700 nm) as required by the ASTM D1003 standard. As the most important spectrum for the photosynthetic organisms, and so for the weeds development, is the whole PAR (or visible) radiation spectrum, it is recommended that the method of the prEN 17033 draft (Annex A) for measurement of light transmission values is replaced by the ASTM D1003 provisions or equivalent.

The water vapour transmittance rate (WVTR) values of the bio-based mulching films exceed significantly the corresponding value of the conventional reference LLDPE film. A disagreement is observed between the WVTR values obtained by the two laboratories following the same ASTM E96 / E96M – 14 standard testing method but different approaches to achieve the required testing conditions. These discrepancies may be attributed to the different approaches used in meeting the testing conditions. To avoid major discrepancies among the reported WVTR values by various laboratories using the same standard (e.g. ASTM E96 /

E96M – 14) it is recommended that the standard provides for the same approach to be used in achieving the targeted testing conditions. As the WVTR rates depend very much on temperature and possibly on the partial pressure difference of WV across the film, it is also recommended that the same standard testing methods at pre-defined standard testing conditions are followed by the manufacturers (in addition to other conditions tested for specific applications) to measure and report the WVTR values, allowing for the comparison between the reported WVTR values of different products.

8 Adhesives and binders

8.1 Introdcution

Adhesives and binders are a very broad product category and specific target (bio-based) products have been identified based on importance for the industry, novelty and identified hurdles. With the replacement of traditional alkyd paints to water-borne systems bio-based binders were replaced by petrochemical binders. Recently water-borne paints with novel bio-based binders have been introduced into the market

8.2 Approach

A bio-based paint and a traditional oil based paint with the same color and for the same application were tested for mechanical and physical properties. Standard testing methods for paints were used and focus was on paint drying, hardness, scurb resistance, cleanability, viscosity and water uptake.

8.3 Summary

Tests done according to the pendulum damping test (NEN-EN-ISO 1522) show that both paints are hard after 16 hours drying. Results of paint drying according to ISO 9117-4 show no difference between the bio-based or the oil-based paint. The top of both paints is completely dried after 17-18 minutes. This test is a surface test, it means that the bulk of the paint can still be wet but this cannot be determined here.

Scrub tests done with a home-made setup different to the one described in ASTM D2486 show that bio-based paint has good adhesion to the glass plate and scrubs less than the oil-based paint, however, weight loss after scrubbing could not be accurately determined with the method used. Standards containing very specific equipment can represent a hurdle for the introduction of bio-based products in the market, since for qualification and comparison with other materials large investments are needed.

Figure 5 Scrub resistance results for both paints tested.

ASTM D2369 method determines the amount of water (or dry solids) present in the paint. This test does not provide any information on other substances, gases released during the water evaporation. However, values on volatile % are important when mechanical properties of the paints are going to be compared and therefore they should be reported by the supplier.

Both paints have similar viscosity, it is not expected that this property will represent a hurdle for introduction in the market. Viscosity is not a crucial parameter since both paints are water based and water can be added to obtain lower viscosities.

Effect of household chemicals was evaluated by using ASTM D1308. Measurements were done in glass plates, it is unknown if when the paint is used in other substrate, the same effect will be observed. When discoloration is observed there is no difference between the start and no difference is observed between both paints. The forming of blisters differs, sometimes it is more for the bio-based and others for the oil-based, depending on the reagent. It is clear that the bio-based form is more water sensitive and it absorbs water because of the presence of starch on it. However, this could not be confirmed with the water uptake measurements since the method used was not sensitive enough for dried paints.

