



Potential uses of *N*-methylmorpholine *N*-oxide for the treatment of agricultural waste biomass

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Abstract. Lignocellulosic fibers have been used in polymer composites for a long time but their treatment process is still challenging due to insufficient efficiency and environmental impact. This research analyses the influence of *N*-methylmorpholine *N*-oxide (NMMO) on the structure and properties of three types of lignocellulosic fibers – sweet clover (SCS), buckwheat (BS) and rapeseed straws (RS). The fibers were treated with an NMMO solution at 90 °C for different periods of time (80 min, 180 min, 300 min and 30 h). It was detected that after treatment in a dilute NMMO solution, non-cellulosic substances were removed, the morphology and aspect ratio were affected and the fibers were characterized by higher thermal stability. However, the results show that before using NMMO for the production of microfibrils, additional research on the pretreatment process will be necessary because an insufficient aspect ratio value and surface morphology were obtained. The current research shows that it is not economically feasible to process fibers using NMMO for the production of reinforcement for polymer composites.

Keywords: NMMO, buckwheat straw, rapeseed straw, sweet clover straw, lignocellulosic fiber.

INTRODUCTION

Environmental pollution is one of the greatest problems in the world due to the growth of the world's population and the overall improvement of living standards. One of the biggest problems is the huge amount of plastic waste that is returned into the life cycle by various routes, including the development of secondary polymer composites with natural fibers, thus efficiently using available resources to produce environmentally friendly, lightweight and cost-efficient composites with preferable mechanical properties. Natural fibers have been used for a long time, but their purification or modification is still a significant problem due to the variable quality of natural substances, which depends on growing conditions and the type of plant. Lignocellulosic fibers contain such unwanted impurities as hemicellulose, lignin and various resin-type

compounds, which affect the development of reinforced polymer composites. The purest cellulose-type fiber is obtained from cotton, but it requires specific growing conditions that cannot be provided in all parts of the world; therefore, other plants, such as wheat, are also widely used to obtain fibers [1,2]. However, the cellulosic content of other crop fibers is much lower, which increases the importance of purification. An increased amount of impurities makes the purification process even more complicated. Thus, scientists are seeking reagents that are more efficient for fiber purification. All treatment methods are aimed at improving adhesion between the matrix and the fiber through the surface morphology and aspect ratio of the fiber, as during the treatment process part of the non-cellulosic constituents of the natural fiber is removed. Current purification processes involve the use of sodium hydroxide dissolved in a large amount of water, as well as silane, acetic acid, dicumyl peroxide, potassium permanganate, benzoyl chloride and isocyanate com-

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pounds, but they contribute to environmental pollution [3,4].

In our previous research, agricultural residues were mercerized and we obtained promising results (aspect ratio increased, fiber surface morphology improved) for the development of reinforced composites [5]. Taking into account the environmental issue in the current study, *N*-methylmorpholine *N*-oxide (NMMO) was used, which is environmentally friendlier and not chemically hazardous compared to the chemical reagents mentioned above. NMMO is mainly used in the textile industry for obtaining the regenerated cellulosic fiber – Lyocell Fiber – from different plants. More recently the potential use of NMMO has been demonstrated for increasing porosity and sugar percentage of lignocellulosic biomass for enhanced methane production [6]. NMMO is a biodegradable cellulosic solvent and its major advantage is that it is chemically non-hazardous and can be recovered from each treatment process up to 98%, ensuring almost a cyclical production process without polluting the environment. This solvent dissolves cellulose directly in aqueous solution at a certain concentration by disrupting its intermolecular and intramolecular hydrogen bonds; it does not cause destruction of cellulose. Cellulosic fibers are regenerated in a diluted NMMO solution. The process requires relatively low temperatures (90–120 °C), but in order to dissolve the fiber, it is necessary to achieve a certain NMMO and water ratio (frequently used ratio is 85:15). A small amount of water provides better cellulose dissolution but its concentration should not be less than 4% because NMMO decomposition can start then. The ratio of NMMO/water/cellulose for the cellulose dissolution process may slightly vary, however, the optimal ratio is 76/10/14 [7–10].

