University of Zagreb Faculty of Textile Technology

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University of Zagreb Faculty of Textile Technology

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NANOBIOCOMPOSITES REINFORCED WITH SPANISH BROOM (*Spartium Junceum* L.) FIBRES

Zorana KOVAČEVIĆ, Sandra BISCHOF, Mizi FAN

Abstract: *Tensile strength of natural fibres used as reinforcement in biocomposite material and treated with microwaves show approximately 60 % higher strength compared to conventional treated fibres and 30 % compared to novel osmotic degumming method. Functionalization of fibres was carried out using montmorillonite (MMT) nanoclay particles and citric acid (CA) as an environmentally friendly crosslinker. Effectiveness of the conducted modifications was examined according to the relevant standardized methods used in current industrial and manufacturing processes (testing of morphological, mechanical, chemical and thermal properties of the final composite material). MMT/CA modified fibres show better thermal stability in comparison to the reference fibre (MWR) which is proved by thermogravimetric analysis. Fibre/polymer interface was also positively influenced by MMT/CA fibre modification. Biodegradability of developed composite materials was examined with serine endopeptidase. Concentration of 50 wt.% enzyme reveals very positive result of composite degradation. Additionally, the possibility of residue stem utilization in bioenergy production was investigated. Proximate and ultimate analysis of residues after MW maceration showed increase in content of positive biomass quality indicators.*
Keywords: *Spartium junceum L., PLA, sustainability, green composites, nanoparticles, flame retardant, biodegradation, bioenergy, solid biofuel.*

1. Introduction

Increased demand for usage of sustainable and biodegradable natural materials initiated wider production of biocomposites. For that reason, composite materials made of sustainable polylactide (PLA) polymer and *Spartium junceum* L. (SJL) bast fibres were designed and produced in the course of research for this thesis. Three fibre extraction (maceration) methods were investigated: water retting (WR), osmotic degumming (OD) and alkali retting under the influence of microwave energy (MW). It was proven that long lasting conventional maceration method can be successfully replaced by ecologically favourable method using microwaves. Natural fibre reinforcements are capable to enhance composite overall properties like mechanical and flame-retardant properties. Alkaline, coupling agents and nanoparticle treatments were used to overcome drawbacks that natural fibres show while used in biocomposite material. Clay nanoparticles were added as a nanofiller which affects the improvement of both mechanical and thermal properties of biocomposites. The increasing use of biocomposites in the normal human life provides a better and healthier life, as well as more holistic view to restoration of eco-system. SJL biomass remaining after fibre extraction was confirmed as promising feedstock for solid biofuel production. The significance of the proposed research lies in the application of innovative, sustainable raw materials for the production of new advanced products of wide application.

2. Materials and methods

2.1 Materials

SJL fibres were obtained from SJL plant, harvested from the area around town Šibenik, Croatia. PLA Ingeo 6201D was purchased from Nature Works LLC, USA with following physical properties: specific gravity is 1.24, relative viscosity is 3.1, melt index is 15-30 g/10 min and melt density is 1.08 g/cm^3. NaOH pellets (purity ≥ 97 %), nanoclay modified with 25-30 wt.% octadecylamine, citric acid, sodium hypophosphite hydrate (NaH_2PO_2) use for this study were obtained from Sigma-Aldrich Inc., UK. The Fluka buffer solutions were used for setting of pH 9.0 (borax/hydrochloric acid). Enzyme Savinase 16 L was obtained from Strem Chemicals, Inc. It is in liquid form with optimum conditions being 30 - 70 °C, pH 8 - 10 and activity of 16 Kilo Novo Protease Unit KNPU (S/g).

2.2 Methods

Numerous methods and testing devices were used in this research [1]. 3 different methods for fibre extraction (WR, OD, MW), chemical modification of fibres, composite manufacturing process, chemical composition of fibres, fibre's moisture regain and moisture content, fibre density determination, tensile properties of fibres and

composites, micromechanical modelling of composites, morphological characterization of sample surfaces by Scanning electron microscopy (SEM), fibre's surface chemistry and its crystallinity indexes by Fourier transform infrared spectroscopy (FTIR), surface properties of fibres by zeta potential determination, thermal degradation of fibres and composites and their kinetic parameters by thermogravimetric analysis (TGA), thermal transition temperatures of composites and the degree of polymer crystallinity of the samples by Differential scanning calorimetry (DSC), the heat of combustion of the gases evolved during controlled heating of composites by Microscale combustion calorimetry (MCC), enzymatic degradation of composites by weight loss measurement, biofuel quality parameters by determination of moisture, ash, coke, volatile matter, fixed carbon, oxygen, carbon, hydrogen, nitrogen and sulphur content, as well as determination of biofuel heating values by using an oxygen bomb calorimeter.

