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

Instrument: **IP**

D5.38 - Report on the mechanical characterization of composites.

Due date of deliverable: **November 06** (month 30)
Actual submission date: **September 07** (month 40)

Start date of project: **2004-06-01** Duration: **4 years**

Organisation name of lead contractor for this deliverable: **UNIVERSITAT DE GIRONA (UdG)**

Dissemination level: PU

Revision 0

WP5.3
Report on the mechanical characterization of composites

Objective
The objective of this deliverable is to report to the WP partners the results of mechanical characterization of composites obtained in WP5.2 task during last 6 months (from June to November 06).

Results obtained

The composites analyzed are obtained in WP5.2, following the methodology that was described in deliverable D5.10. The mechanical characterization is in accordance to tests described in D5.20. The matrices used to obtain the composites are PLLA, starch and polyester.

The results will be analysed in different blocks.

<u>Block 1. Reinforced matrices using modified fibres from University of Aveiro (UA).</u>

Table 1 summarises the different composites prepared in this block. Matrices used have been PLLA and polyester-Materbi. Modification of fibres has been described in previous deliverables (D5.04; D5.08; D5.29). The percentage of reinforcement is included.

Table 1: Composites based on PLLA/polyester-Materbi and different modified fibres provided by University of Aveiro (UA)

Reference	**Fibrous reinforcement**	**Reinforcement**
P_{PLLA}	PLLA pure	0 %
$C_{AVEIRO}5$	Non modified fibres	30 %
$C_{AVEIRO}NPF6$	Fibres modified with nanoparticles TiO_2	
C_{AVEIRO} 2D	2Delivery Non modified fibres	
C_{AVEIRO} 2D NPF	2nd modification with nanoparticles TiO_2	
$P_{PESTER\_MATERBI}$	Polyester_MATERBI pure	0 %
C_{AVEIRO} Si1	Fibres modified with SiO_2	30 %

The graphic representation of the results corresponding to mechanical properties, under tensile, flexural and impact stresses, of the formulations of composites reinforced with the modified fibres provided by University of Aveiro are showed in figure 1.

