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MORPHOLOGY AND FLEXURAL PROPERTIES OF BIOCOMPOSITES FROM MISCANTHUS × GIGANTEUS IN POLYESTER RESIN

MORFOLOGIJA I SAVOJNE KARAKTERISTIKE BIOKOMPOZITA OD MISCANTHUS × GIGANTEUS U POLIESTERSKOJ SMOLI

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- Miscanthus × giganteus
- microstructure
- mechanical properties
- polyester

Ključne reči
- biokompoziti
- Miscanthus × giganteus
- mikrostruktura
- mehanička svojstva
- poliester

Abstract

The paper presents the study of polyester-based biocomposites with different amounts of Elephant grass (Miscanthus × giganteus) particles. The influence of hot-water pretreatment, amount and particle size of Miscanthus on the flexural properties and microstructure is analysed. SEM analysis shows the hot washing pre-treatment of Miscanthus fibres causes a porous fibre structure, and consequently lowers the mechanical properties. Composites with smaller fibres have shown higher flexural stiffness and strength as compared to composites with larger fibres. The increase of the amount of fibres has significantly increased flexural stiffness, while slightly reducing the flexural strength. The use of Miscanthus fibres as a filler in polyester biocomposite materials may be appropriate.

Izvod

U radu su prikazani rezultati ispitivanja svojstava biokompozita na bazi poliesterske smole s različitim udelom čestica miskantusa (Miscanthus × giganteus), poznatog i pod nazivom slonova trava. Ispitan je uticaj tretiranja čestica vrućom vodom, udela i veličine čestica na savojna svojstva i mikrostrukturu. SEM analiza pokazuje da tretiranje vrućom vodom uzrokuje poroznu strukturu vlakana miskantusa, sa posledicom po lošija mehanička svojstva. Kompoziti sa manjim vlaknima pokazali su bolju savojnu krutost i čvrstoću u odnosu na kompozite s većim vlaknima. Povećanje udela vlakana značajnije povećava savojnu krutost, a malo smanjuje savojnu čvrstoću. Rezultati pokazuju da bi se vlakna miskantusa mogla koristiti kao ojačivač u biokompozitnim materijalima na bazi poliesterske smole.

INTRODUCTION

Biocomposites or reinforced composites with natural fibres are recently becoming more and more attractive to engineers and scientists as an alternative for conventional reinforced polymer composites as they offer positive environmental characteristics, lower costs, good overall mechanical properties, etc. /1/. The term biocomposite is applied to materials that are obtained either completely or partially from renewable resources. This approach is environmentally friendly because it restrains the use of toxic materials and minimizes the energy consumption. Natural fibres may be of vegetable, bacterial, animal or mineral origin. Different vegetable fibres have already been widely used, Fig. 1, /2/.

Typical fibres used in biocomposites are wheat, rice or oat straw, barley, rye, flax, sunflower, wood, bamboo, cotton, jute, kenaf, hemp, date, etc. /3/. Flax, for example is currently applied in the interior design of automotive parts, like door panels, roof and boot linings, parcel shelves, /4/.

A successful composite material application of fibres produced form energy production crops like Miscanthus × giganteus would contribute to the agricultural multifunctionality, and improve the farmers' economic stability. One of the future promising areas of application of Miscanthus fibres is in the paper production, since currently less than 1% of total European wood production is based on non-woody crops, /5/. Miscanthus is also an environmentally very friendly plant – its production does not require fungicides, insecticides, pesticides, uses minimal amounts of fertilizers, significantly increases soil organic matter content, which may indirectly contribute to a reduction of atmospheric CO_2 concentration, /6/. Mostly used matrix materials in biocomposites are polypropylene (PP), polyethylene (PE), polylactic acid (PLA), polyester, etc. Flexural properties of different biocomposites based on polyester matrix have been studied, /7-10/, Table 1. This paper shows the use of Miscanthus fibres grown in Croatia for the preparation and characterization of polyester based biocomposites.