According to the analyses provided in literature, the influence of NMMO treatment on the chemical composition of lignocellulosic biomass differs – for example, NMMO treatment of almond shells only yields a change in extractives, but in the case of hazelnut skins, the lignin content is reduced by up to 29% [6]. These findings are believed to also contribute to the pretreatment of lignocellulosic biomass for the preparation of reinforcing fibers, because the NMMO treatment method has the potential of obtaining fibers with a lower content of non-cellulosic components and a more opened surface, thus allowing easier mechanical interlocking with the polymer matrix. No less important is the fiber aspect ratio, which could be increased by removing non-cellulosic components; a higher aspect ratio provides better reinforcing ability.

Consequently, this study evaluates the potential use of NMMO for the treatment of lignocellulosic fibers and assesses the influence of process time on the chemical composition, thermal stability, aspect ratio and morphology of the fibers of agricultural waste biomass – buckwheat, rapeseed and sweet clover straws.

MATERIALS AND METHODS

Materials

Rapeseed straw (RS), buckwheat straw (BS) and sweet clover straw (SCS) were provided by the national farms “Brasliņi”, “Pasiles” and “Susuri”. The RS, BS, SCS fibers were obtained with a Retsch SM300 rotary grinder at a speed of 700 rpm using a 0.25 mm sieve. 50 wt% solution of NMMO in H₂O was supplied by Merck (Biotecha Latvia). Propyl gallate was purchased from Biosynth Carbosynth.

Treatment

Firstly, the commercial NMMO solution (50 wt% in H₂O) was concentrated to 85% (w/w) solution by vacuum evaporation. Secondly, the lignocellulosic biomass fibers were added to the prepared NMMO solution to achieve the optimal NMMO/water/cellulose ratio of 74:10:14. To prevent thermooxidative degradation, 1% (w/w) of propyl gallate was added. The vessel was placed in an oil bath at 90 °C under continuous mixing for different periods of time (80 min, 180 min, 300 min and 30 h) to allow the treatment to occur. At the end of the treatment, the fibers were washed using distilled water until pH = 7. The treated fibers were dried in an oven at 60 °C for about 24 h.

Characterization

Chemical composition

Characterization of the chemical composition was carried out at the Latvian State Institute of Wood Chemistry as reported elsewhere [11].

Microscopy

The surfaces of untreated and treated fibers were characterized using a scanning electron microscope (SEM) Tescan Mira/LMU, operated at 5 kV. The samples were coated with a gold layer by means of a plasma sputtering apparatus Emitech/Quorum sputter coater K550X.

The aspect ratio of the fibers was determined by using an optical microscope Leica DMRM, and images were analyzed by means of Fiji image analysis software.

Infrared spectroscopy

Fourier transform infrared (FTIR) spectra were obtained on a Thermo Fisher Scientific Nicolet 6700 spectrometer equipped with attenuated total reflectance (ATR) technique. All spectra were recorded in the range of 650–4000 cm⁻¹ with a resolution of 4 cm⁻¹.

Thermal properties

Thermogravimetric analysis (TGA) was performed by using thermogravimetric analyzer Mettler-Toledo TGA 1/SF under air atmosphere from 25 °C to 600 °C at a heating rate of 10 °C/min. About 10 mg of test sample was used.

RESULTS AND DISCUSSION

The chemical composition of the raw fibers and treated fibers is presented in Table 1. After treatment with NMMO, the cellulosic content of BS fibers decreased by about 18%, while the changes in the cellulosic content of SCS and RS fibers were insignificant, about 3% (increment and decrement, respectively). The highest decrement in the content of hemicelluloses was observed for RS fibers, about 15%, but that of acid insoluble lignin for SCS fibers was about 8%. However, it is important to note that not all straw biomass compounds were analyzed during the compositional analysis. Acid soluble lignin, waxes, pectin, moisture, extractives may influence the content of total lignin and other biomass components in the fibers [12]. Moreover, part of the cellulose may be washed out during the filtration process.

