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ORIGINAL PAPER

Mechanical properties for bio-based thermoset composites made from lactic acid, glycerol and viscose fibers

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Abstract Regenerated cellulose fibers were used to produce thermoset composites from a bio-based thermoset resin synthesized from lactic acid and glycerol. The resin was impregnated into the regenerated cellulose fiber and compression molded at elevated temperature to produce thermoset composites. Different fiber alignments (unidirectional and bidirectional), different reinforcement type (warp-knitted and non-woven) and varying fiber loading (65, 70 and 75 wt%) were investigated. The composites were characterized by flexural, tensile and Charpy impact testing and by dynamical mechanical thermal analysis. Water uptake and ageing properties in climate chamber were also characterized for the composites. The results showed that the composites had good mechanical properties. They can be produced with up to 70 wt% fiber content when using unidirectional (UD) and bidirectional fiber (BD) alignment, and with up to 65 wt% fiber content when using the non-woven (NW) reinforcement. The tensile modulus ranged between 11 and 14 GPa for UD composites, 7 and 8.5 GPa for BD composites and 5 and 7.5 GPa for NW composites. The flexural modulus ranged between 10 and 11.5 GPa for UD composites, 5 and 6.5 GPa for BD composites and 5 and 6 GPa for NW composites. The impact strength ranged between 130 and 150 kJ/m^2 for UD composites, 98 and 110 kJ/m^2 for BD composites and 17 and 20 kJ/m^2 for NW composites. The result of the ageing test showed that the mechanical properties of the composites deteriorate with ageing but the addition of styrene somewhat counteracts the degradation, making the composite applicable for indoor use.

Keywords Composite · Regenerated cellulose fiber · Lactic acid · Thermoset · Reinforcement

Introduction

Glass fiber reinforced (GFR) composites are very versatile materials, offering a reasonable compromise between price and technical properties, and are used in many structurally demanding applications. However, this type of composite material is produced from non-renewable constituents and the recycling of them is known to be problematic (Pickering 2006). Finding an alternative to glass fiber reinforcement is therefore of paramount importance. Plant fibers, such as flax and hemp, have been subject for many studies (Faruk et al. 2012; La Mantia and Morreale 2011) and are both renewable and biodegradable. Plant fibers have however also some drawbacks such as annual variation of the properties and low impact properties. The man-made or regenerated cellulose fibers have been used

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since ages in the textile industry, and it is evident that they do have some properties that make them interesting candidates in composite applications. Regenerated cellulose fibers are produced in an established industrial process as continuous filaments, and with a constant quality. These fibers are almost 100 % based on renewable cellulose, and should therefore be a good candidate when seeking for bio-based composites. Man-made cellulosic fibers, however, have not been studied extensively as reinforcement in composite applications. Most researchers have presently focused on the study of thermoplastic composites reinforced with viscose. Composites have been produced from viscose fibers together with polypropylene (Ganster et al. 2008; Paunikallio et al. 2003), polylactic acid (Reinhardt et al. 2013) and cellulose acetate resins (Franko et al. 2001) while Ganster and Fink (2006) reinforced various thermoplastic resins with both Lyocell and Viscose.

For many composite applications, thermosetting resins are preferred since the viscosity is low, thus various impregnation techniques are allowed. The reinforcement of thermoset resins with viscose fibers has not yet been reported extensively. Patnaik and Tejyan (2014) reinforced an epoxy resin with a needle punched fiber mat of viscose and they obtained relatively good mechanical properties. Mader et al. (2012) reinforced an epoxy resin with unidirectional viscose fibers and they also obtained good mechanical properties close to that of GFR samples.

Using a petroleum based resin together with cellulosic fibers will create a composite that is only partly made from renewable materials. While there are many bio-based thermoplastic polymers on the market, bio-based thermoset resins are still under development. We have earlier reported on a thermosetting resin based on lactic acid (Åkesson et al. 2010). This resin was reinforced with various cellulosic fibers (Åkesson et al. 2009, 2011) with a relatively good result, but the resin also had a high viscosity which limits how the impregnation of the fiber reinforcement can be done. We have recently reported the synthesis of a new thermosetting resin based on glycerol and lactic acid (abbreviated GLA; Bakare et al. 2014). This bio-based unsaturated polyester type resin has a relatively low viscosity which facilitates impregnation of the fibers and it can be cured by a free radical polymerization.

The purpose of this study is to prepare composites from this lactic acid-based resin (GLA) with viscose reinforcements. By this combination, composites with almost 100 % bio-based content can be made, which is a clear improvement compared to the previously reported similar composites. The composites are prepared from a viscose yarn by using two different fiber alignments of warp-knitted fabrics, and by using a non-woven viscose fabric. The fiber content was also varied, and the composites were characterized by flexural, tensile and Charpy impact testing, by dynamical mechanical thermal analysis and by scanning electron microscopy (SEM). The ageing test and water absorption of the composites were also investigated to ascertain its possible applications.