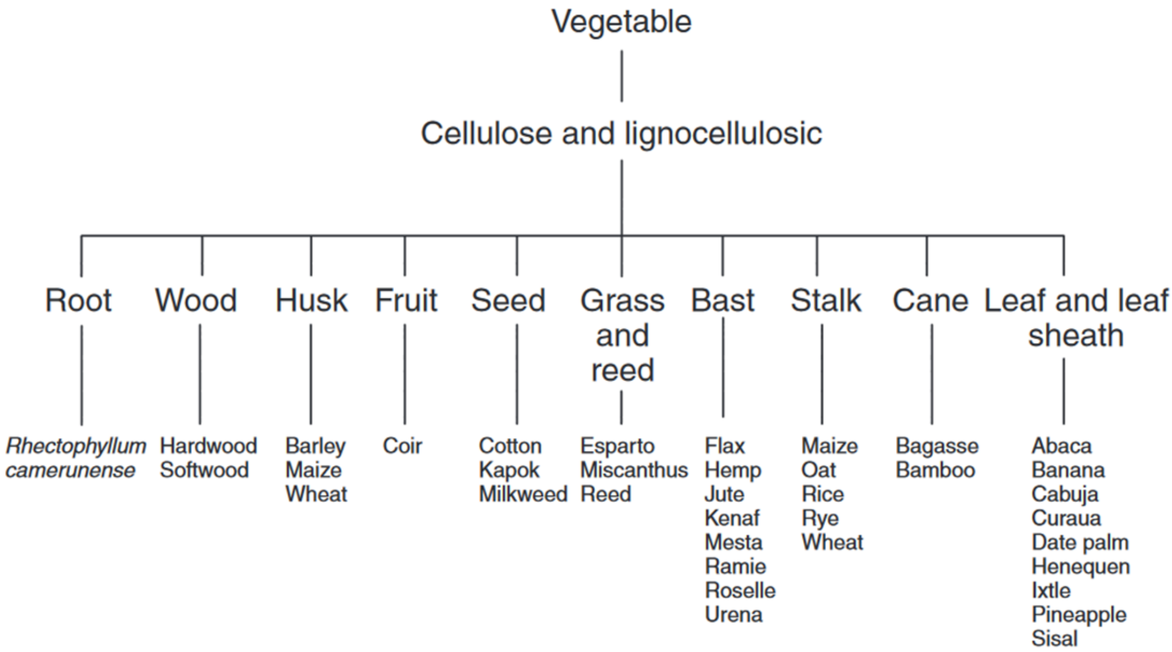

Figure 1. Classification of fibres with vegetable origin /2/.

Table 1. Polyester-based biocomposite flexural properties, /7-10/.

	Strength [MPa]	Modulus [GPa]
Jute laminate (45%) in polyester	92.5	5.1
Parallel laminated glass fibre	590-720	31-38
Treated long kenaf	123	13
Treated long hemp	101	10
Pineapple leaf fibre (30%)	80.2	2.76
Short abaca in biodegradable polyesters	~110	4-6

COMPOSITES PREPARATION

Dry Miscanthus × giganteus grown on a Croatian farm was hammer-milled and mechanically sieved with standard mesh No. 20. A half of the obtained particles with 3.5 mm average size were set as first sample, i.e. sample with large particles. The other half of Miscanthus fibres was further finely sieved using the sieve analysis set Retsch AS 200. Finely sieved fibres of size up to 200 μm were used as the second sample for composite material preparation.

As previous research has shown that hot washing of Miscanthus fibres increases flexural modulus compared to a cold washing regime, /11/, a part of both samples, coarse and fine, was hot washed in distilled water at 90-95°C for 3 h. After hot washing, the fibres are filtered and flat dried in the laboratory drying-oven at 80°C for 8 h. 5 test specimens are made for each of the following 12 different composite material types: 5, 10, and 15 wt.% fibres in polyester resin, hot washed and non-washed fibres, and with fibres below and above 200 μm. The amount of fibres above 15 wt.% resulted in a too viscous mixture, so it was not applied. Weights of two samples of short and long Miscanthus fibres before and after the hot-washing procedure points to a fibre weight loss after hot-washing and drying at about 15-16 wt%, Table 2.

Table 2. Fibre mass loss after hot washing.

	Untreated fibres	After hot-wash	After drying
Fibres shorter than 200 μm	60 g	170.8 g	50.4 g
Fibres longer than 200 μm	60 g	183.7 g	50.9 g

The selected polymer resin was polyester resin from the Croatian manufacturer Kemosan. It is a thixotropic pre-accelerated unsaturated orthophthalic polyester resin, of average reactivity and medium viscosity, mostly used for producing GFRPs and certified by The Lloyd's Register. The recommended amount of 2.5 wt% of catalyst is added and different types of prepared fibre specimens are used to mix up different composite materials.

The obtained suspensions are poured into the waxed steel mould, with mould cavities cut according to the requirements for standard mechanical properties testing, EN ISO 14125 Fibre-reinforced plastic composites - Flexural properties. Each testing body had the dimension as 80 × 10 × 4 mm. The mould and two glass panels were waxed with Spacewax 300 in three layers, each with a thickness of 0.5 g/m^2, and by allowing each layer to dry prior to applying the next layer. The mould was put between two glass panels, and loaded under constant pressure at room temperature for 24 h.