The estimation of chemical composition changes after NMMO treatment based on FTIR spectra is also limited because of the overlap of the absorption intensities of lignocellulosic components. Typical absorbance bands of cellulose are characterized by hydroxyl moieties in a broad range of 3100–3600 cm^{-1} and 1618–1630 cm^{-1} , CH stretching vibrations of methyl and methylene groups at 2950–2800 cm^{-1} , CH_2 bending and C–H deformation in the range of 1420–1430 cm^{-1} , C–H bending at 1365–1373 cm^{-1} , CH_2 wagging at 1313–1317 cm^{-1} , C–O–C stretching near 1160 cm^{-1} , C–O stretching at 1022–1053 cm^{-1} , C–O–C and C–H out-of-plane bending or stretching in amorphous cellulose at 895–899 cm^{-1} [13,14]. The absorbance bands of hemicellulose are attributable to OH and CH stretching vibrations of methyl and methylene groups, C = O stretching at 1735 cm^{-1} , C–O stretching at 1618 cm^{-1} , CH_2 bending and C–H deformation in the

range of 1420–1430 cm^{-1} , C–H bending at 1365–1373 cm^{-1} , CH_2 wagging at 1313–1317 cm^{-1} , C–O–C stretching near 1160 cm^{-1} , C–OH stretching and C–O deformation at 1022–1053 cm^{-1} , C–O–C stretching at 898 cm^{-1} [14,15]. Lignin is characterized by absorbance bands of O–H stretching of phenolic and aliphatic OH in the range of 3450–3400 cm^{-1} , CH stretching vibrations of methyl and methylene groups at 2960–2925 cm^{-1} , C–H stretching of OCH_3 at 2855–2840 cm^{-1} , C = O stretching of unconjugated ketone, carbonyl and ester groups at 1715–1705 cm^{-1} , C–C stretching of aromatic skeleton at approx. 1600 cm^{-1} and 1513 cm^{-1} , C–H deformation at 1460 cm^{-1} , C–C stretching at 1425 cm^{-1} , C–O stretching at 1270 cm^{-1} , C–O–C stretching at 1245 cm^{-1} , C–O stretching at 1030 cm^{-1} , C–H deformation of aromatic rings at 1022–1026 cm^{-1} and C–H bending at 815 cm^{-1} –795 cm^{-1} [13,15]. Typical absorbance bands of other lignocellulosic fiber components such as extractives, pectin and lipids are CH stretching at 2950–2800 cm^{-1} , intensities of carbonyl/carboxyl groups and aliphatic fatty acids at 1730–1735 cm^{-1} , C = C stretching or aromatic ring deformation at 1600 cm^{-1} , benzene ring deformation vibrations at 1510 cm^{-1} [13,16].

Similarly, components of the investigated lignocellulosic straw biomass types share many common bands in FTIR spectra, as depicted in Fig. 1. However, some changes in absorption intensities after the fiber treatment may be highlighted to confirm the change in chemical composition. Thus, BS (Fig. 1b) with the highest extractive content [17] has more intense peaks at 2918 cm^{-1} and 2855 cm^{-1} than SCS and RS. After the treatment, these sharp peaks become less intense. The bands at 1732 cm^{-1} , 1612 cm^{-1} , 1445 cm^{-1} , 1416 cm^{-1} , 1325 cm^{-1} , 1150 cm^{-1} , 1014 cm^{-1} (Fig. 1d) show reduction in the content of lignin, hemicellulose and pectin, as well as waxy fraction after the treatment of BS, especially when longer treatment times have been used. However, not all lignin and hemicellulose fractions are removed because their typical bands are observed at 1506 cm^{-1} and 1234 cm^{-1} (SCS), 1241 cm^{-1} (BS) and 1235 cm^{-1} (RS).

