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STRUCTURAL CHARACTERISATION OF THE FIRST PROTOTYPE BIAXIAL FIBRE SEMI-PRODUCT

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PP LEADER UoPortsmouth

European Regional Development Fund

Partners

PP Leader: UoPortsmouth

Partners involved: UBS, INRA

Content

❖ Objectives

The objective of this deliverable is to characterize the architecture of the bi-axial semi-finished products developed by TV. Observation methods (electron microscopy, optical microscopy and nano-tomography) will be used to better understand the structural organization of the preforms as well as to quantify the defects induced by the reinforcement manufacturing process. The samples developed by TV will be compared with commercially available reference samples made of flax and glass fibres.

❖ Materials

Sample	Producer	Fibre	Areal weight (g/sqm)
BX TV 312	TV	Flax	312
BX TV 400	TV	Flax	400
BX SS 600	Safilin	Flax	600
BX TDL 250	Terre de Lin	Flax	250
BX BC 350	B Comp	Flax	350
BX GF 600	Sicomin	Glass	600
TAPE TV	TV	Flax	x

The last sample (TAPE TV) is the flax tape use to manufacture BX TV 312 and BX TV 400

❖ Experiments to do

Sample	SEM (UBS and UoP)	Tomography (UoP)	Analysis of Defects (INRA)
BX TV 312	x	x	x
BX TV 400	x	x	-
BX SS 600	x	x	-
BX TDL 250	x	x	-
BX BC 350	x	x	-
BX GF 600	x	x	-
TAPE TV	-	-	x

❖ Optical microscope and SEM analysis (UoP and UBS):

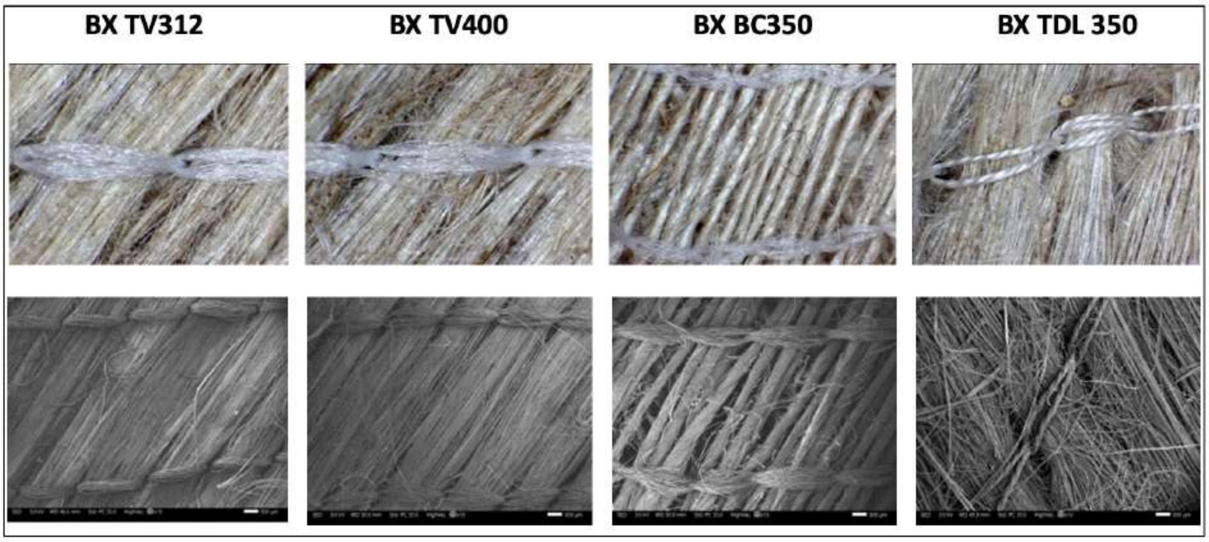

Figure 1 : Optical and SEM observation of BX TV312, BX TV400, BX BC350 and BX TDL 350 at macro scale

These observations provide a good understanding of the macroscopic organization of the reinforcements. The difference with the BComp product, made of much more twisted yarns can be noted. In the same way, the stitching yarn of the Terre de Lin product is also less open and probably more penalizing within a composite.