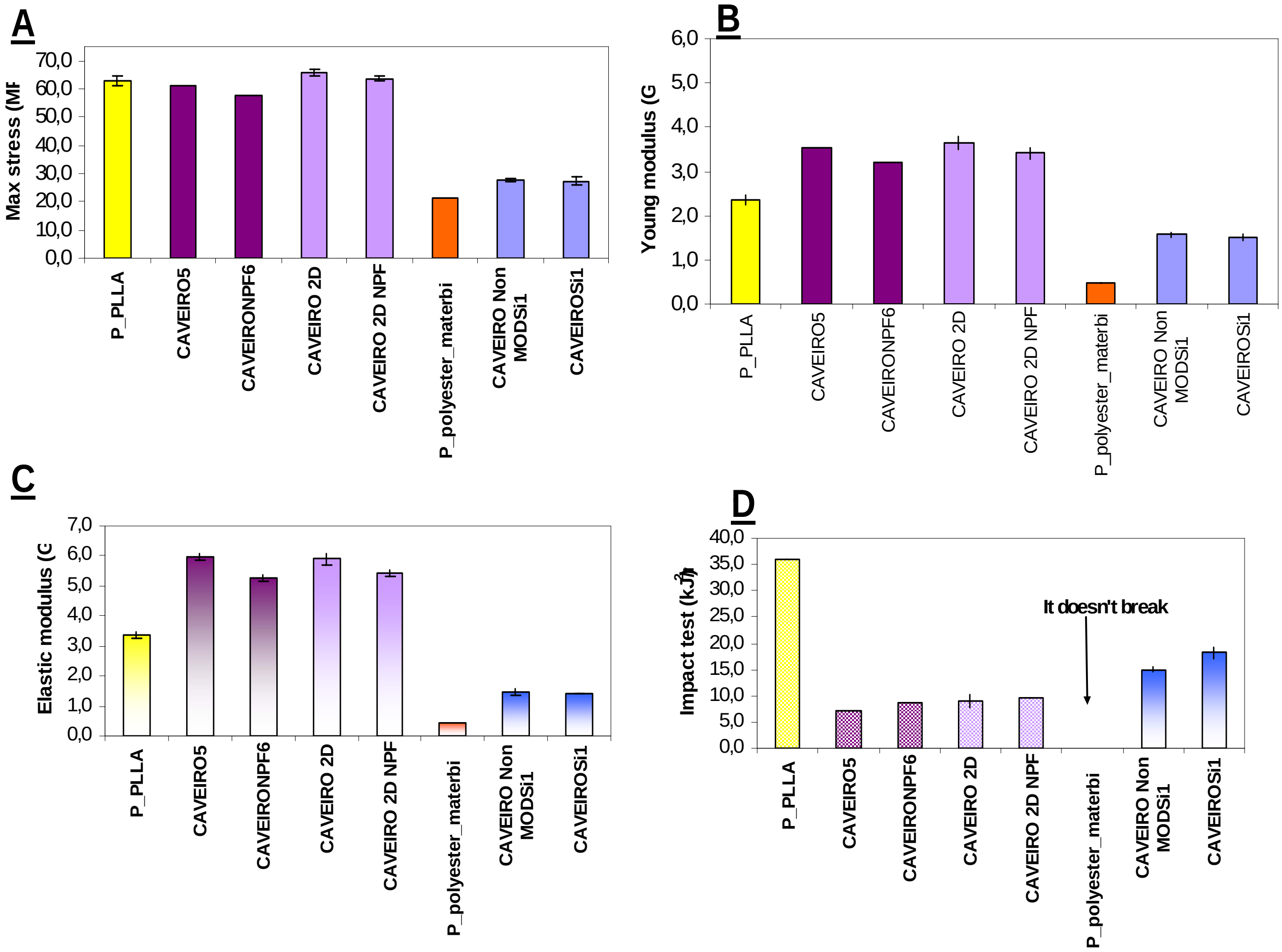

Figure 1: Mechanical properties under tensile, flexural and impact strength of composites reinforced with fibres provided by University of Aveiro. A) Ultimate tensile strength, B) Young Modulus, C) flexural modulus and D) impact strength.

From a point of view of the stiffness of the materials, the addition of the hybrid fibres showed a higher value compared to polymer matrix (PLLA and polyester) but a decrease was observed when the values were compared to that of composites reinforced with non-treated fibres. In the particular case of CAVEIRONPF6, there is a significant decrease of Young and flexural modulus which is less acute for flexural modulus. This effect can be attributed to a hypothetical hindering effect of the inorganic particles that avoid achieving an optimal mechanical anchoring of the matrix in the surface of the fibres. The polyester has a better behaviour, compared to PLLA since if UTS is taken into account, only the formulations based on the polyester increase significantly the values of these properties after fibre addition. From the impact point of view, every addition of fibres produces a decrease in this mechanical property, compared to the value of the plain polymer matrix. But the addition of hybrid fibres produced a significant improvement in this value when they were compared to composites reinforced with non-treated fibres. The presence of the inorganic component improves the resistance against impact stresses allowing an increase in the capacity to be deformed by such stress.

<u>Block 2. Reinforced matrices using modified fibres from STFI-Packsfork (STFI).</u>

The biopolymer used has been PLLA in all the composites corresponding to this block. Fibres used have been modified as it was explained in deliverable D5.25. The different composites that were prepared are summarised in table 2.

Table 2:Composites based on PLLA and different fibres provided by STFI

Reference	**Fibrous reinforcement**	**Reinforcement**
P_{PLLA}	PLLA pure	0 %
HF ref	Reference pulp (Unbleached sulphate pulp)	30 %
HF BTCA grease proof	Grease proof treated fibres proceeding from commercial 50:50 mixture of bleached sulphate and sulphite fibres	
HF BTCA ref	Fibres treated with 1 % butyl tetra carboxylic acid (BTCA)	
HF hornified ref	Hornified dried fibres heated till 125ºC	

The results of the mechanical properties, under tensile, flexural and impact stresses, of the formulations of composites reinforced with the fibres provided by STFI are showed in figure 2.