Experimental

Materials

L-Lactic acid (88–92 %, Sigma-Aldrich, Sweden), glycerol (99.5 %, Scharlau, Fisher Scientific, Sweden) and methacrylic anhydride (94 %, Alfa Aesar, Sweden) were used for the resin synthesis. Methanesulfonic acid (98+ %, Alfa Aesar) and toluene (99.99 %, Fisher Scientific) were used as catalyst and solvent. Hydroquinone (99 %, Acros Organics, Fisher Scientific, Sweden) was used as an inhibitor prevent cross-linking during the synthesis. Dibenzoyl peroxide (75 % in water, Merck-Schuchardt, Germany) was used as initiator for the free radical-initiated cross-linking of the synthesized resin. Styrene (99 %, Sigma-Aldrich, USA) was used as reactive diluent for making composites for ageing tests. All reagents were used as received.

Viscose non-woven fiber mat with surface weight of 60 g/m^2, and 0.66 mm thickness were supplied by Suominen Non-Wovens Ltd., Finland. Warp-knitted uniaxial fabric with surface weight of 182 g/m^2 made from a viscose yarn from Cordenka, (twist Z40, linear density of 2,440 dtex with 1,350 filaments), was delivered by Engtex AB, Sweden.

Resin synthesis

The resin was synthesized according to the method described in the previous article (Bakare et al. 2014). Shortly, the resin was prepared in two steps. In the first step, lactic acid and glycerol were mixed in a three neck flask with a molar ratio of 9:1 in toluene. The

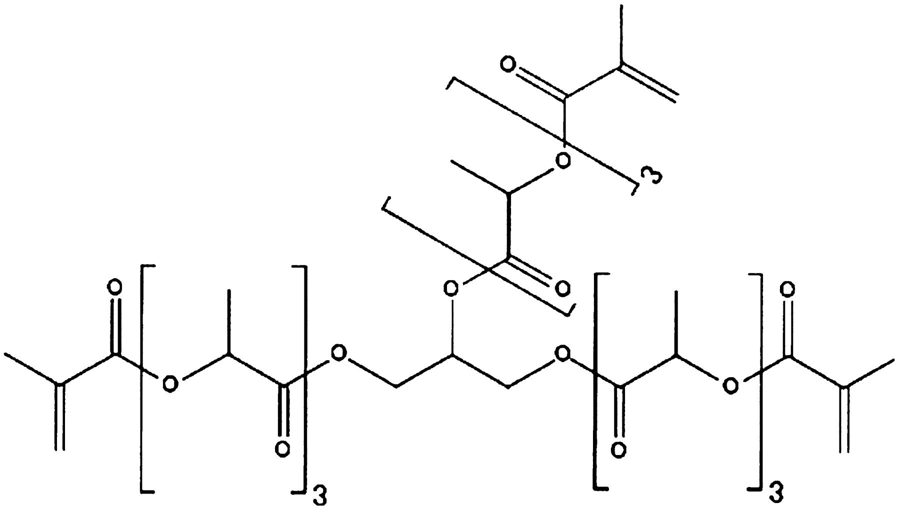


Fig. 1 Idealized structure of final GLA oligomers

reaction mixture was heated for 5 h at 145 °C in the first 2 h, increased to 165 °C for the next 2 h and 195 °C in the last hour, then the water was distilled off azetropically. After the reaction the temperature was reduced to 90 °C by moving the flask into another oil bath. The flask was equipped with an ordinary reflux condenser and nitrogen purge inlet and then 3.3 mol of methacrylic anhydride per moles of glycerol was added drop wise during 1 h. The temperature was kept for three more hours, and then the resin was isolated by distillation of the solvent and the methacrylic acid formed from the reaction. An idealized structure for the final oligomers is shown in Fig. 1.

Composite preparation

The reinforcement mats were cut in 21 × 21 cm size and dried in vacuum oven at 15 mbar and 105 °C for 1 h. The resin impregnation was done by hand lay-up. The resin was first mixed with 2 wt% of dibenzoyl peroxide and then it was applied onto the surface of the fiber sheet. The laminates have 12 sheets of woven fabrics and 34 sheets of non-woven fabrics in respective laminate type. The fabric stacks were then compression molded (Rondol press) for 5 min at 150 °C and at 0.5 to 4 MPa pressure (depending on the desired fiber content). This gave laminates with an approximate thickness of about 2–3 mm.

Composite laminates were prepared using only the non-woven fabric (NW) and only the warp-knitted fabric in the laminates. Composites made from the warp-knitted fabrics were produced with all weft yarns in one direction (unidirectional or UD) and with a 0°/90° alternating lay-up (bidirectional or BD). Furthermore, three different fiber loads of 65, 70 and 75 wt% were investigated. The exact fiber weight load was monitored by weighing the dery fabrics and the cured composite.

Characterization

All samples were cut by laser cutting to obtain specimens for DMTA, tensile, flexural, Charpy and water absorption testing. The DMTA was done on a Q800 instrument (TA Instruments, DE, USA) and in the dual cantilever bending mode. The specimen dimensions were: length 35 mm, width 8 mm and thickness 2–3 mm. The temperature ramp was from −20 to 150 °C, a heating rate of 3 °C/min, amplitude of 15 µm and frequency of 1 Hz was used.

The composites were further characterized by a three point bending flexural test according to ISO 14129 using a H10KT (Tinius Olsen, USA) machine equipped with a 5 kN load cell. The cross head speed was 5 mm/min and the holders distance apart was 64 mm. A minimum of six specimens were tested for each combination of fiber alignment type and load. Standard deviation and mean values were calculated.