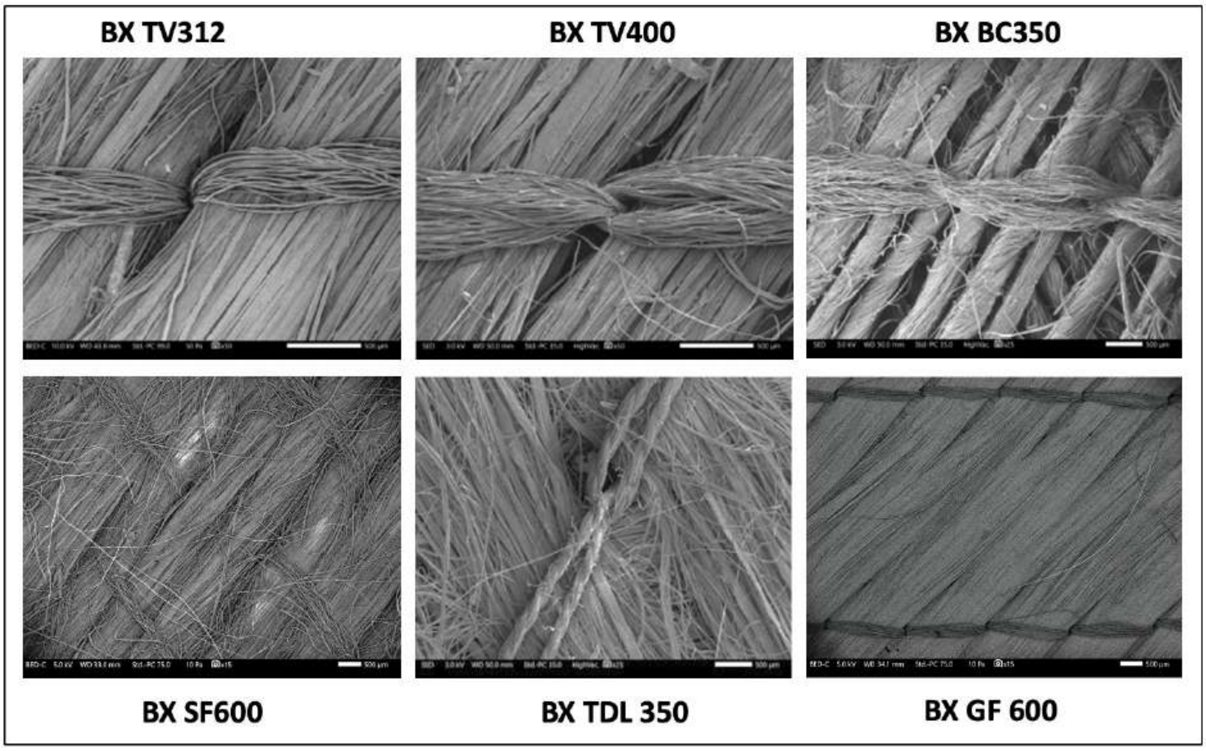

Figure 2 : More detailed optical and SEM observation of BX TV312, BX TV400, BX BC350 and BX TDL 350

The good alignment of flax fibres within TV products is highlighted, despite some local imperfections. One can notice the difference of twisting, especially compared to BComp product. Local disorganisations of glass fibres can also be noticed on BX GF 600.