Moreover, after the treatment with NMMO, the peak becomes more intense at 896 cm^{-1} for BS fibers. In

Table 1. Chemical composition of untreated and treated BS, SCS and RS fibers

Amount of compound, % of dry mass	Before treatment			After treatment for a fixed period of time*		
	BS	SCS	RS	BS_80 min	SCS_150 min	RS_80 min
Cellulose	38.1	33.7	33.9	31.2	34.7	32.9
Hemicellulose	19.3	23.9	28.8	19.5	24.5	24.6
Acid insoluble lignin	18.7	25.8	21.8	18.4	23.8	20.3

* shortest time when the fibers looked visually dissolved

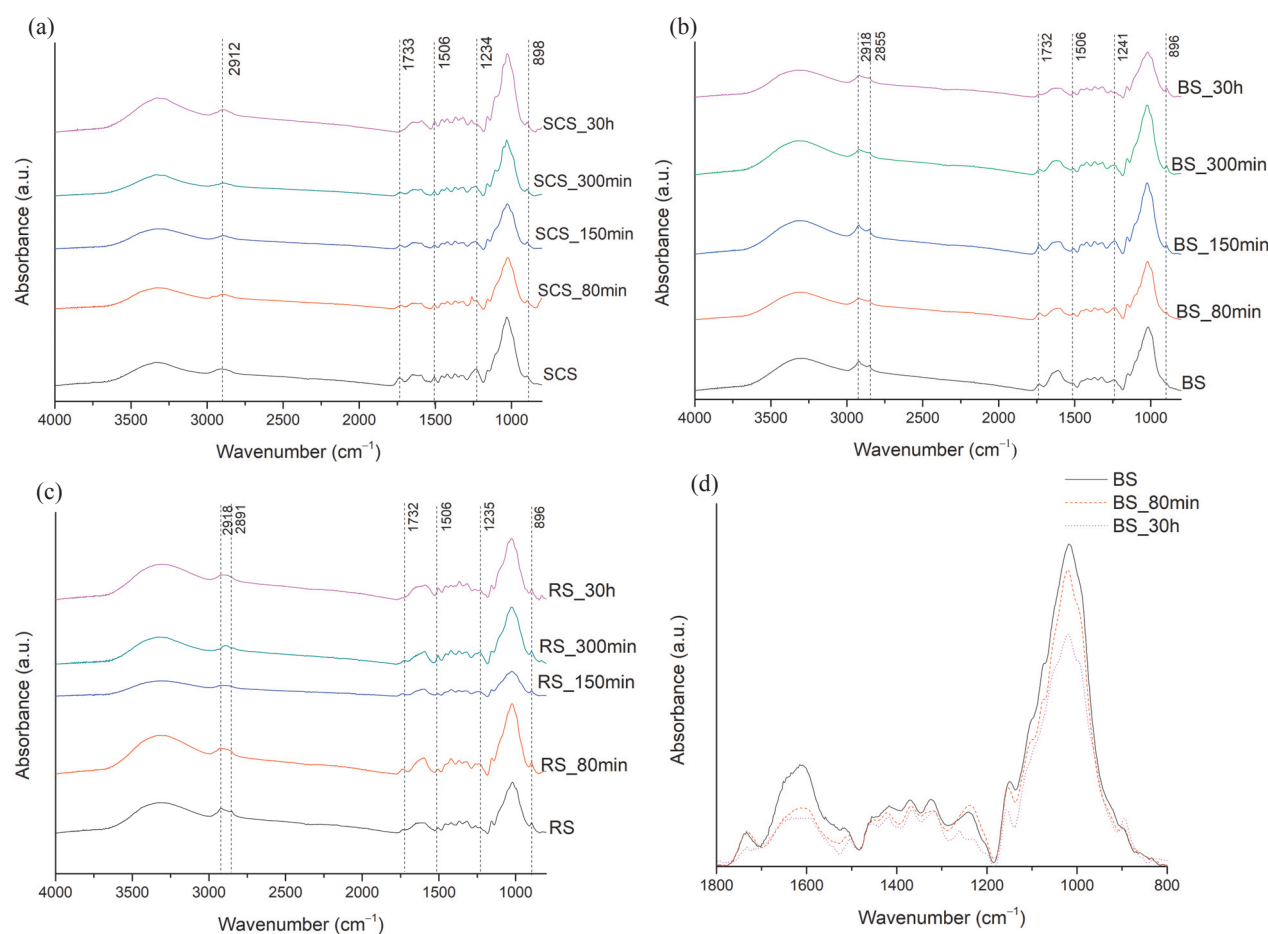


Fig. 1. FTIR spectra of untreated and treated SCS (a), BS (b), RS (c), and the fingerprint region of BS (d).