9 Solvents

9.1 Introduction

A solvent is a substance can dissolve another liquid, a solid or a gas. Solvents are usually liquids but can also be a solids or a gasses. There are many different uses for solvents and also the nature of the solvent varies depending on the application. The largest application segment for solvents is in paints and coatings (60% of the total volume). Other important commercial uses include dry cleaning (e.g., tetrachloroethylene), glue solvents and nail polish removers (acetone, methyl acetate, ethyl acetate), spot removers (e.g., hexane, petrol ether), detergents (citrus terpenes) and perfumes (ethanol). There is an increasing interest in bio-based solvents. Drivers are the increase of the oil price, the low carbon footprint of renewable chemicals and the potential of bio-based solvents to be safer, more readily biodegradable and less toxic. Within the solvents community there is certainly a need for a set of technical parameters to characterize a pure solvent and identify its potential uses. Especially for bio-based solvents that aim to replace existent, well known petro-based solvents, a set of characteristics to determine its functionality can be crucial in its successful introduction in the market.

9.2 Approach

Three bio-based solvents (lactate esters) were selected for testing against common solvents two commonly used relatively toxic petro-based materials. Focus was on common properties like volatility, colour and water content

9.3 Results and recommendations.

We have shown here that ASTM-D6304 is a suitable method to evaluate the water content not only on petroleum based products but also in bio-based solvents as the ones based on lactic acid presented here.

Hansen solubility parameters are crucial for the introduction of bio-based solvents in the market, its knowledge and widespread can be helpful to make them of common use.

There are several ways of characterizing thermal properties of solvents, here we have shown that ISO 4626:1980 is a suitable method to determine boiling points if the values are not too high such as for Galasolv NV300 (above 300°C). And that TGA curves give already a first impression on the volatility of the solvents and the temperatures of complete and 50% evaporation. This is certainly a valuable method.

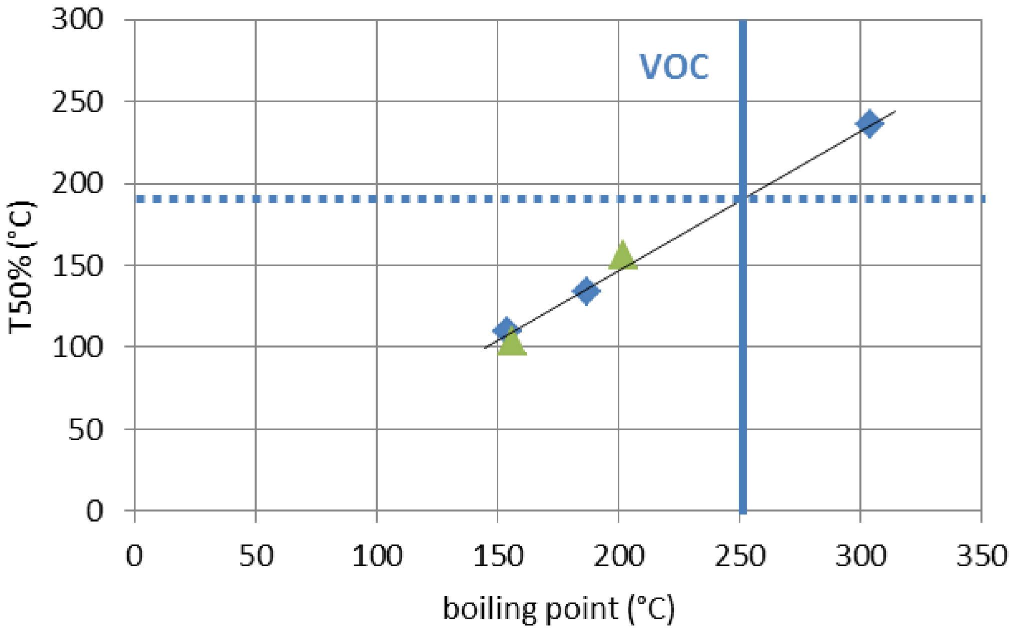

Figure 6 Correlation between T50% estimated from TGA measurements and boiling points of three bio-based solvents and two reference materials. Literature data on boiling points was used

We did not find a suitable way to determine acidity in esters. However it is not clear what will be the added value if for example Hansen parameters are known.

ISO 6271 as used here is a suitable method to determine color of bio-based solvents. However, probably a time factor or an exposure factor has to be included in product specifications or a warning that color might change with time.