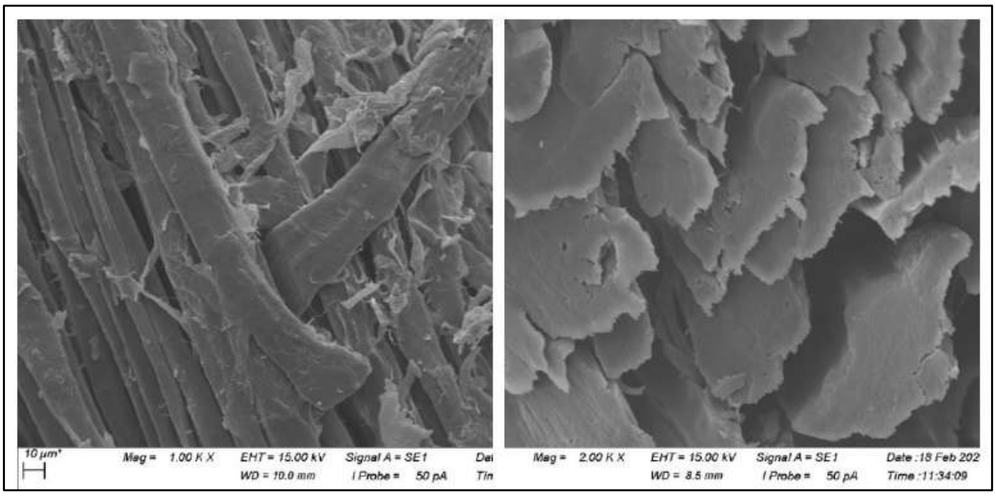

Figure 3 : SEM observation of BX BC350

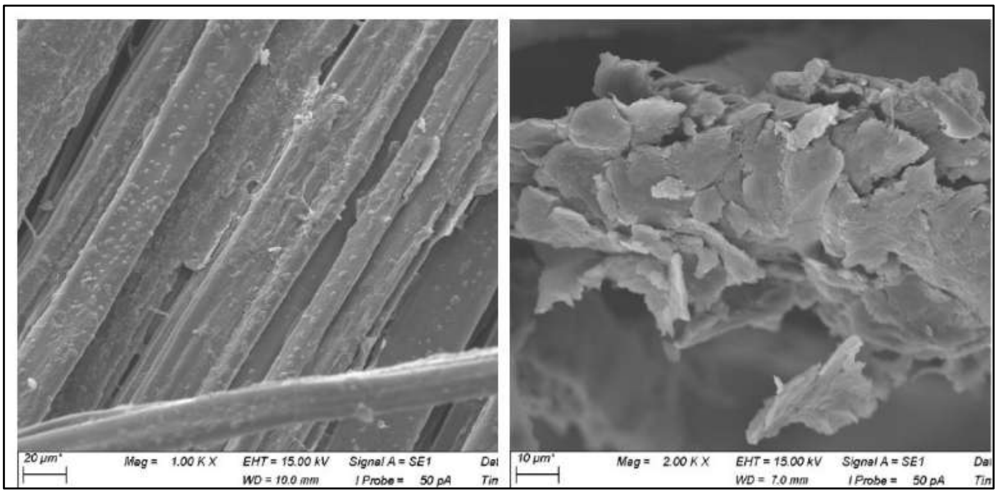

Figure 4 : SEM observation of BX TV312

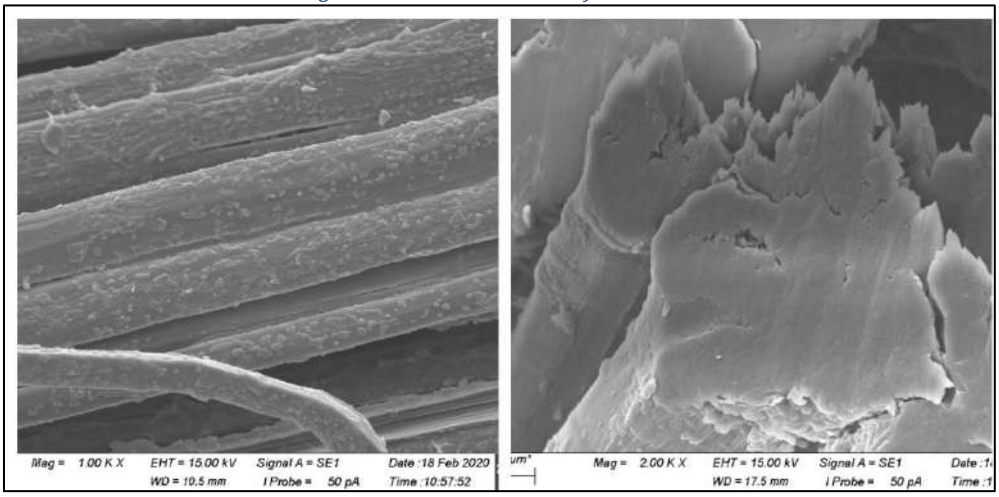

Figure 5 : SEM observation of BX TV400

As one would expect for flax fibres, in sample BX BC350 the diameter of the elementary fibres in a bundle is not uniform showing polygonal cross-section along the fibre length. No visible surface impurities is present in BX BC350 sample whereas for samples BX TV312 & BX TV400 surface does not seem so smooth, impurities are highlighted. In samples BX TV312 & BX TV400 some lumens are also visible at the end of the fibres.

- **Tomographic analysis (UoP):**

As a part of the fibre characterization task, the diameter and distribution of the three types of flax fibres preforms (BX BC350; BX TV400 & BX TV312) were analysed using X-Ray computed tomography (XCT). The fibre samples were scanned with a Zeiss Xradia Versa 510 Microtomography X-Ray Computer Tomography (XCT) system with an isotropic voxel resolution of 2 μm^3 and 10 μm^3.