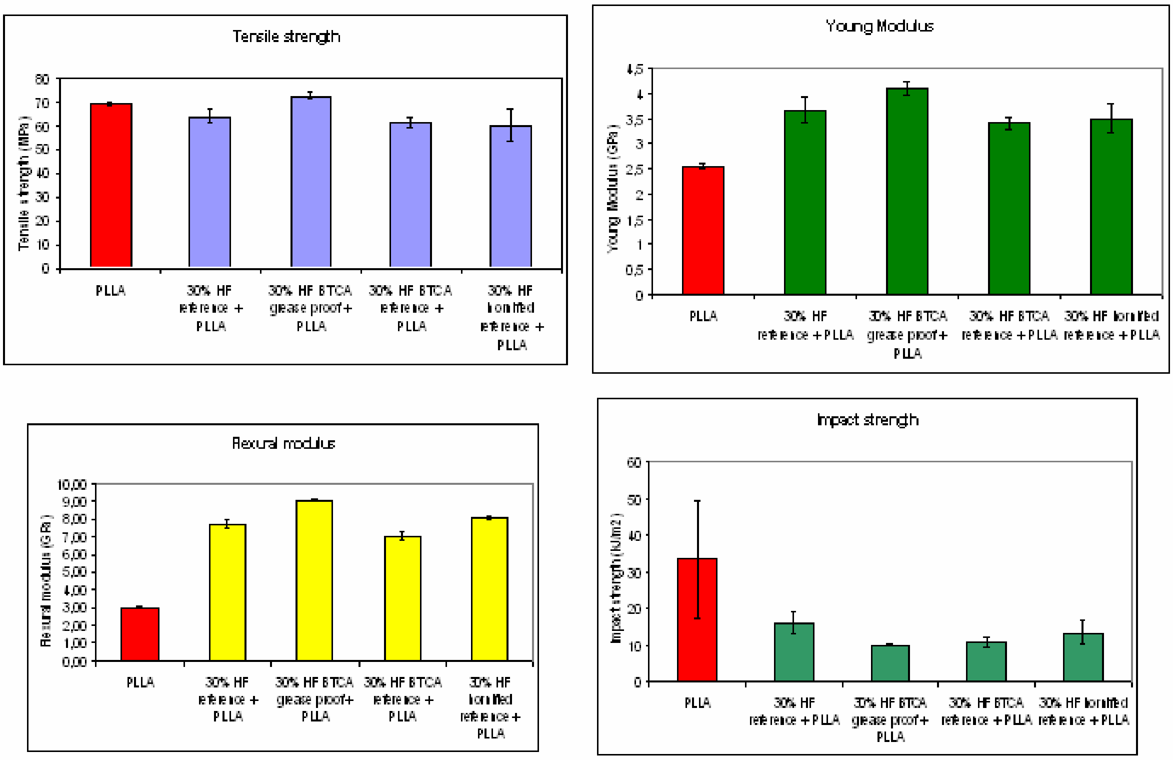

Figure 2: Mechanical properties under tensile, flexural and impact strength of composites reinforced with fibres provided by STFI.

Young modulus increases with the increase of the amount of reinforcement in the composition. In the case of these fibres, higher increases are achieved with fibres based on HF BTCA grease proof treatments.
Taking into account the ultimate tensile strength, this property also undergoes a decrease due to a non good compatibility between both components. To solve this problem, it is possible to add a third component to the composite that compatibilizes the matrix and the fibres at the interphase level.
In the case of the flexural strength, the addition of the fibres produces an important increase in the values, which are double than that of plain PLLA. The obtained composites have a higher capacity to modify their structure under flexural stresses, in other words, they can be deformed under this kind of stress at a higher level compared to PLLA.
Finally, the case of the impact resistance, the values of the composites are lower than that of PLLA. Again, this result is consistent with the presence of fibres that can produce areas were there are concentration of stresses which, in the presence of an impact, a crack can be produced and start its propagation until the mechanical destruction of the material.

