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**Sustainpack**

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

Instrument: **IP**

**D5.51. Particle size distribution in selected composite samples**

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Dissemination Level		
<b>PU</b>	Public	X
<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	
<b>CO</b>	Confidential, only for members of the consortium (including the Commission Services)	

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Revision [0]

## 1 Summary

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During extrusion of filled polymers substantial degradation of the filling material can occur. The degree of degradation occurring during compounding and extrusion will influence key characters of the composite material such as mechanical and hygro-dynamic properties. In Sustainpack attempts to use different kinds of modified pulp fibres as reinforcement of bio-degradable polymers are tested. The level of fiber degradation has been studied using a Malvern mastersizer equipment. The tested samples were produced in Girona University (UDG), Spain, using different fibres that were developed and produced by the partners in SP5.

## 2 Introduction

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The use of organic fillers in polymeric materials such as PP and PE has mainly been done to substitute relatively expensive polymer with scrap wood. The filler is then normally introduced into the polymer as a powder. The addition of a powder such as wood flour into the polymer will decrease important properties. New regulations concerning the reuse of materials has created a need for a new type of fillers able to both fulfil re-cyclability demands and give the finished composite improved properties. Typically one is looking for alternatives to glass fibres.

Pulp fibres inherently have many attractive properties such as high specific mechanical properties. They also have a high aspect ration and can theoretically give the finished product good properties. However, extensive shearing during compounding and extrusion will degrade the fibres and therefore reduce the potential improvements attainable.

We therefore wanted to investigate the size of the filler in produced test materials.

### 3 Experimental

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The investigated composites were produced using Fibres supplied within SustainPack. As matrix PLLA and two qualities of MaterBi (starch and ester) were used. The following composites were tested:

#### **Material**

Between 0.5 g and 4 g of material, depending on the density of the composites, were put into 10 mL centrifuge tubes. The tubes were thereafter filled with dichloromethane (DKM) and the composites were let to dissolve over night. The samples were subsequently centrifuged for 5 to 10 minutes in 3 000 x *g* in a bench centrifuge or in the highest possible speed on the lab centrifuge. The supernatants were poured off and discarded. Two additional washes with DKM followed until the supernatants were seemingly free from the composite matrices. The achieved fibres were centrifuged and thereafter let to dry in the tubes to remove any excess DKM.

Before particle size measurements the fibres were mixed in 1 - 2 mL deionised water by magnetic stirring to achieve a homogenous slurry.

#### **Particle size measurements**

Optical microscopy was performed on the samples to get an initial overview of the degree of degradation of the fibres.

The particle size was measured by laser diffraction using Malvern Mastersizer ms20 (Malvern Instruments, Worcestershire, United Kingdom). The 300 mm lens was utilised detecting particles in the range of 1.2  $\mu\text{m}$  to 600  $\mu\text{m}$ . A polydisperse model was used with the settings for standard particles and with MilliQ water as carrier fluid. Samples were filled in the MSX3 stirred cell unit up to an obscuration of about 15%, indicated by green colour in the software. The mass median diameter (MMS) for each sample was used as particle-size aspect.

#### **Verification of the laser diffraction method**

For verification of the Malvern method, three samples were sent for manual image analysis. The length, mean width and area for around 320 particles were recorded, and the length median value was used in the comparison of the MMS obtained by laser diffraction.

## 4 Results and discussion

PLLA show good solubility in methylene chloride enabling easy extraction of fibres from extruded samples. It was also possible to extract particles from MaterBi/polyester samples. However, despite repeated attempts to extract particles from MaterBi/starch we were not able to collect material free from matrix. In figure 1 the results from Malvern measurements of selected samples are shown. The results in figure 1 clearly indicate that considerable degradation of fibre geometry takes place during the compounding/injection moulding process.

The instrument (Malvern) used is really intended to measure the size of particulate materials. Thus the instrument is calibrated using spherical particles and the diffraction pattern created by the sample is compared to the pattern determined for spherical particles. In an attempt to see to which degree the measurements from the Malvern differed from manual determination, three samples were investigated using both techniques, figure 2. The values given as  $d(0,5)$  means that 50 % of measured fibers/particles were smaller than this value. Thus  $d(0,1)$  and  $d(0,9)$  refers to 10 and 90 % respectively.