For tensile test 150 mm long dog bone shaped specimens were tested on the same testing machine using a 10 kN load cell and an mechanical extensometer. At least five specimens were tested in the tensile test. Standard deviation and mean values were calculated.

Charpy impact tests were performed using Zwick pendulum type mechanical impact testing machine (Zwick GmbH and Co. KG, Germany) according to ISO 179 standard. The unnotched specimens were located for edgewise impact. Minimum of ten tests were performed and mean value and standard deviations were calculated for each material.

For the ageing under humid conditions only the composites with 0/90 lay-up and a fiber ratio of 65 wt% were tested. The specimens of length 30 mm by 10 mm width was placed in a climate chamber (HPP 108/749) supplied by Memmert GmbH, Germany at 50 °C and 85 % relative humidity for 1,000 h. The aged specimens were then tested by tensile testing to determine the effect of humidity on the mechanical properties.

In the moisture absorption test, the specimen weight was measured before placing in the climate chamber and after keeping in the chamber for 1, 7 and 40 days. Moisture absorption is reported as the percentage of weight increase and was calculated by Eq. 1, where W_f is the weight of sample after the moisture absorption and W_i is the weight of the dry sample.

$$\textit{Percentage of moisture absorption} = \frac{W_f - W_i}{W_i} \times 100 \tag{1}$$

Results

Scanning electron microscopy

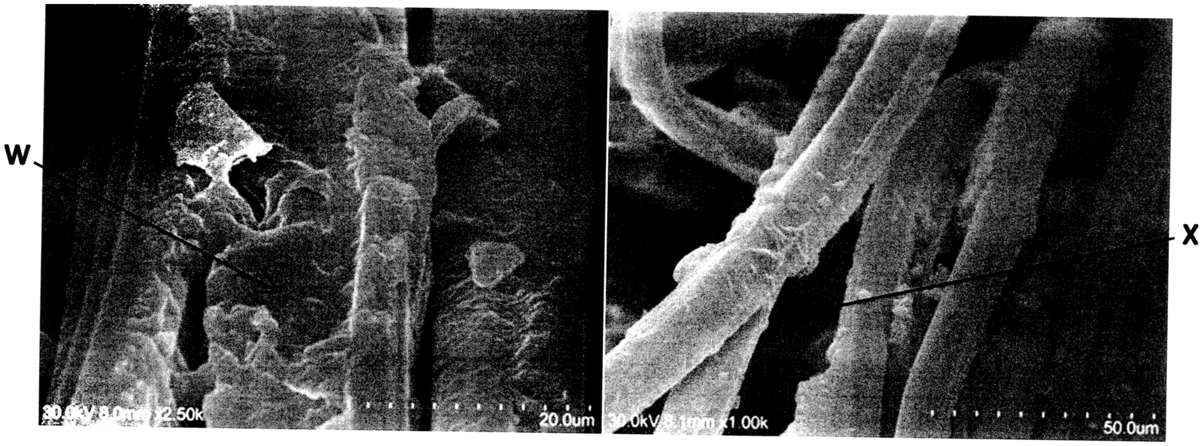
Scanning electron microscopy (SEM) micrographs of the cross-sections are shown in Fig. 2. Individual fibers fully covered by the resin can be identified with an arrow W in (a) but fibers that are only partly covered with the resin can also be seen with arrow X in (b). This indicates a fairly good adhesion. The composite with UD type warp-knitted reinforcement at 75 wt% is shown in (c), and a very high amount of poorly impregnated fibers can be seen with arrow Y. This indicates that this fiber content is too high for the chosen process method. The composite with 65 wt% non-woven is shown in (d), where debonding between the polymer matrix and the fiber can be seen in arrow Z which indicates that the adhesion is not optimal.

Flexural testing

Figure 3a, b show the flexural modulus and strength for the composites with various fibre layup and fiber weight content. It is evident that an increase in fiber content from 65 to 70 wt% has a minor effect when considering the standard deviation. Increasing the fiber content to 75 wt% did not further improve the flexural properties. Inspection of fractured surface for the composite with 75 % showed individual, unimpregnated fibers, as indicated in the SEM micrograph in Fig. 2c. So it could be concluded that 70 % is the higher limit for fiber load for employed knitted viscose fiber fabrics with the chosen process conditions. In case of NW composites increasing fiber content from 65 to 75 wt% caused some reduction in flexural properties. Considering the relatively high flexural strength and modulus for NW composites with 65 wt% fiber load and the existence of dry fiber in some areas in the laminates with 70 and 75 wt% fiber content, it could be concluded that 65 wt% is the upper fiber content for these composites. Figure 3c indicates that flexural strain at maximum for the UD type composites decrease when increasing fiber load but considering the standard deviation, this trend is not statistically significant. For the BD type composite, the flexural strain at maximum increased by increasing fiber load to 70 and to 75 wt%, but considering the standard deviation the effect is not significant. For the NW fiber composites, there is significant increase in strain at max with increasing fiber load to 75 wt%, while increasing fiber load to 70 wt% has no significant effect.