2.3 Samples

Table 1 presents sample description.

Table 1: Sample description

Fibre maceration	Fibre modification	Composites	Composites after biodegradation	Residues after fibre extraction
WR – water retting	MWR – reference fibre	PLA – neat polymer	PLA/CR/C1/C2/C3 20%E – material treated with 20 wt.% enzyme	0_R – SJL stem before fibre extraction
OD – osmotic degumming	1F – fibre modified with NaOH	CR – composite material made of PLA and MWR fibres	PLA/CR/C1/C2/C3 50%E – material treated with 50 wt.% enzyme	SW_R – SJL residue after fibre extraction in salty water
MW – alkali retting under microwave energy	2F – fibre modified with MMT nanoclay and NaOH	C1 - composite material made of PLA and 1F fibres	PLA/CR/C1/C2/C3 100%E – material treated with 100 wt.% enzyme	MW_R – SJL residue after MW fibre extraction
	3F - fibre modified with MMT nanoclay and citric acid CA	C2 - composite material made of PLA and 2F fibres		
		C3 - composite material made of PLA and 3F fibres		

3. Results and discussion

Results are presented in five (5) different chapters.

3.1 Fibre quality

Crucial fibre quality criterion are strength, fineness, length and length uniformity, method of fibre extraction, moisture content, color grade, climate factors throughout the season and soil quality [2]. In this research three methods for natural bast fibre extraction were examined: biological/mechanical (WR with mechanical decortication), physical/mechanical (OD with mechanical decortication) and physical/chemical (MW) method.

Table 2: Prime quality parameters of SJL fibres extracted by various treatments. Results are presented as mean value within 95 % confidence interval

Fibres	Breaking tenacity (cN/tex)	Fineness (dtex)	Elongation (%)
WR	40.66 ± 1.85	41.17 ± 1.74	3.48 ± 0.14
OD	46.21 ± 1.94	40.97 ± 1.46	5.01 ± 0.19
MW	64.44 ± 1.80	36.75 ± 1.81	6.03 ± 0.18

In Table 2 it can be seen that MW extracted SJL fibres show increase in fineness comparing to other two types of fibre treatment (WR and OD) which leads to less stiffness. MW extracted SJL fibres show the highest elongation at break (6.03 %) and breaking tenacity (64.44 cN/tex) values implying the increased toughness of the SJL fibres obtained by MW treatment. The moisture regain of SJL fibres obtained from various treatments

was investigated under 65 % of relative humidity at 22°C and it ranges between 7 and 8 % as presented in Figure 1. Fibres processed under MW treatment have higher moisture regain, which is due to the more succesful pectin, lignin and wax removal [3].

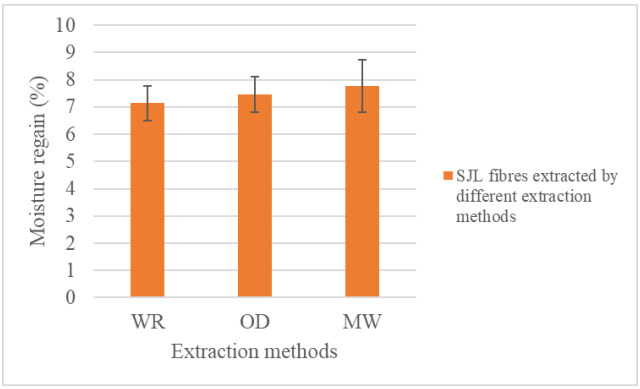

Figure 1: Moisture regain of Spartium junceum L. fibres extracted by different methods where WR is water retting extraction method; OD is osmotic degumming method and MW is alkali retting under microwave energy

FTIR spectra of SJL fibres treated by different extraction methods show that in sample MW lipophilic components are successfully removed and its secondary cell wall is more developed which influence higher mechanical strength of such fibres [4].

3.2 Fibre functionalization

Since MW fibres has showed better properties regarding WR and OD fibres they were further modified by alkali, coupling agent and nanoparticle treatments in order to meet demands for materials used in attractive industries like automotive and construction industry [5]. Structural, physico-chemical, thermal and mechanical properties of MW fibres were investigated but in this conference paper only thermal and mechanical results are presented.