Block 3. Reinforced matrices Avicell up to 50 wt% of reinforcement

All the biopolymers have been tested with Avicell fibres till 50% of reinforcement. PP and PS has also been included to compare their mechanical properties. All formulations tested are summarised in table 3.

Table 3: Composites based on PLLA, starch, polyester Materbi, PP and PS reinforced with different amounts of microcrystalline cellulose (Avicel).

Matrix	**%wt reinforcing**
PLLA	0, 10, 30, 40
Starch	0, 10, 30, 40
Polyester Materbi	0, 10, 30, 50
PP090	30 (fibreglass/pine), 50 (Avicel)
PS	50

Formulations based on PP and PS were used for comparing the results obtained with the biodegradable polymer matrices.

3.- The results of the mechanical properties, under tensile, flexural and impact stresses, of the formulations of composites reinforced with microcrystalline cellulose (Avicel) are summarised in figure 3.

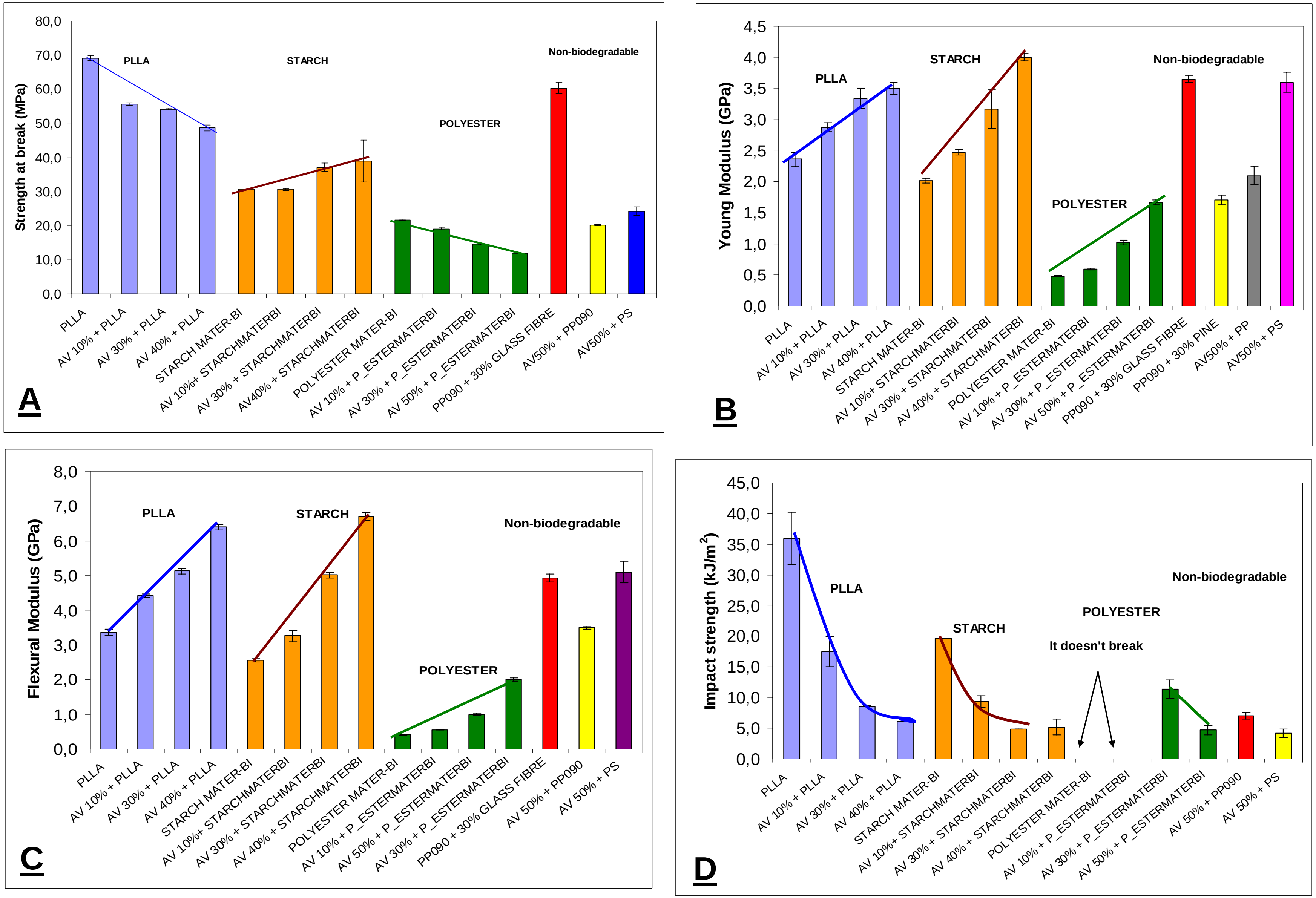

Figure 3: Mechanical properties under tensile, flexural and impact strength of composites reinforced Avicel. 1A) Ultimate tensile strength, 1B) Young Modulus, 1C) flexural modulus and 1D) impact strength.

This task was discussed in the last Sustainpack meeting (May'06, Grenoble) were the different partners agreed to study the effect of the addition of a higher amount of reinforcement than that used until now (30 wt%). Thus, PLLA, starch and polyester Materbi were reinforced with 40 or 50 wt% of Avicel (depending on the loading capacity of each matrix). In this sense only polyester Materbi was able to accept 50 wt% of Avicel and in the case of PLLA and starch the material was impossible to handle at 50 wt% reinforcing level. Once this phenomenon has been informed, the profiles of the evolution of each property was similar than that of the reinforcement until 30 wt%. These profiles can be