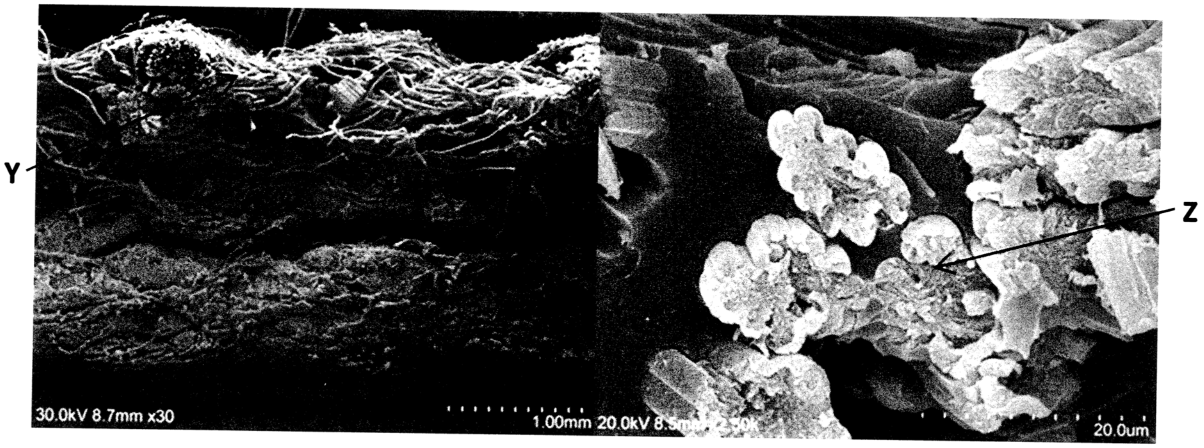
Tensile testing

Figure 4a, b and c shows tensile modulus, tensile strength at break and maximum elongation at break for the different composite types. The tensile modulus and strength increases with increasing fiber load from 65 to 75 wt% for UD and BD composites. The UD composites are relatively strong with tensile strength above 200 MPa and with relatively high moduli, since the strength of the composite is doubled compared to the BD composites when the composite is tested in the fiber direction. In the case of the tensile strength of the BD composites; there is roughly half of the value in comparison to the UD composites. This is expected



(a) UD 65 wt-% fibre load

(b) UD 65 wt-% fibre load



(c) UD 75 wt-% fibre load

(d) NW 65 wt-% fibre load

Fig. 2 SEM micrographs of the cross-sections for composites with warp-knitted and non-woven type reinforcement

since only 50 % of the fibers are aligned the tensile testing direction. For the NW composites a decrease in tensile strength and moduli was obtained, which also agrees with the results from the flexural testing that it is not possible to use higher fiber ratios than 65 wt%. The results from Fig. 4c indicates that maximum elongation for UD and BD type composites decrease by increasing fiber load but considering the standard deviation, the decrease is not significant. For the NW type composites, there is a significant decrease in elongation compared to the UD and BD composites, while effect of fiber load is not significant.

The tensile stiffness for composites prepared from various man-made cellulosic fibers and thermoplastic PLA matrices have previously been studied. Composites were for example prepared from viscose-PLA with 25 wt% fiber load, lyocell-PLA with 40 wt% fibers and viscose-PP with 30 wt% fiber load and the Young's modulus was reported as 4, 6.8 and 2.88 GPa (Ganster and Fink 2006; Graupner et al. 2009; Ganster et al. 2008) respectively which are considerably lower compared to our made GLA-viscose composites which had 14.2 (75 wt% UD) and 7.5 GPa (65 wt% NW) respectively. The use of thermoplastic PLA as matrix will of course limit how the composites can be prepared. Injection molding, as used in the studies above, will limit the fiber length, giving lower mechanical properties. That exemplifies the advantage of using a thermosetting resin based on lactic acid. Composites from aligned viscose fibers (Cordenka) and epoxy resin have previously been studied (Mader et al. 2012). They received roughly similar mechanical properties with a

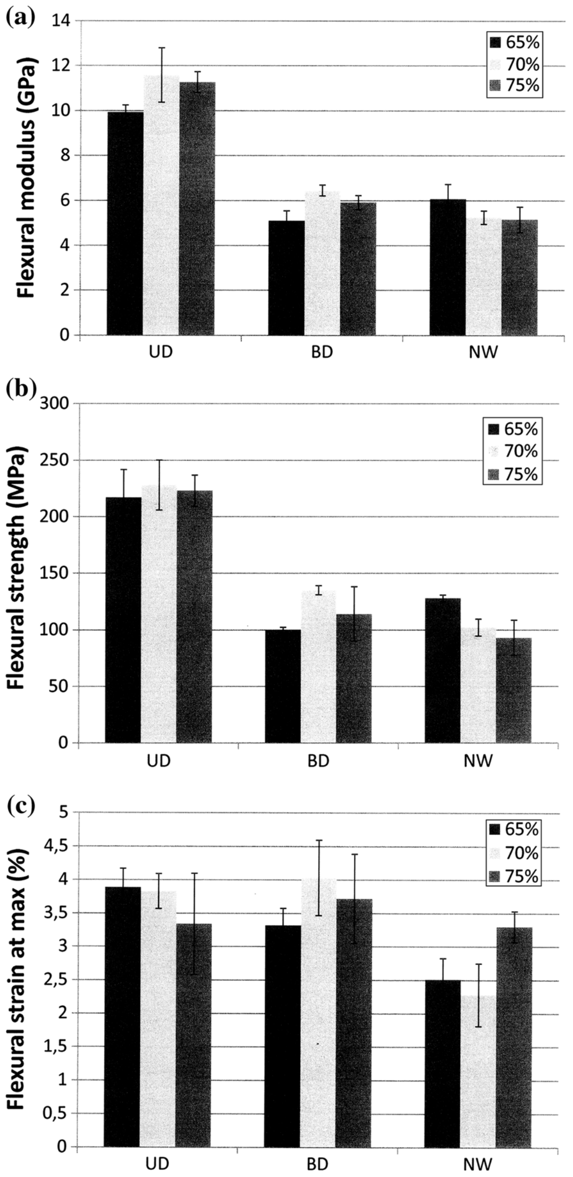


Fig. 3 **a** Flexural modulus comparison of the composites. **b** Flexural strength comparison of the composites. **c** Flexural strain at maximum comparison of the composites