After removing from the mould, the test specimens were post-cured at 80°C, until no increase in hardness was detected. Composites with non-washed fibres resemble very much to the fibreboard structure. Finally, the samples are cut for standard flexural testing, Fig. 2.

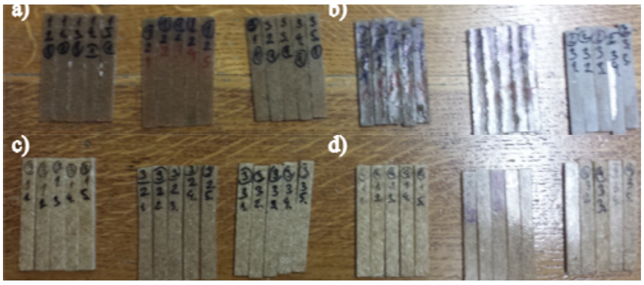

Figure 2. Composites: a) small fibres, non-washed, b) small fibres, hot-washed, c) large fibres, non-washed, d) large fibres, hot-washed.

RESULTS AND DISCUSSION

Scanning electron microscopy (SEM) analysis of composites with large fibres (Fig. 3a) and with small fibres (Fig. 3b) shows a homogenous and well-dispersed distribution of particles. A significantly different morphology of non- and hot-washed Miscanthus fibres is detected also, Fig. 4. The loss of water-soluble starch during hot washing resulted in a porous Miscanthus particled structure.

Influence of the particle size, their weight content and hot washing procedure on the composite material mechanical properties is determined using the standard three-point bending test, by which the flexural modulus and flexural strength are measured (Figs. 5 to 8).

In general, the highest values of both flexural modulus and flexural strength are measured on biocomposites with shorter fibres (< 200 μm) which are not hot-washed. If short and long fibre reinforced composites are compared, better flexural properties are detected when smaller Miscanthus particles are added to the polyester resin.

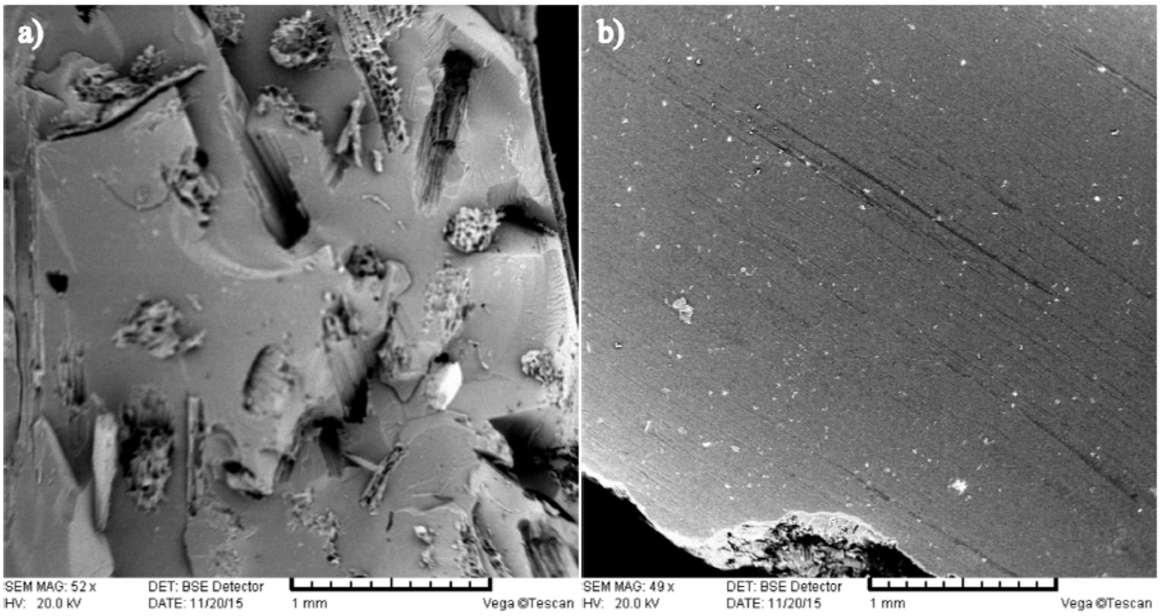

Figure 3. SEM images of the biocomposite with: a) larger, and b) smaller fibres, ~ 50 × magnification.