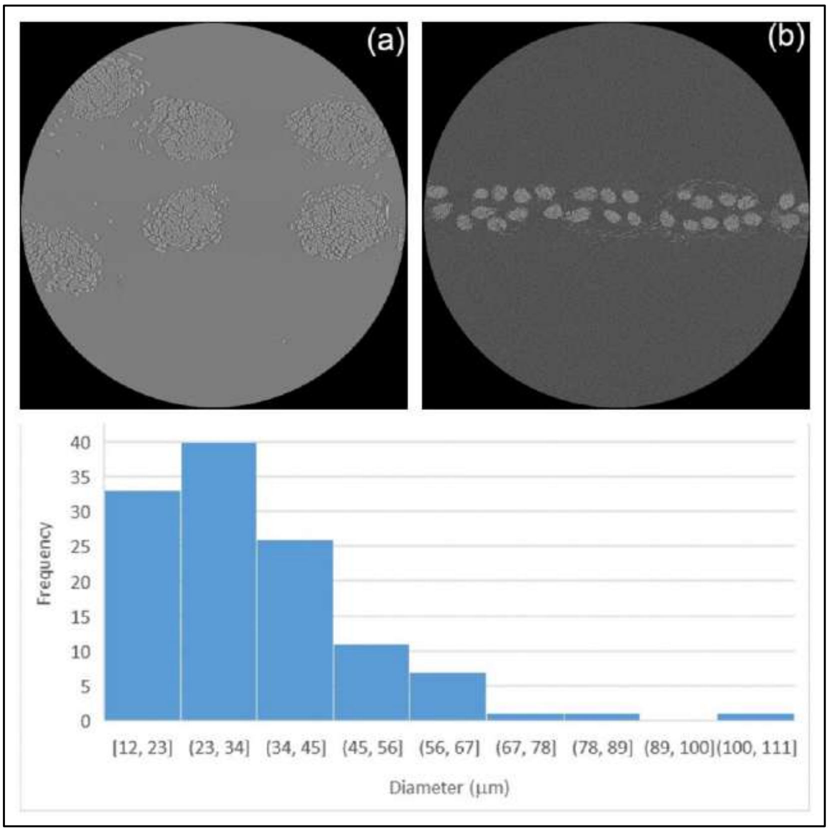

Figure 6: Cross sections of BX BC350 from the XCT image (a) at 2 µm³ voxel size and (b) at 10 µm³ voxel size and histogram of fibre diameter

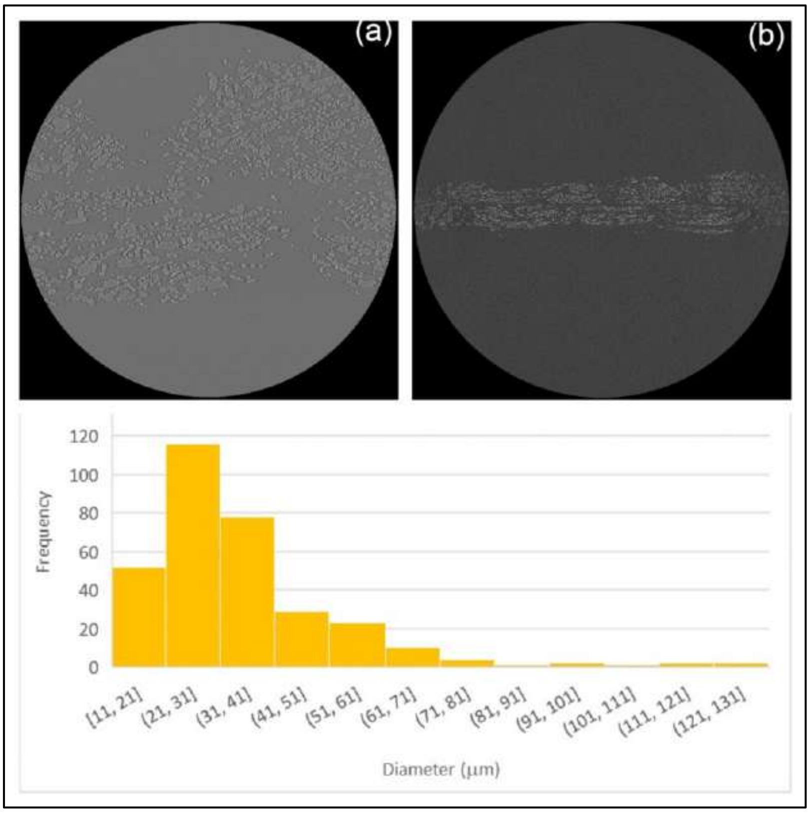

Figure 7: Cross sections of BX TV400 from the XCT image (a) at 2 μm³ voxel size and (b) at 10 μm³ voxel size and histogram of fibre diameter