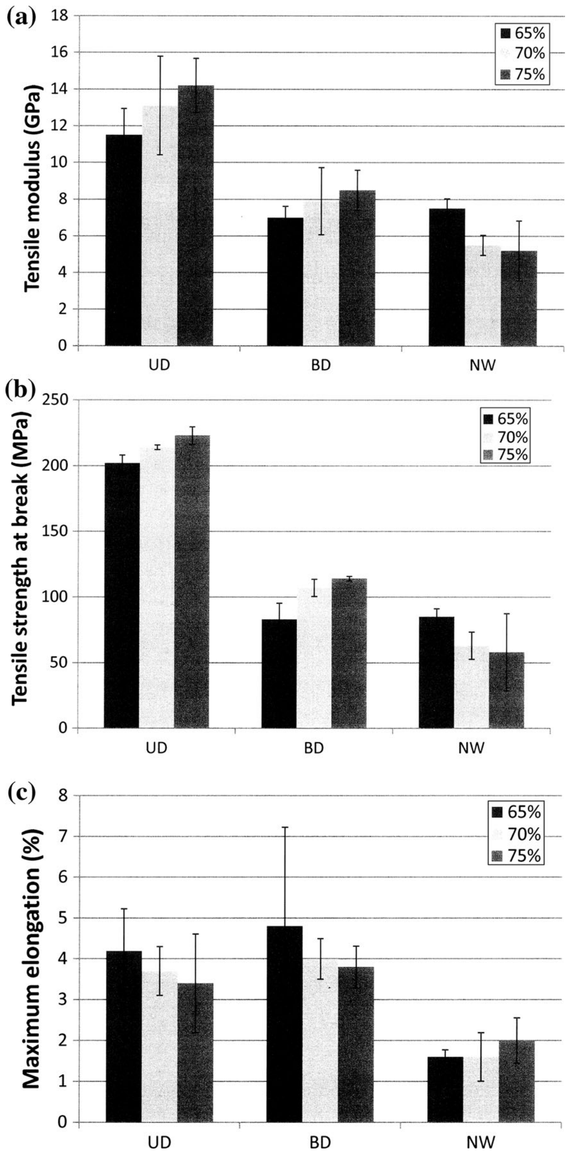


Fig. 4 **a** Tensile modulus comparison of the composites. **b** Tensile strength at break comparison of the composites. **c** Tensile maximum elongation comparison of the composites

tensile strength of about 250 MPa. A different impregnation technique was used in that study with somewhat lower fiber content so the results are not directly comparable.

Charpy impact resistance

Charpy impact strength is one of the important mechanical properties for many practical applications. Figure 5 shows absorbed energy for unnotched Charpy impact test for the composites. A relatively high Charpy impact strength for the UD type composites which increase with increasing fiber load from 65 to 70 wt% and decrease by increasing fiber content to 75 wt%. Due to the relatively high standard deviation it is impossible to draw a definite conclusion when comparing the composites with different fiber loadings. High impact energy is a sign of good

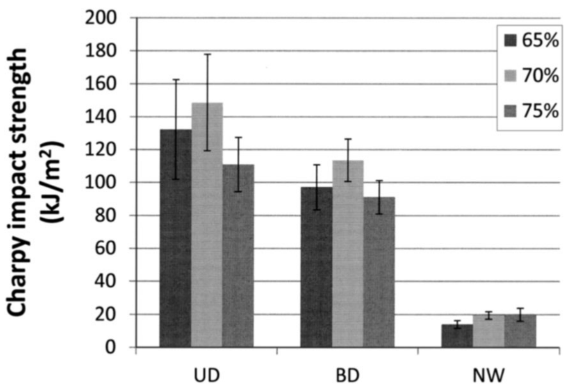


Fig. 5 Impact strength comparison of the composites

adhesion between fiber and matrix and by increasing fiber load contact area and so energy absorbed in fracture increases. Decrease in Charpy impact energy could be a sign of improper impregnation for higher fiber loads. Investigation on fractured area of sample shows that a minority of the fibers was broken but fiber pull out did occur in a wide area around the impact point. Increase in absorbed energy at higher fiber load could be explained by an increase in interfacial area which is more likely to be responsible for impact energy dissipation. The decrease in impact energy dissipation in case of improper impregnation for higher fiber load could be explained by a decrease in interfacial area.