the case of other fibers, this low-intensity amorphous cellulose peak is visible before the treatment at 896 cm⁻¹ for RS and at 898 cm⁻¹ for SCS, whereas after the treatment no significant changes occur. It is expected that after dissolution of the lignocellulosic fibers, its crystallinity is affected. FTIR analysis may be used for characterization of the crystallinity of lignocellulosic fibers. The lateral order index (LOI) is expressed as the ratio of intensity between the bands related to the crystalline structure of the cellulose (the band at around 1419 cm⁻¹–1424 cm⁻¹) and the amorphous region (the band at around 895 cm⁻¹–899 cm⁻¹) [18]. Overall, Table 2

shows that the LOI decreases by increasing the treatment time, which testifies to the fact that the amorphous part of the cellulose increases, especially in the case of RS fibers.

The changed chemical composition of the fibers considerably affects their thermal properties. As shown in Fig. 2, SCS, BS, RS and their treated samples have a mass loss in three decomposition steps. The first step occurs from 25 °C to 130 °C, where moisture is eliminated. The mass loss at this stage is minor, the highest 7.7% was observed for BS treated with NMMO for 180 min. This may be due to the extractive content, as can be seen from

Table 2. LOI indexes of BS, SCS and RS fibers before and after the treatment

Fiber	LOI				
	0 h	80 min	150 min	300 min	30 h
BS	$A_{1418}/A_{897} = 1.6$	$A_{1421}/A_{896} = 1.4$	$A_{1421}/A_{898} = 1.3$	$A_{1421}/A_{895} = 1.3$	$A_{1421}/A_{895} = 1$
SCS	$A_{1420}/A_{899} = 1.5$	$A_{1422}/A_{897} = 0.9$	$A_{1422}/A_{897} = 1$	$A_{1424}/A_{899} = 1.5$	$A_{1422}/A_{896} = 1.3$
RS	$A_{1423}/A_{895} = 2$	$A_{1421}/A_{895} = 1.3$	$A_{1421}/A_{895} = 1.3$	$A_{1419}/A_{895} = 1.1$	$A_{1419}/A_{895} = 1.4$