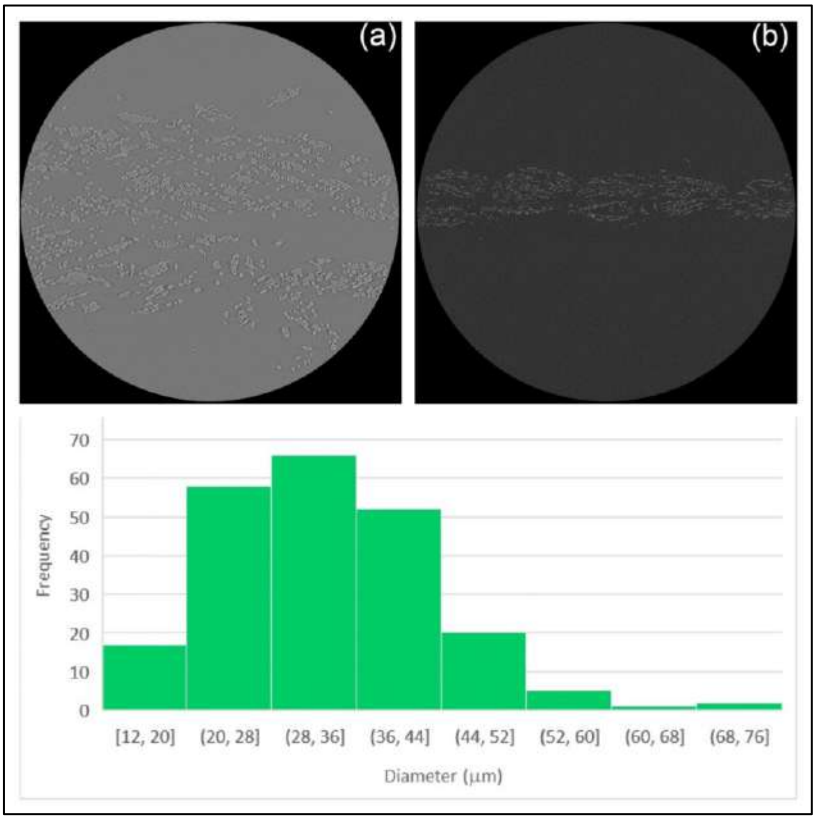

Figure 8: Cross sections of BX TV312 from the XCT image (a) at 2 μm³ voxel size and (b) at 10 μm³ voxel size and histogram of fibre diameter

The object diameters measured on the different preforms show a good individualisation of the fibres, whatever the preform considered, this shows the good initial quality and the level of retting of the fibres selected for the manufacture of the preforms. Concerning the general architecture of the reinforcements, the tomographic analysis shows much more cohesive yarns in the case of BC350 preforms due to the torsion applied to the rovings. In the case of Depestele products, the boundary between the rovings is less visible, the reinforcements are more open, which is probably an advantage for the future impregnation of composites.

- **Thermogravimetric (TGA) characterisation (UoP):**

To complement characterisation obtained from X-ray Micro CT and scanning electron microscopy (SEM), TGA tests were carried on three flax samples namely : BX BC350; BX TV400 and BX TV312, respectively. Some results and observations are illustrated in Figures 9-13. Table 1 summarises the TGA results for three different samples.