Results in Fig. 5 depict that absorbed energy for non-woven composites are, as expected, much lower compared to the aligned warp-knitted fiber composites. Comparing to other reports about thermoplastic PLA reinforced with various natural fibers, the obtained results are roughly comparable. (Oksman et al. 2003) reported on PLA reinforced with 40 wt% flax fibers and got about 12 kJ/m^2. (Bledzki et al. 2009) reported on PLA reinforced with viscose fibers and reported on a notched Charpy impact strength of about 8 kJ/m^2. The results of the impact strength of the non-woven type composites are in line with what we reported on earlier for a lactic acid-based thermoset resins reinforced with a non-woven flax fiber mat (Åkesson et al. 2009). Also the maximum fiber content for proper impregnation for our thermoset PLA and man-made cellulose fibers is around 70 % while it is around 30 % for thermoplastic PLA (Bax and Müssig 2008).

Dynamic mechanical thermal testing

Figure 6a shows storage modulus of neat resin compared to composites with different reinforcement types for 65 wt% fiber load. A considerable improvement in stiffness of composites compared to the neat resin is seen, and the storage modulus of the composites at room temperature is in a very good agreement with their flexural modulus, as expected. The strong increase in the storage modulus of UD composite can be interpreted as good adhesion between the fiber and the matrix.

Figure 6b presents tan delta, which indicates the damping properties of neat resin and UD, BD and NW composites with 65 wt% fiber load. The damping properties is said to be good if the tan delta intensity is high, which means there is a balance between the elastic and viscous phases in the polymer structure. The peak of tan delta can be interpreted as the glass transition temperature (T_g), and the results can be interpreted as increase in T_g for the composites from 80 °C in neat resin to above 100 °C in best cases. An increased T_g when a polymer matrix is reinforced with cellulosic fiber has been observed also by several authors. For example, Åkesson et al. prepared composites from acrylated soybean oil resin reinforced with air laid flax mat with fiber ratio between 30 and 70 wt%. Saw et al. 2012 reinforced an epoxy novolac resin with jute-coir hybrid fiber, and an increase in the glass transition temperature was observed which was attributed to the strong interaction between the fiber and the matrix caused by restrict mobility of the polymer chain in the interphase (Åkesson et al. 2011).

Figure 6c shows that T_g is increasing by increasing fiber content for almost all reinforcement types which is expected for a composite with good interaction between fiber and matrix.

Ageing testing

Ageing test was performed to identify the optimal application fields of the made composite. This is done by subjecting the composite to accelerated ageing condition to make the composite reach the same aged end-state as a real time aged composite but in less time. According to Duta and Cazan, who studied ageing under UV radiation and saline fog on a composite based on rubber, recycled plastic and fly ash. The Young's modulus showed a major decrease

