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**Sustainpack**

**Innovation and sustainable Development in the Fibre Based Packaging Value Chain**

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**D5.36. Report on the food contact application of composites and cushioning materials<sup>i</sup>.**

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**Report on the food contact application of composites and cushioning materials**

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<b>Dissemination Level</b>		
<b>PU</b>	Public	<b>X</b>
<b>PP</b>	Restricted to other programme participants (including the Commission Services)	
<b>RE</b>	Restricted to a group specified by the consortium (including the Commission Services)	
<b>CO</b>	Confidential, only for members of the consortium (including the Commission Services)	

## **Objective**

Description of the food contact application of composites materials.

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## 1) INTRODUCTION

European legislation on food contact materials and articles aims to keep food safe by requiring that materials and articles do not transfer their constituents into food so as to endanger consumer health or affect food quality.

EU legislation includes the Framework Regulation (EC) N. 1935/2004 of the European Parliament and of the Council and a certain number of Directives that can be *Specific Directives*, applicable to one group of materials and articles, or *Individual Directives* relating to individual substances.

EU legislation does not regulate composite materials made of plastics and other materials for food-contact use, neither do national regulations.

## 2) EVALUATION OF NEW FOOD CONTACT MATERIALS

Evaluation of food contact materials involves legal, regulatory, scientific and technical considerations.

When a material or article does not have its own EU Directive or is explicitly excluded from the scope of a specific Directive, then it must still meet the general requirements of the Framework Regulation. The procedure to analyse this type of materials must prove they comply with the general requirements applicable to all materials and articles: “purity” of foodstuffs, “inertness” of the materials and articles in food contact, “good manufacturing practice”.

This means that in normal use they will not transfer their constituents to food in quantities which could endanger human health or cause unacceptable changes in the composition of food or a deterioration in its organoleptic properties.

When a material not covered by specific (EU or national) legislation is produced, in order to have compliance with the general requirements it is necessary to prove that excessive migration of components from the packaging into the food is prevented (Art. 3 Reg.1935/2004/EC) and to assure that the packaging will not cause organoleptic changes to the food and will be free of contamination.

For most composite materials EU legislation is still missing so there are no clear rules to follow and you have to pay attention in the choice of raw materials, finishing agents and production techniques.

The best way to deal with this problem is:

- to verify the compliance with food contact legislation of each component, so that you can ensure you have taken all possible measure to control the composition of the new material;
- to ensure that all materials are stable and compatible so that unintended chemical changes or reactions cannot take place during the production stage;
- to check that the level of possible residuals will not give rise to unacceptable changes in the food.

Moreover you must consider the required performance of the packaging material, which is correlated to the nature of the product you want to pack, to its shelf life, and to the processes (heating, cooling, sterilisation etc.) to which the packaging material and the content may be exposed.

### 3) IDENTIFICATION OF COMPONENTS

PLLA, poly(L-lactide), Polyester Mater-Bi and Starch Mater-Bi were chosen by WP 5.2 as matrices for the composites preparation together with cellulose. Three types of celluloses were used:

- BleachsoftDRF;
- MixBleachsofthardDRF;
- HF hornified reference.

### 4) CHECK OF THE COMPONENTS

**Lactic acid** (CAS 000050-21-5) is present in Commission Directive 2002/72/EC, Annex II “List of monomers and other starting substances which may be used in the manufacture of plastic materials and articles”- Section A “List of authorised monomers and other starting substances”. The monomer is present with no restrictions and/or specifications.

**Polyester Mater-Bi** and **Starch Mater-Bi** are products obtained by forming complexes between the amylose and natural or synthetic molecules. These complexes create a new crystalline order, which increases the water-resistance and changes the mechanical properties of the original starch, without modifying its chemical structure. Esters and starch are present in the positive lists of Commission Directive 2002/72/EC.

**Cellulose** derived from unrecycled wood or cotton is the starting material for regenerated cellulose film (Commission Directive 93/10/EEC of 15 March 1993 relating to materials and articles made of regenerated cellulose film intended to come into contact with foodstuffs).

Cellulose is also present in the positive list of the Italian legislation for paper and board for foodstuffs (D.M. 21/3/73 Annex II- Section 4 Part A), and in German Recommendation XXXVI. “Paper and Board for Food Contact”.

Not recycled cellulose is present in the French “Index alphabétique des matériaux pour contact alimentaire et leurs constituants” Brochure n°1227/2002.

## 5) COMPOSITE SPECIMENS

The following eight composite specimens were delivered by WP 5.2 together with the corresponding matrices.

<u>COMPOSITES</u>	<u>MATRICES</u>
Polyester Mater-Bi (10 WT%)+BleachsoftDRF Polyester Mater-Bi (10 WT%)+ MixBleachsoftDRF	Polyester Mater-Bi NOVAMONT
PLLA (10 WT%)+ BleachsoftDRF PLLA (10 WT%)+ MixBleachsofthardDRF	PLLA BIOMER
Starch Mater-Bi (10 WT%)+ BleachsoftDRF Starch Mater-Bi (10WT%)+ MixBleachsofthardDRF	Starch Mater-Bi NOVAMONT
PLLA + 30% HF reference PLLA + 30% HF hornified reference	PLLA BIOMER

## 6) OVERALL MIGRATION TESTS

Overall migration testing is intended to simulate the conditions of package use and consists of two parts: the exposure of the test specimen to the food simulant and the determination of the substances in the food simulant.

### **Exposure.**

In the choice of exposure conditions (time and Temperature) it must be considered that tests performed with short times and high temperatures of exposure will have a lower precision than tests performed with long times and low temperatures. In fact there are many difficulties in controlling exposure conditions, particularly temperature; high temperature tests performed with exposure times less than 24 h need special care to control the heating-up and cooling-down stages of the exposure of the specimen to the hot simulant.