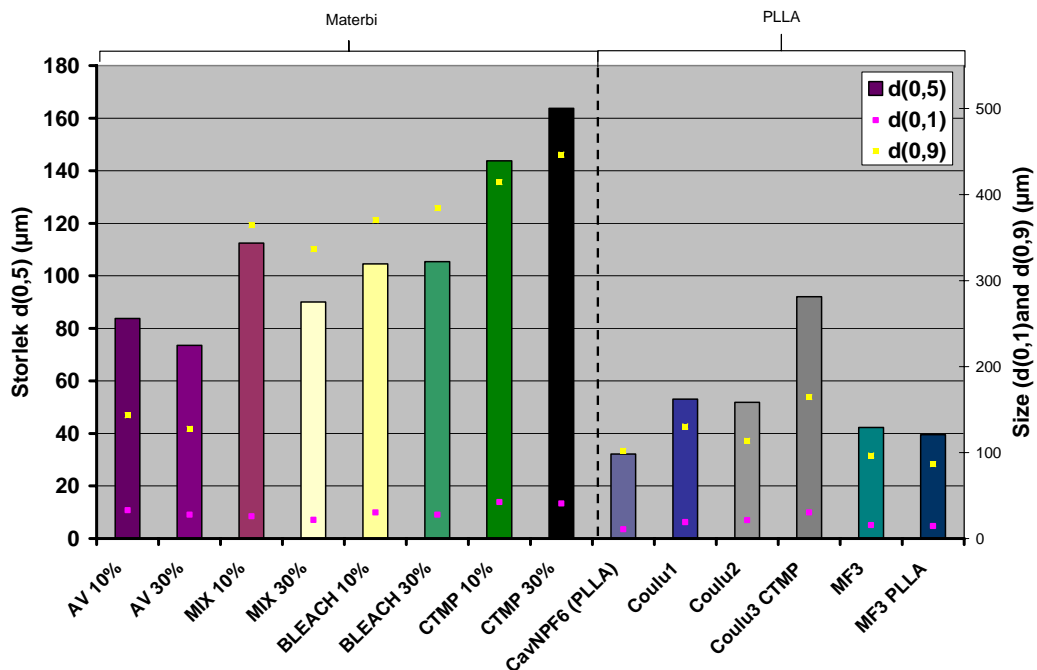


Figure 1. Determined particle size using Malvern. To the left results from MaterBi/polyester samples are shown and to the right results from PLLA samples are shown.

It is clear from the comparison that the difference in size determined using the two techniques is quite large. However, based on the result it seems possible to use the Malvern technique to compare samples and observe relative size differences. The fact that manual determination does not discriminate large particles in all likelihood is the main explanation to the decreasing size increase observed on going from sample 1 to sample 3.

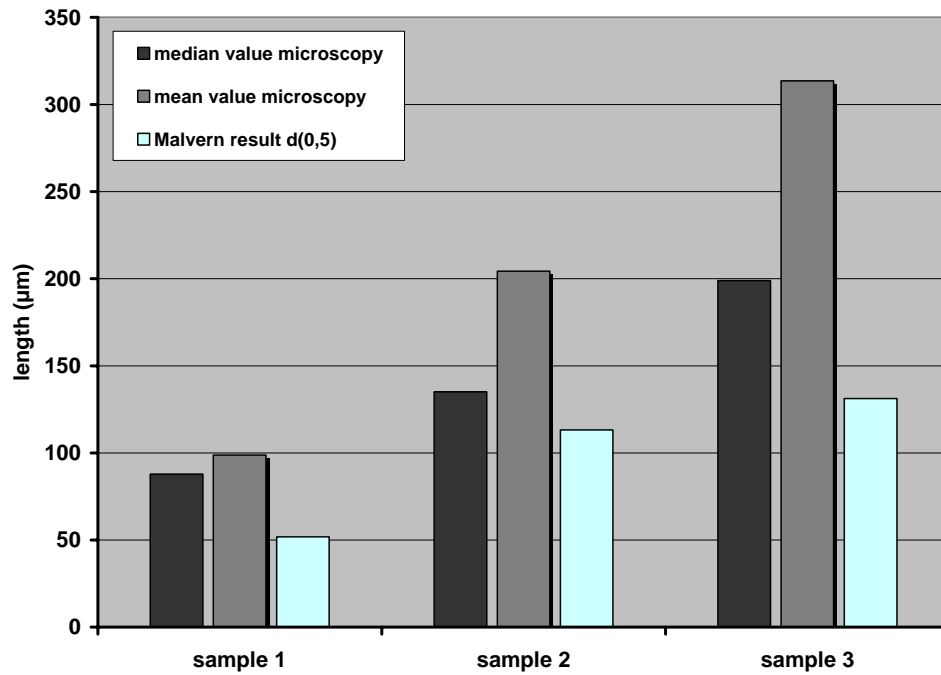


Figure 2. Comparison of determined particle size using Malvern and manual characterization.