observed in figure 3. In terms of tensile and flexural tests, the only polymer matrices that undergo a decrease in some properties are PLLA and polyester Materbi, since in both cases ultimate tensile strength decreases. The other mechanical properties are benefited by the presence of Avicel. Impact strength, as in the other cases of fibres, decreases with the increase of fibre reinforcement in the composite.
Finally, a comparative study between the behaviour of the composites formulated with biodegradable polymer matrices and non-biodegradable matrices was carried out. It is possible to accept that PLLA and starch, reinforced with a very high content of Avicel, have a better behaviour under mechanical stresses than PP and PS. This effect indicates that these two matrices can be a good alternative to substitute non biodegradable polymer matrices in the preparation of composites.

Block 4. <u>Reinforced matrices using dry refined fibres from OULU.</u>
Preparation of composites based on PLLA, starch and polyester Materbi as polymer matrix (under independent experiments) reinforced with dry refined fibres (DRF) from University of OULU. A summary of the formulations that were prepared is showed in table 4.

Table 4: Composites based on PLLA, starch and polyester Materbi reinforced with different percentages of Bleachsoft$_{DRF}$.

Matrix	**%wt reinforcing**
PLLA	0, 10, 30
STARCH MATER-BI	0, 10, 30
POLYESTER MATER-BI	0, 10, 30
PP090 + 30% GLASS FIBRE	30

PP formulation was used to compare results between composites based on biodegradable and non-biodegradable polymer matrices.
The results of mechanical properties, under tensile, flexural and impact stresses, obtained with the different formulations of composites reinforced with dry refined fibres (DRF) provided by University of OULU are summarised in figure 4.