### **Determination of substances.**

Determination of substances from overall migration tests differs depending on the simulant used.

In the overall migration test with the three aqueous simulants (distilled water, 3 % w/v acetic acid aqueous solution and 10 % v/v ethanol aqueous solution) the simulant is evaporated to dryness and the migrated substances are measured by weighing the residue. In this case the overall migration test determines only the non-volatile substances migrated into the food simulant.

In the overall migration test with fat simulant (olive oil) the migrated substances are determined by measuring the loss in mass of the test specimen after exposure to the simulant. As the test specimen absorbs and retains some of the fat simulant at the end of the exposure, this has to be determined separately and subtracted from the final mass of the test specimen. This test measures the migration of volatile and non-volatile substances.

Substitute tests which use the test media (iso-octane and 95% ethanol) under the conventional substitute test conditions, as set out in Directive 97/48 EC, are carried out if the migration test using the fatty food simulants is not feasible for technical reasons connected with the method of analysis. In these tests the simulant is evaporated to dryness and the migrated substances are measured by weighing the residue, so the non-volatile substances are only determined.

Migration tests were performed exposing the specimens to food-simulating solvents under conditions of temperature and time reproducing the most severe conditions to which food in contact with composites would be exposed.

### **Materials and Methods.**

Preliminary migration tests were carried out on PLLA, Polyester Mater-Bi and Starch Mater-Bi samples supplied by University of Girona. The following migration conditions were used:

- temperature = 40°C
- time = 10 days
- surface/volume ratio = 2
- food-simulating solvents: 3% acetic acid, oil and 95% ethanol.

Each test was carried out in duplicate; for acetic acid and ethanol blank values were considered.

For each specimen and for each food-simulant 32 cm<sup>2</sup> were placed in a flask, 16 ml of food-simulating solvent at 40°C were added and the flask was placed in an oven at 40°C. After 10 days the flasks were removed from the oven and the specimens removed from the flasks.

Acetic acid and ethanol extracts were evaporated to dryness and weighed. The specimen in contact with oil was extracted with 1,1,2 trichlorotrifluoro-ethane and then weighed. The amount of migrated substances was calculate by difference between the initial weight and the weight after extraction.

### Migration Results.

#### MIGRATION CONDITIONS:

- T = 40° C
- t = 10
- surface / volume ratio = 2

#### FOOD SIMULANTS:

- ACETIC ACID 3% w/v
- ETHANOL 95%
- OLIVE OIL

SAMPLE	ACETIC ACID 3% MIGRATION (mg/dm <sup>2</sup> )	ETHANOL 95% MIGRATION (mg/dm <sup>2</sup> )	OLIVE OIL MIGRATION (mg/dm <sup>2</sup> )
Polyester Mater-Bi NOVAMONT	7.59	206.65	*
P-esterMater-Bi (10 T%)+BleachsoftDRF	9.60	201.13	*
Polyester Mater-Bi (10 WT%)+ MixBleachsoftDRF	12.50	203.02	*
PLLA BIOMER	0	0	0.16
PLLA (10 WT%)+ BleachsoftDRF	*	*	*
PLLA (10 WT%)+ MixBleachsofthardDRF	2.49	0	**
Starch Mater-Bi NOVAMONT	1611.37	1088.92	*
Starch Mater-Bi (10 WT%)+ BleachsoftDRF	1035.21	3395.66	*
Starch Mater-Bi (10WT%)+ MixBleachsofthardDRF	*	*	*
PLLA BIOMER	0	0	0.16
PLLA + 30% HF reference	21.48	2.99	**
PLLA + 30% HF hornified reference	24.45	11.54	**

\* Analysis in progress

\*\* We couldn't remove the oil from the sample by solvent extraction, so it is not possible to have a migration value

Migration conditions ( $T = 40^{\circ}\text{C}$  and  $t = 10$  days) were selected assuming the following conditions of contact in actual use:

- contact time  $> 24$  h
- contact temperature  $20^{\circ}\text{C} < T \leq 40^{\circ}\text{C}$ .

The specimens were not in quantity enough to carry out the overall migration test with all six food simulants so we choose acetic acid 3%, ethanol 95% and olive oil.

## **CONCLUSIONS**

The results obtained up to now show migration values for composites in some cases higher than the respective matrices alone.

## 8) REFERENCES

A practical guide for users of European Directives.

EUROPEAN COMMISSION HEALTH & CONSUMER PROTECTION DIRECTORATE-GENERAL

*Updated to 15 April 2003*

82/711/EEC Council Directive of 18 October 1982 laying down the basic rules necessary for testing migration of the constituents of plastic materials and articles intended to come into contact with foodstuffs.

85/572/EEC Council Directive of 19 December 1985 laying down the list of simulants to be used for testing migration of constituents of plastic materials and articles intended to come into contact with foodstuffs.

93/08/EEC Commission Directive of 15 March 1993 amending Council Directive 82/711/EEC laying down the basic rules necessary for testing migration of the constituents of plastics materials and articles intended to come into contact with foodstuffs.

97/48/EC Commission Directive of 29 July 1997 amending for second time Council Directive 82/711/EEC laying down the basic rules necessary for testing migration of the constituents of plastics materials and articles intended to come into contact with foodstuffs.

2002/72/EC Commission Directive of 6 August 2002 relating to plastics materials and articles intended to come into contact with foodstuffs.

1935/2004/EC Regulation (EC) No 1935/2004 of the European Parliament and of the Council of 27 October 2004 on materials and articles intended to come into contact with food and repealing Directives 80/590/EEC and 89/109/EEC.

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<sup>i</sup> **Note.** This report contains the results of the tests carried out in order to evaluate the possible food contact application of composites materials. No cushioning materials were available for these tests.