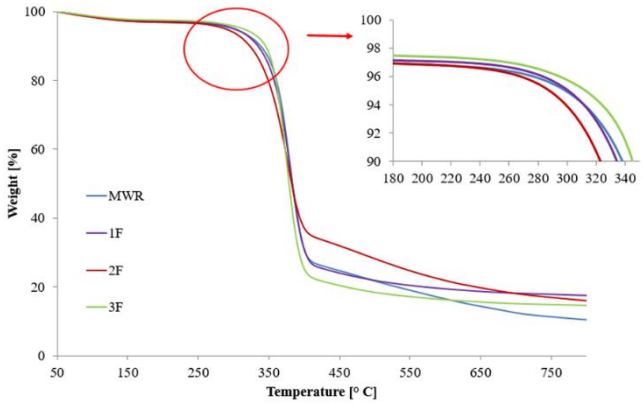

Figure 2: TGA analysis of SJL fibres

In Figure 2 is noticeable that sample 3F shows higher thermal stability than others and higher residue content at 800 °C which is due to more inorganic content inside the modified fibres.
Table 3 shows that fibre strength is increased by nanoclay modification due to the MMT nanolayered structure. Young modulus of modified fibres is slightly higher than modulus of MWR. Lower modulus indicates softer fibres with higher cohesion forces. MMT modified fibres show increase in elongation at break as well [6] which indicates strong and tough fibre that can be used for wide-range industrial purposes.

Table 3: Fibre strength and Young modulus of SJL fibres. Results are presented as mean value within 95 % confidence interval

Sample	Fibre Density g/cm^3	Fibre Strength		Young Modulus	
		cN/tex	MPa	cN/dtex	GPa
MWR	1.55 ± 0.0026	64.44 ± 1.80	998.85 ± 27.97	114.45 ± 4.22	17.87 ± 0.66
1F	1.55 ± 0.0019	60.00 ± 1.33	930.04 ± 20.62	118.71 ± 4.19	18.53 ± 0.65
2F	1.55 ± 0.0027	68.84 ± 1.54	1067.04 ± 23.85	116.65 ± 4.30	18.21 ± 0.67
3F	1.55 ± 0.0028	67.40 ± 1.42	1044.67 ± 22.00	114.82 ± 3.95	17.93 ± 0.61

3.3 Biocomposites – mechanical and thermal properties

Manufactured composite material is based on PLA biodegradable matrix and natural SJL fibres. One of the most important parameters that influence tensile properties of composite materials is the interfacial adhesion between the matrix and the fibres [7]. Poor interface causes reduction of stress transmission from matrix to the fibre thus diminish the tensile strength of the biocomposite material. Tensile strengths for CR, C1 and C3 fibre reinforced composites were increased compared to the sample C2 because of its poor interface properties. As can be seen from our papers [6, 8] sample C3 shows the highest tensile strength, Young's modulus and elongation at break of 46.67 MPa, 2.60 GPa and 7.40 %, respectively pointing to strong and tough material. Mathematical modelling was used for prediction of micromechanical properties, which is very useful in composite designing process. Hirsch model offers relatively good correlation between experimental and predicted results, especially for tensile strength. Predicted tensile strength values were about 10 % lower in comparison to experimental values, except for sample C2, where predicted values are 113.5 % higher than experimental ones.
Flammability properties of tested composites investigated by MCC method revealed that nanoclay treated SJL fibres, which serve as an reinforcement in the sample C2 and C3, affect the occurrence of lower heat release values (W/g) indicating much higher flammability of CR and C1 samples. Table 4 shows corresponding combustion data of tested samples. It could be observed that materials reinforced with SJL fibres show lower peak heat release rate (HRR) and total heat release (THR) in comparison to the neat PLA. The formation of residue in Sample C2 after exposure to 750 °C affects the creation of a thermal barrier [9] that decreases the heat release of the nanoclay treated samples.

Table 4: MCC data of PLA and SJL composite materials where HRR is heat release rate, THR is total heat release and THC is total heat capacity. Results are presented as mean value within 95 % confidence interval

Samples	HRR (W/g)	THR (kJ/g)	THC, gas (kJ/g)	Yield of pyr. residue (g/g)
PLA	475.133±22.633	17.133±0.131	17.330±0.161	0.011±0.003
CR	388.400±24.894	14.433±0.653	15.160±0.436	0.048±0.035
C1	395.567±10.329	14.800±0.408	15.562±0.385	0.049±0.014
C2	280.926±16.735	13.833±0.663	14.581±1.212	0.074±0.006
C3	341.437±6.637	14.800±0.299	15.447±0.172	0.042±0.009

3.4 Biodegradability

Composite materials were subjected to enzymatic degradation in duration of three and five days. Composites reinforced with nanoclay modified fibres show higher biodegradation regarding the presence of excess –OH groups in MMT that may accelerate the hydrolytic decomposition responsible for degradation. C2 sample shows much higher value because of poor adhesion between PLA matrix and 2F fibre that is noticeable in Figure 3. It shows the linearity of PLA sample and polynomial regression of other tested samples regarding their weight loss/time function. According to prediction results, composite materials CR, C1, C2 and C3 will degrade by minimum of 90 % weight loss within 6 months of biodegradation treatment, more accurately within 114, 40, 8 and 36 days, respectively. PLA showed linear proportionality and it will degrade by minimum of 90 % weight loss within 315 days.