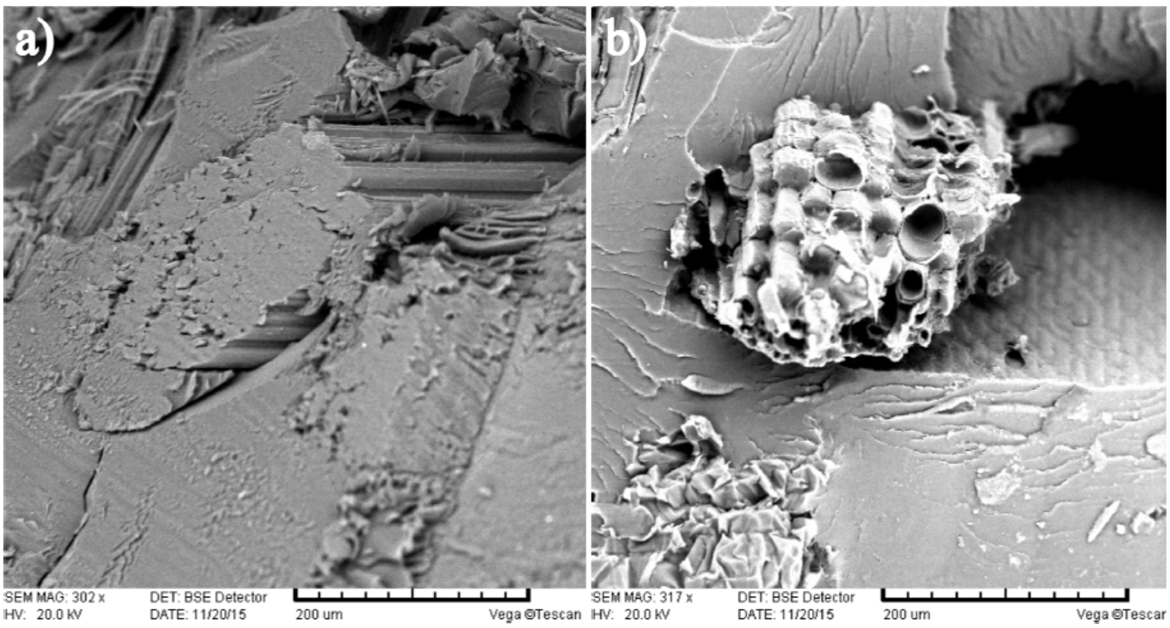

Figure 4. SEM images of: a) non-washed, and b) hot-washed large fibres, ~ 300 × magnification.

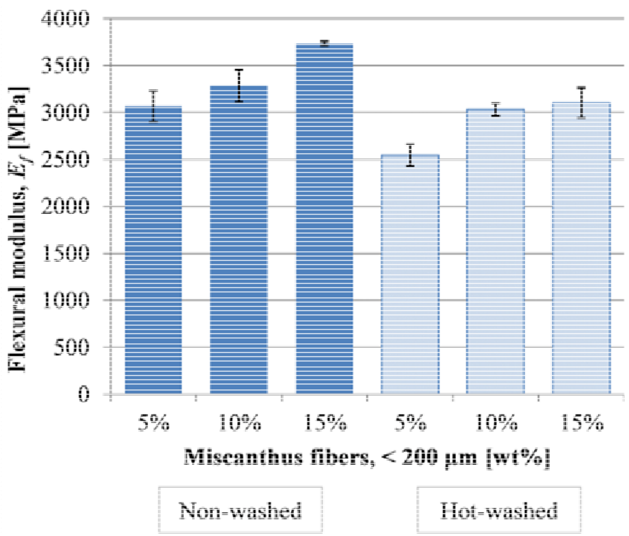

Figure 5. Flexural modulus of the composites with short fibres.

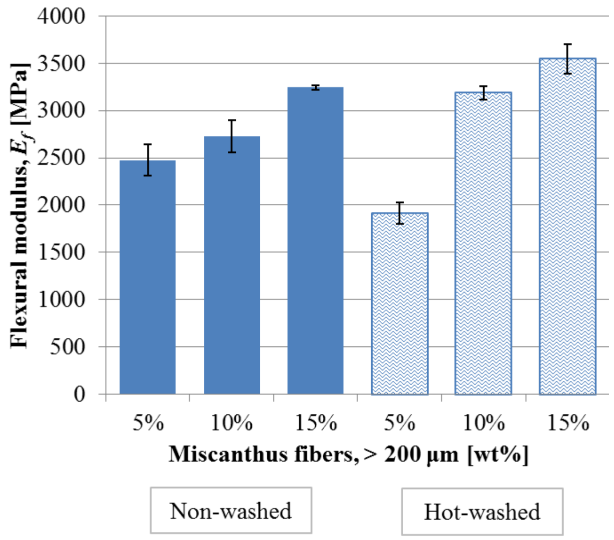

Figure 6. Flexural modulus of the composites with short fibres.