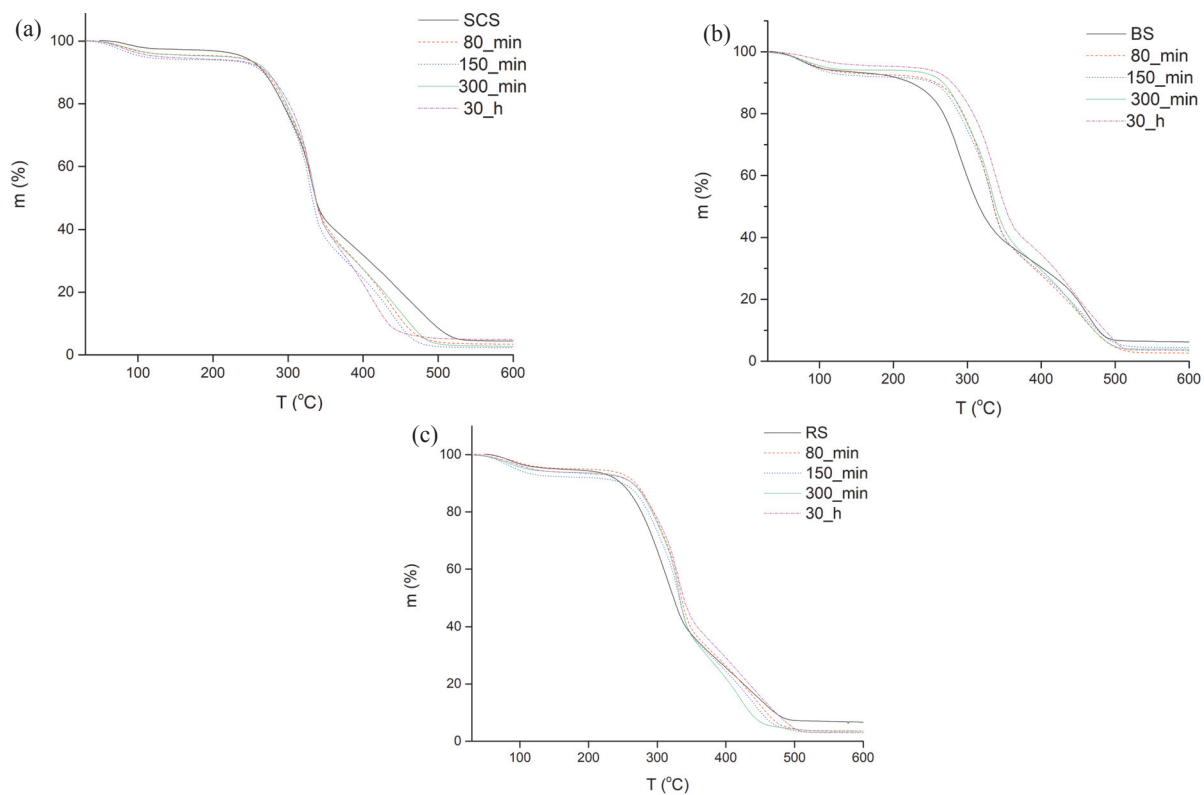


Fig. 2. Mass loss curves of untreated and treated fibers of SCS (a), BS (b) and RS (c).


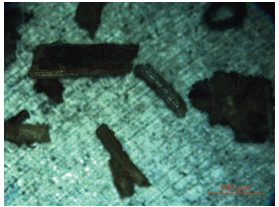


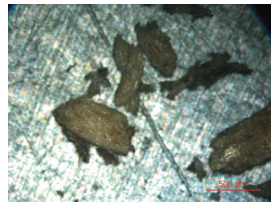
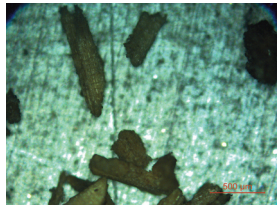

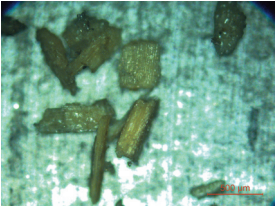

Fiber type	NMMO treatment time		
	0 h	80 min	30 h
BS			
SCS			
RS			

Fig. 3. Micrographs of untreated and treated SCS, BS, RS fibers.

the FTIR spectra demonstrated in Fig. 1b: intensive peaks at 2918 cm^{-1} and 2855 cm^{-1} . The second step (from $180\text{ }^{\circ}\text{C}$ to $370\text{ }^{\circ}\text{C}$ (SCS, RS) and from $180\text{ }^{\circ}\text{C}$ to $380\text{ }^{\circ}\text{C}$ (BS)) mainly characterizes the degradation of hemicellulose and cellulose. The mass loss in this temperature range is detected to be $\sim 60\%$ for all the fiber types. The degradation onset temperature (T_{onset}) of all the fiber types increases – for example, comparing the untreated and 30 h treated fibers, the T_{onset} changes from $256\text{ }^{\circ}\text{C}$ to $294\text{ }^{\circ}\text{C}$ for BS, from $242\text{ }^{\circ}\text{C}$ to $272\text{ }^{\circ}\text{C}$ for RS and from $257\text{ }^{\circ}\text{C}$ to $282\text{ }^{\circ}\text{C}$ for SCS. Furthermore, after the treatment, BS fibers experience the most significant changes in the degradation temperature (T_d , which increases from $290\text{ }^{\circ}\text{C}$ to $335\text{ }^{\circ}\text{C}$ (treated for 30 h)), whereas for RS fibers the temperature increment is from $314\text{ }^{\circ}\text{C}$ to $334\text{ }^{\circ}\text{C}$, and SCS demonstrates almost no increment (from $331\text{ }^{\circ}\text{C}$ to $334\text{ }^{\circ}\text{C}$). This result is a consequence of structural changes after the treatment. During the treatment, part of the extractives and waxy compounds are removed, also a transformation of cellulose I into cellulose II is possible, while the latter is a more thermally stable cellulose polymorph [19,20]. Consequently, the ash content of the treated fibers is lower than that of the untreated ones, e.g., after 300-minute treatment, the ash content decreases from 6.2% to 3.9% for BS, from 6.6% to 3.6% for RS and from 4.4% to 2.3% for SCS.