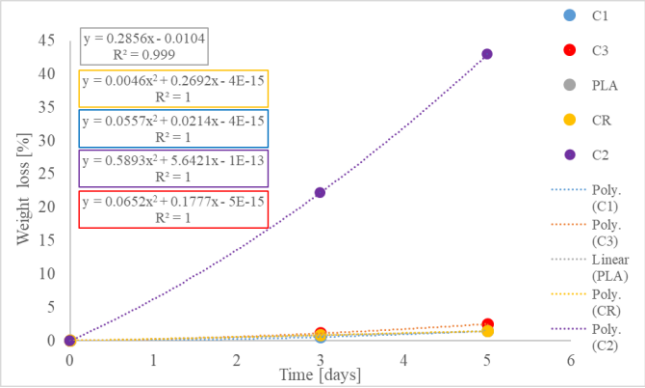

Figure 3: Linearity and polynomial regression of weight loss/time function for neat PLA and its composites when using 50 % of enzyme Savinase 16 L

3.5 Biofuels

After fibre extraction there is almost 90 % of organic residue from SJL plant which can be used as raw material for second-generation biofuel production. Proximate and ultimate analysis were conducted in order to determine energy properties of such biomass. Non-combustible matter content of residue after MW maceration shows low moisture content of 6.5 %, ash content below 5 % and higher fixed carbon value of 13.2 %, while combustible matter content is presented in Table 5.

Table 5: Combustible matter content with higher and lower heating values in the SJL residues after fibre extraction Where db – dry basis; VM – volatile matter; HHV – higher heating value; LHV – lower heating value; Different letters within a column indicate significant differences at the 5 % level; significance * p< 0.05, NS – non significant [10]

	Carbon (%)	Sulphur (%)	Hydrogen (%)	Oxygen (%)	VM (%, db)	HHV (MJ/kg)	LHV (MJ/kg)
0_R	46.51 a ± 0.167	0.28 a ± 0.039	7.12 a ± 0.062	45.12 a ± 0.044	83.50 b ± 2.60	18.83 a ± 0.058	17.28 a ± 0.057
SW_R	43.46 c ± 0.127	0.29 a ± 0.010	6.07 a ± 0.918	49.80 a ± 0.793	77.76 a ± 1.85	17.23 a ± 0.122	15.90 ba ± 0.121
MW_R	44.13 b ± 0.039	0.18 ab ± 0.007	6.76 a ± 0.000	48.75 ba ± 0.050	75.52 a ± 2.64	18.16 a ± 1.112	16.69 a ± 1.112
Significance	< 0.05*	< 0.05*	0.1467 NS	< 0.05*	< 0.05*	0.0907 NS	0.1293 NS

Higher heating value for MW_R sample was 18.16 MJ/kg while lower heating value was 16.69 MJ/kg which indicates high quality biomass that can be used in solid biofuel production.

4. Conclusion

This research gives an insight into possible usage of *Spartium junceum* L. plant as a raw material for fibre production and its application as reinforcement in the composite material manufacturing. This research has examined the effects of surface functionalization of SJL fibres on the properties of natural fibre reinforced PLA composites, including thermal and mechanical behavior, biodegradability and agro-waste utilization.
The results of the present work confirm that the extraction process aided by microwave energy can be successfully used to produce fibres, with properties suitable for textile and composite applications inter alia automotive applications. Additionally, the fibre production time was significantly shortened and the energy consumption was notably lower.
The surface of SJL fibres was modified by alkali and nanoparticle treatment with the addition of environmentally friendly crosslinkers in order to enhance fibre/polymer interface and to achieve better flame retardancy. Composite materials reinforced with fibres of optimum quality show tensile strength and modulus improvement, as compared to the sample C2. The experimental values of composite tensile strength were compared to the values predicted by the Hirsch model and offer a relatively good correlation, since predicted tensile strength values were about 10 % lower in comparison to experimental ones. Biodegradability examination indicates significant biodegradation over 5-day test and 37 °C temperature. Sample C3 show weight loss of 2.5 % after 5-day test with the 50 wt.% enzyme, pointing to high probability of sample degradation by a minimum of 90 % of its weight/volume within period of 35 days.

SJL residues after fibre extraction proved to be good quality biomass for solid biofuel production based on the obtained results of moisture, ash, fixed carbon, coke, volatile matter, nitrogen, sulphur, carbon, hydrogen and oxygen content, as well as the obtained heating values, in order to achieve more efficient and sustainable production. Poverty reduction through the revitalization of SJL fibres would be a tangible outcome of the production of feedstock and the development of bioproducts.

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