The obtained results show that the flexural modulus is increasing in all composites with the increase in weight content of Miscanthus fibres. When all composite samples are compared, composites with larger fibres exhibit lower flexural strength than the composites with small fibres.

Previous research, /12/, of composites with particles of Miscanthus fibres in PP- or PLA-matrix has shown an increase in the tensile modulus and at the same time a reduction in tensile strength. This is assigned to the insufficient stress transfer from the fibres to the matrix.

Similarly, the research presented in this paper has shown that the increase of small amount, i.e. below 200 μm Miscanthus fibres in the composite material increases the flexural modulus, but reduces the flexural strength. This indicates that Miscanthus fibres act more as fillers, rather than as a reinforcement in polyester resin-based biocomposites.

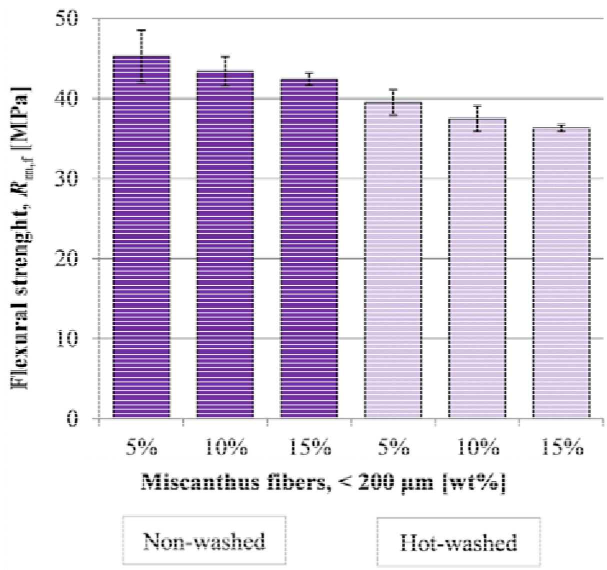

Figure 7. Flexural modulus of the composites with longer fibres.

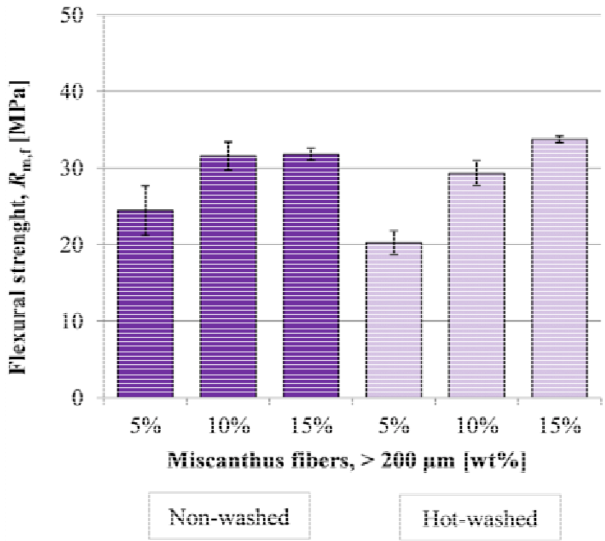

Figure 8. Flexural modulus of composites with longer fibres.

CONCLUSIONS

The research presented in this paper has shown how polyester-based biocomposites with different amounts of Miscanthus × giganteus fibres are prepared. Fibres are either hot water pre-treated or untreated, either small (≤ 200 μm) or large (3.5 mm). Morphology is studied using the scanning electron microscopy. Flexural modulus and strength are determined for each composite material type based on five testing bodies per material type. The following conclusions can be drawn, based on the obtained results:

Scanning electron microscopy has shown that the hot washing pre-treatment of Miscanthus fibres causes a porous fibre structure, and consequently composites with such fibres exhibit lower flexural properties.

Composites with smaller fibres have shown higher flexural stiffness and strength as compared to the composites with large fibres.

The highest values of flexural modulus and flexural strength are observed in biocomposites with non-washed fibres. The increase of the amount of fibres in these composites has significantly increased the flexural stiffness, while slightly reducing flexural strength. This shows that the use of non-washed small (below 200 μm) Miscanthus fibres could be recommended as filler in polyester biocomposite materials, rather than as reinforcement.

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