Table 1: Results from thermal gravimetrical analysis for three different types of flax fibres

Fibre types	Mass Loss at 100 ºC (%)	1st Thermal Degradation Rate Peak Temperature (ºC)	2nd Thermal Degradation Rate Peak Temperature (ºC)
BXTV312	5.96	349.33	450.33
BXTV424	5.84	347.57	448.13
BXBC350	4.98	352.16	479.81

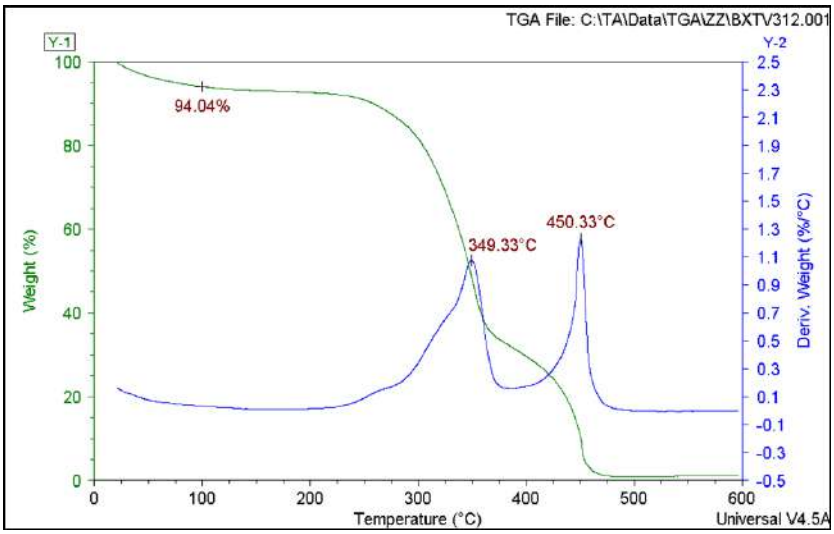

Figure 9: Thermal degradation trace of BX TV312

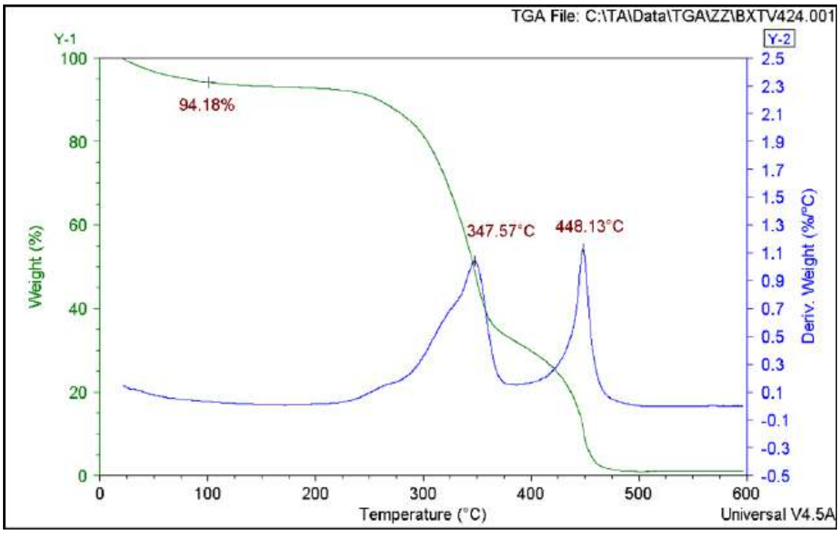

Figure 10: Thermal degradation trace of BX TV4400

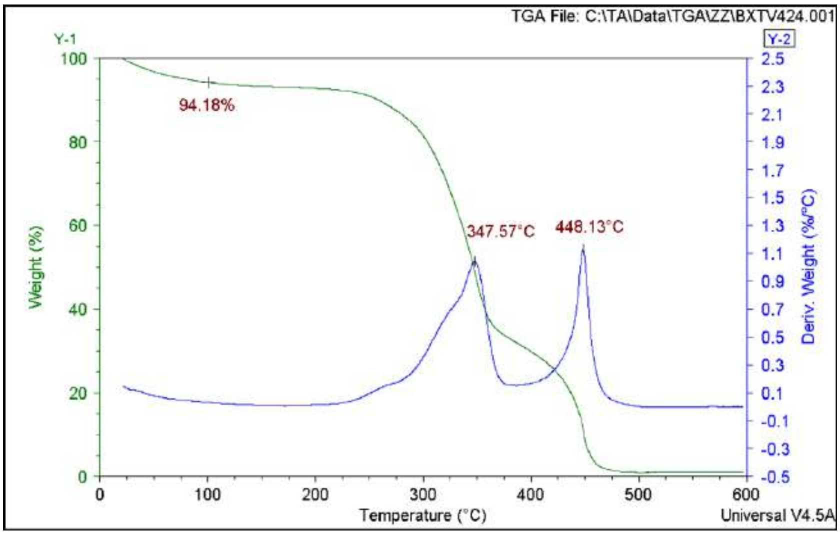

Figure 11: Thermal degradation trace of BX BC350

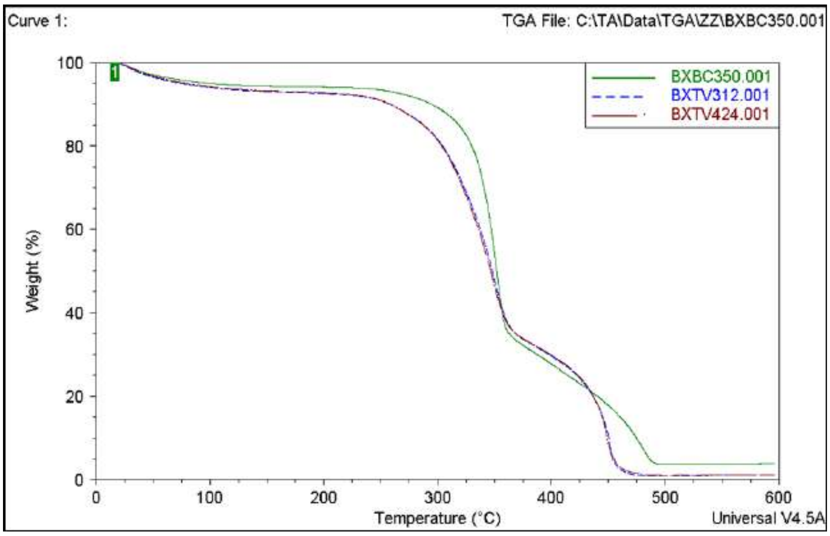

Figure 12: Comparison of mass loss trace of three flax fibres

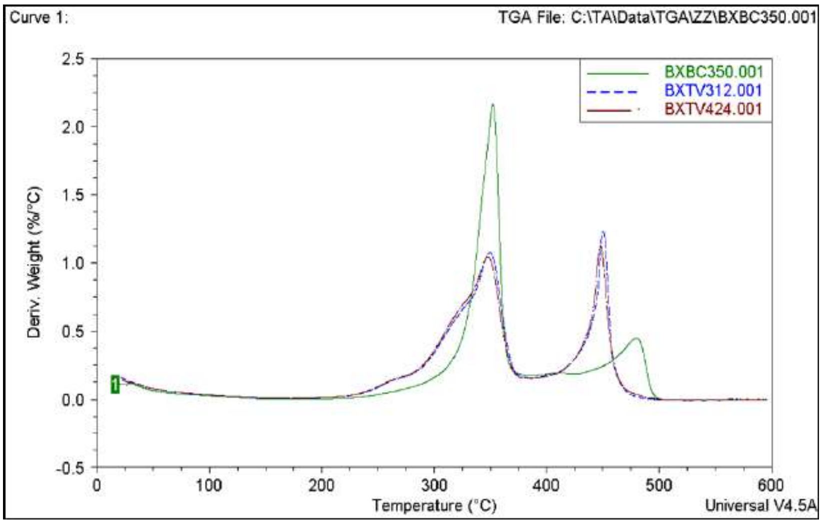

Figure 13: Comparison of the first derivative of mass loss to temperatures of three flax fibres

Key observations from TGA test results

1. The initial weight loss at around 100 °C can be attributed to the inherent moisture in the flax fibres
2. BXTV312 and BXTV400 flax fibres have similar thermal degradation behaviours and they are different from those form BXBC350 flax fibre
3. BXTV312 and BXTV400 flax fibres have lower peak temperatures and balanced magnitude compared with those for BXBC350 flax fibre
4. After approximately 240 °C, the flax fibres start decomposing in a rapid rate.
5. The maximum processing temperature for flax fibres lie around 240 °C.
6. Note: in TGA diagrams, Figures 12 and 13, it should read as BXTV400 instead of BXTV424.

- **Quantification of defects (INRA) TO DO**

Conclusion

The morphology of the biaxial preforms developed in the Flower project was compared with commercially available products. Microscopic analyses showed a good alignment of the flax rovings in the FLOWER products as well as a good level of retting, as evidenced by the fibre division detected in tomography. Additional TGA analyses have shown a usual thermal behavior of flax in FLOWER preforms, which is not always the case for competitors' products, perhaps due to the use of binders or specific sizing treatment. TGA results exhibit that no appreciable weight loss is observed until 240 °C, which indicates good thermal stability of the flax fibres up to such temperature.