After the treatment, the purity of the fibers increases but the shape irregularity of BS, SCS and RS particles is still maintained, as can be clearly seen in Fig. 3. It is commonly accepted that after conventional treatment with alkali solution along with the increase in purity, the particle diameter and the mean aspect ratio (AR) increase – for example, after alkali treatment, the AR is 12 for RS, 9 for BS and 5 for SCS [5]. However, in this study it was

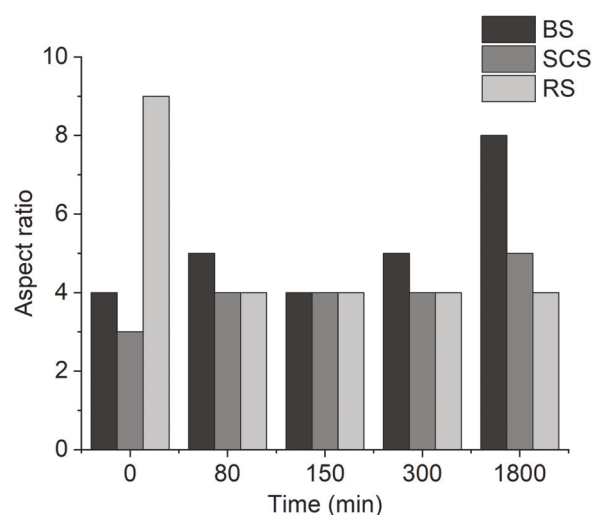


Fig. 4. Mean aspect ratio of untreated and treated SCS, BS, RS fibers.

observed that after NMMO treatment, the AR (Fig. 4) significantly decreased from 9 to 4 for RS fibers, there was a negligible increase from 3 to 4 for SCS fibers, and a considerably increase from 4 to 8 for BS fibers.

Moreover, after the NMMO treatment process, the morphology of the external surface of the fibers becomes more even and is partly covered with a layer of lignocellulosic constituents, such as lignin and hemicellulose. Furthermore, the fiber bundle damage and some pores are visible, e.g., as clearly depicted in the micrographs of BS (Fig. 5). It has been previously reported that after alkali treatment, the surface of lignocellulosic fibers becomes