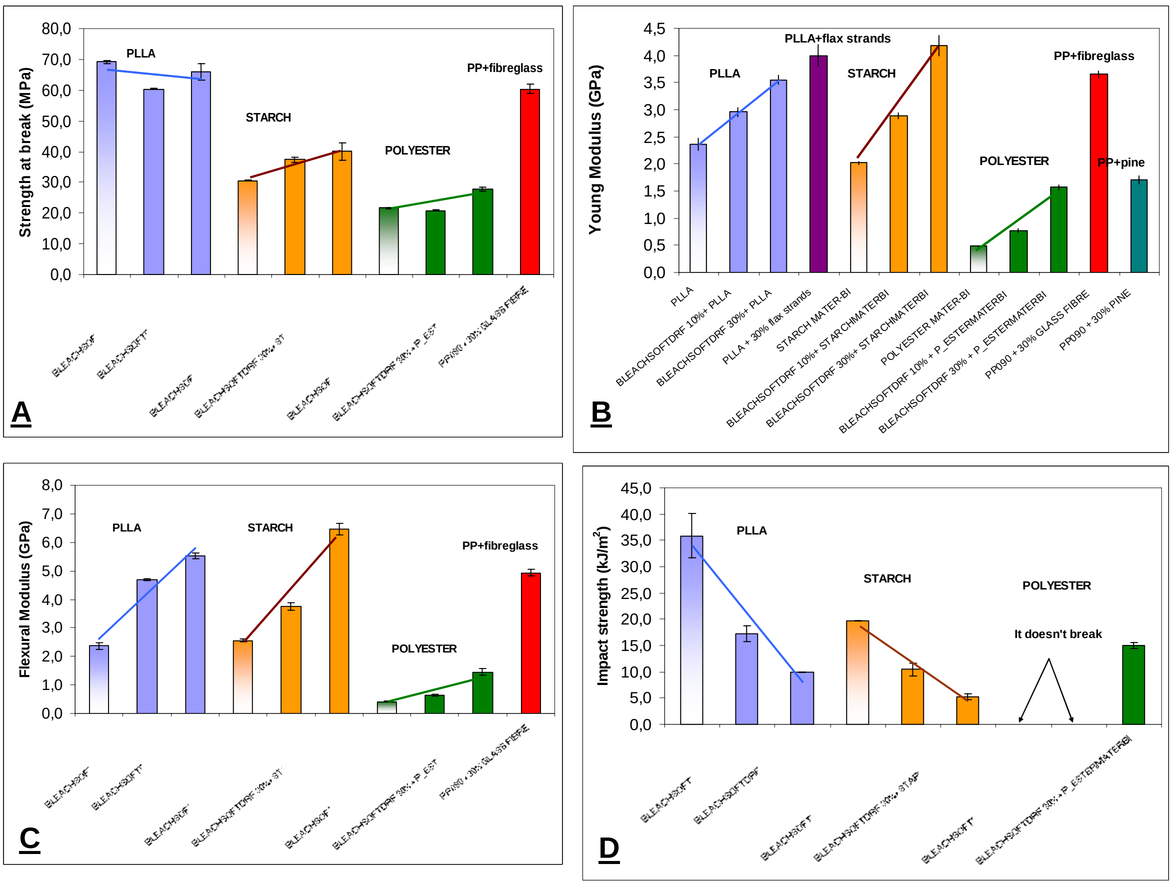

Figure 4: Mechanical properties under tensile, flexural and impact strength of composites reinforced with dry refined fibres (DRF). 1A) Ultimate tensile strength, 1B) Young Modulus, 1C) flexural modulus and 1D) impact strength.

In figure 4 it is possible to observe the influence of the percentage of DRF fibres added to the polymer matrices. Thus, 0, 10 and 30 wt% of fibres were added to each polymer matrix. Only the formulations based on PLLA showed a non linear trend for Ultimate tensile strength. The other mechanical properties showed an increasing trend for all of the polymer matrices. It means that the composite system is reinforced by the addition of the fibres and this reinforcement increases with the amount of fibres in the structure. On the other hand, impact strength showed the typical decreasing profile related with the presence of areas with stress concentration, where with the increase of the amount of fibres in the structure the impact resistance decreases.

For a comparative point of view PP reinforced with fibreglass are showed in several trends. It is possible to observe that several of the formulation of biodegradable composites have similar or even better mechanical properties compared to PP reinforced with fibreglass what indicates the positive use of such matrices for obtaining composites.