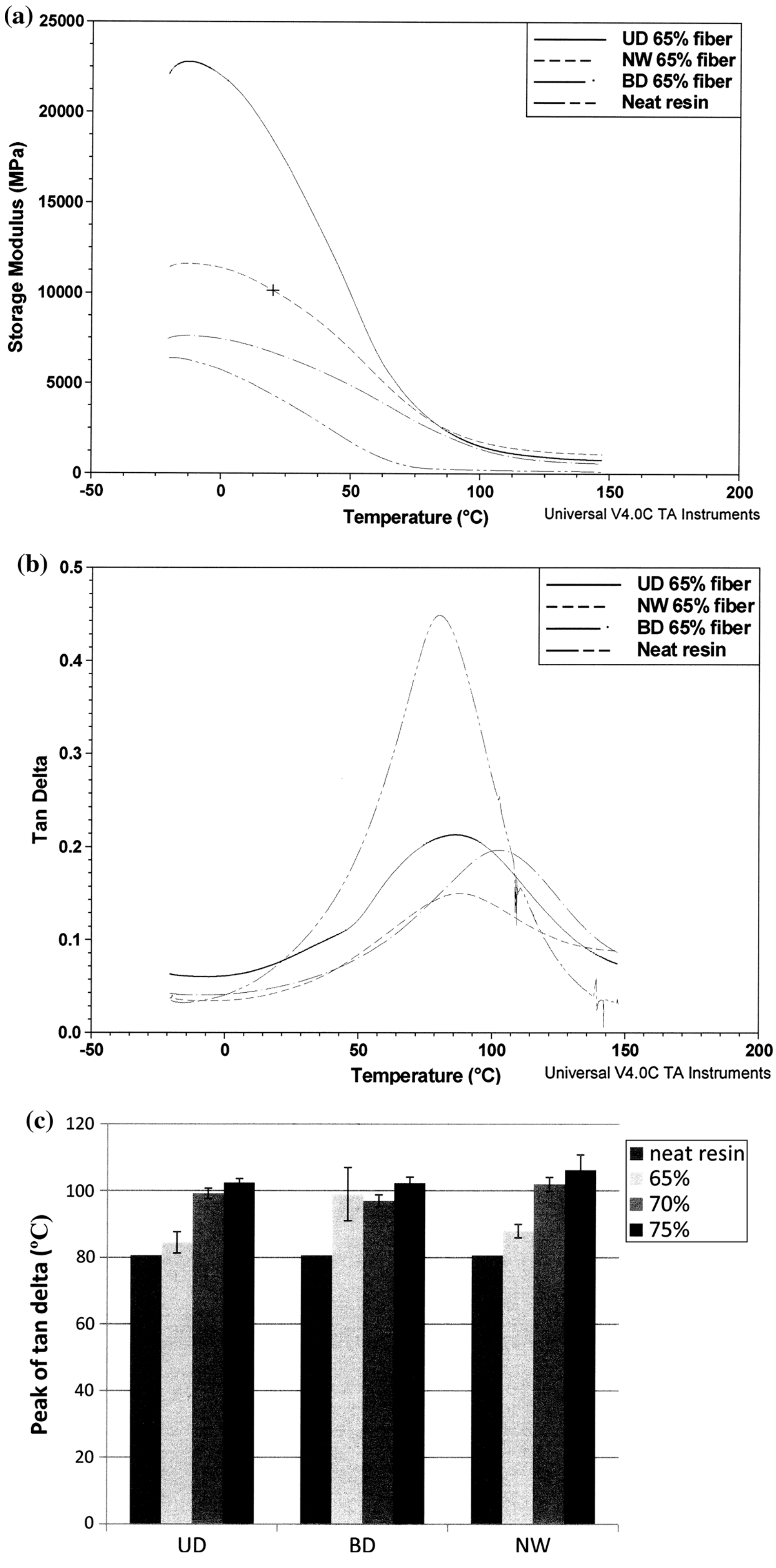
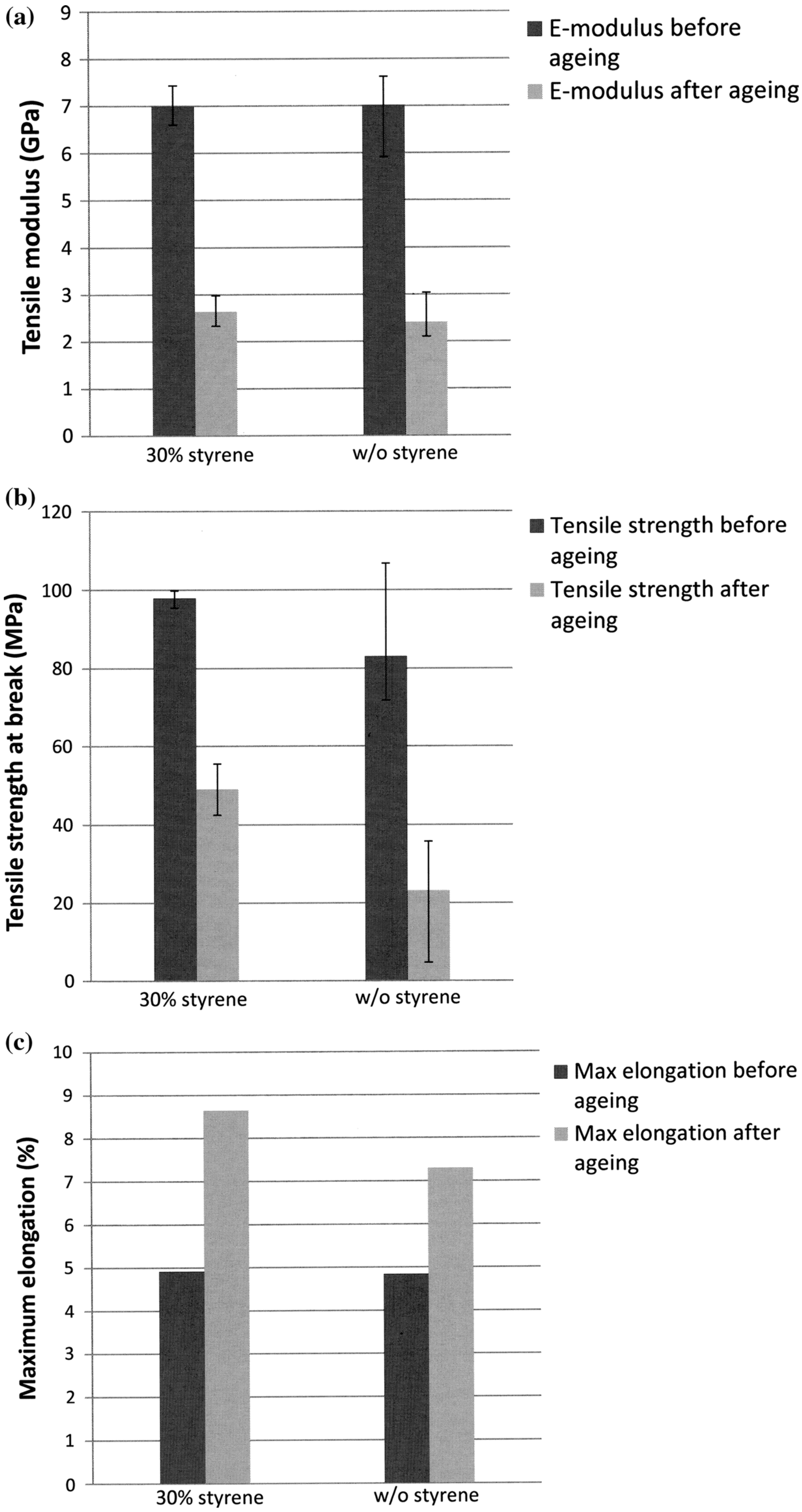


Fig. 6 **a** Storage modulus of neat resin and UD, BD and NW type composites with 65 wt% fiber load. **b** Tan delta of neat resin and UD, BD and NW type composites with 65 wt% fiber load. **c** Peaks of tan delta of neat resin and the composites

Fig. 7 **a** Tensile modulus of BD type composites with 65 % fiber load with and without styrene before and after ageing in climate chamber. **b** Stress at break of BD type composites with 65 % fiber load with and without styrene before and after ageing in climate chamber. **c** Tensile elongation of BD type composites with 65 wt% fiber load with and without styrene before and after ageing in climate chamber



from 25.75 to 6.25 N/mm^2 in the UV radiation ageing test and 4.34 N/mm^2 in the saline fog test (Duta and Cazan 2011).