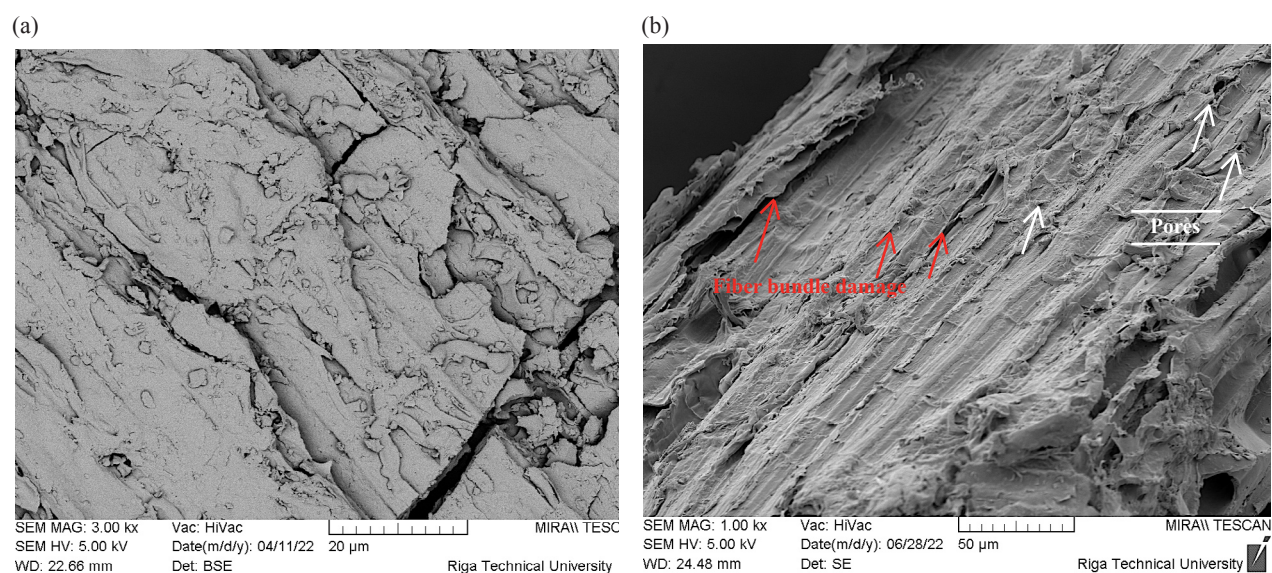


Fig. 5. SEM images of untreated (a) and for 80 min treated BS fibers (b).

rougher and is covered with open pores or pits, which could contribute to improved adhesion during the development of polymer composites [5,21,22]. Consequently, based on the results of the current research, it may be concluded that treatment with NMMO is less efficient in comparison to mercerization [5]. Hence, the potential of using NMMO for obtaining reinforcement for polymer composites is limited.

CONCLUSIONS

The current paper has discussed the microscopy, FTIR spectra and TGA results of three types of lignocellulosic fibers (RS, SCS, BS) regenerated by using NMMO. The results confirm that NMMO treatment has remarkable influence on the thermal stability of the fibers because during the treatment process part of the chemical compounds (extractives, waxy compounds, etc.) is removed. Evaluating the effect of NMMO treatment on the dimensions, aspect ratio and surface morphology of the investigated biomass fiber types, and taking into account that the NMMO treatment process is more time-consuming compared to classical purification methods (e.g., alkali solution treatment), the current research suggests that it is not economically feasible to treat fibers using NMMO to produce reinforcement for polymer composites.

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N-metüülmorfoliin-n-oksiidi kasutamise võimalikkusest põllumajandusjäätmete biomassi töötlemisel

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Lignotselluloosi kiude on polümeerkomposiitmaterjalides kasutatud pikka aega, kuid nende töötlemisprotsess on vähese efektiivsuse ja suure keskkonnamõju tõttu endiselt keeruline. Uurimistöö tutvustab n-metüülmorfoliin-n-oksiidi (NMMO) mõju kolme tüüpi lignotselluloosi kiudude struktuurile ja omadustele.

Lignotselluloosi kiududena kasutati mesika (SCS), tatra (BS) ja rapsi (RS) kiude, mis lisati NMMO lahusele 90 °C juures erinevaks ajaks: 80 min, 180 min, 300 min ja 30 h. Leiti, et pärast koagulatsiooni olid mittetselluloossed ained eemaldatud, mis mõjutas ka kiudude morfoloogiat ning omavahelist suhet.

Analüüsi kiudude (SCS, BS, RS) mikroskoopi, infrapunaspektreid (FTIR) ning termogravimeetrilise analüüsi (TGA) tulemusi pärast NMMO lahuses töötlemist. Tulemused kinnitasid, et NMMO-l on märkimisväärne mõju kiudude termilisele stabiilsusele, kuna töötlemisprotsessi käigus eemaldatakse osa keemilistest ühenditest (ekstrakt, ligniin jt). Hinnati NMMO mõju kiudude mõõtmetele, nende suhtele, pinna morfoloogiale. Võttes arvesse, et NMMO töötlemisprotsess on klassikaliste puhastusmeetoditega, näiteks leelilahuses töötlemisega võrreldes aeganõudvam, on käesolevas uuringus leitud, et kiudude töötlemine polümeerkomposiitide tootmiseks NMMO lahusega ei ole majanduslikult otsustav.