Due to the evident effect of the ageing on the composite, the possibility of modifying the ageing behavior of the resin and the composite were investigated. This was done by using styrene as a reactive diluent, and then the mechanical properties and ageing behavior of the composites were investigated. Styrene has been proofed to increase crosslinking density for some thermosetting resins (Abdelwahab et al. 2012). Fibers and resins are hygroscopic, that is, they can absorb and give off moisture in response to environmental humidity. Styrene is not affected by variations in moisture content in the environment, it remain stable even when subjected to large changes in humidity. The addition of styrene in the composite could help to reduce the hygroscopic nature of the fiber and resin.

Figure 7a, b and c presents the tensile properties for BD composites with 65 wt% fiber load with and without added styrene before and after ageing. The properties were measured after a 24 h reconditioning at room temperature. The tensile strength decreased dramatically after ageing but adding styrene considerably improved the composite's ageing properties and also improved tensile max stress before ageing. Figure 7b shows that ageing affect tensile stiffness of the composites significantly but adding styrene preserved a small portion of the loss. Figure 7c shows that maximum elongation increased after ageing, this could be due to the presence of water in the sample, even after reconditioning water could act as plasticizer.

Water absorption testing

The water absorption of a composite can be influenced by the characteristics of the reinforcement and the matrix, but could also be influenced by the reinforcement volume fraction and porosity (Saw et al. 2012). Water absorption of a composite could also be due to direct diffusion of water molecules between the polymer chains or between the reinforcement and matrix interface. Figure 8 shows the variation of the percentage water absorption with increasing number of days for BD type composite with 65 % fiber load with and without styrene. The water absorption in the composites with and without styrene increased rapidly by 7 % by day 2, but for the composite without styrene, there was also a rapid increase between day 2 and day 7 of about 3 %, but a small increase between day 7 and day 40. For the composite with styrene, there was about <1 % increase in the water absorption between day 2 and day 40. Adding styrene to a thermosetting resin can increase the cross-linking density. Thus, it will be more difficult for water molecules to penetrate the resin. This explains why the composite with styrene is more stable against water uptake and this is also in s agreement with the mechanical properties. After drying of the composites, there was a rapid decrease in the water uptake of the composite without styrene up to about −3 % of its initial weight before it was placed in the climate chamber. This implies that some of the lactic acid-based resin had been hydrolysed while adding styrene to the resin counteracted the hydrolysation of the polyester resin.

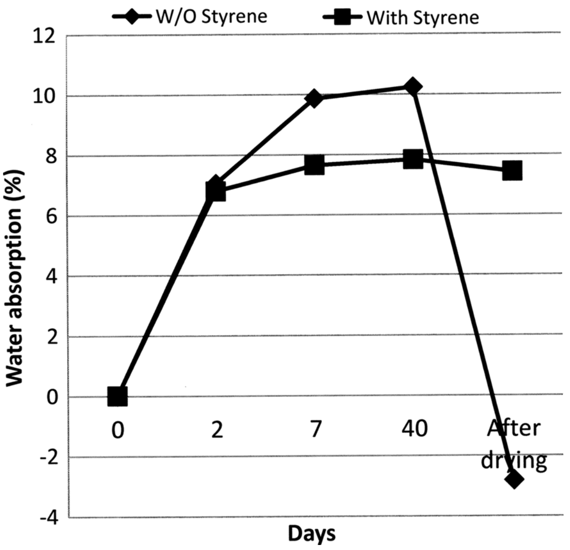


Fig. 8 Water absorption of BD type composites with 65 % fiber load with and without styrene

Conclusions

In this study, the possibility of using a novel thermosetting resin prepared from lactic acid and glycerol as a matrix for regenerated cellulose fiber reinforcements was investigated. The resin was impregnated into the regenerated cellulose fiber and compression molded at elevated temperature to produce thermoset type composites, with different alignments (unidirectional and cross wise oriented), different reinforcement type (warp-knitted and non-woven) and different fiber loads (65, 70 and 75 wt%). The results indicates that the glycerol-lactic acid-methacrylic acid resin could be used as matrix for cellulosic fibers with fiber load up to 70 wt% fiber load when using the UD and BD type warp-knitted reinforcement, but 65 wt% fiber load when using the NW type reinforcement. The composites produced from the resin would yield good mechanical properties. Considering the high renewable ratio and relatively low price for the cellulosic fibers, this resin shows promising properties for making strong affordable composites with a high renewable content.

The composites showed relatively good flexural, tensile and impact strengths. The flexural modulus ranged between 10 and 11.5 GPa for UD composites, 5 and 6.5 GPa for BD composites and 5 and 6 GPa for NW composites. The tensile modulus ranged between 11 and 14 GPa for UD composites, 7 and 8.5 GPa for BD composites and 5 and 7.5 GPa for NW composites. While the impact strength ranged between 130 and 150 kJ/m^2 for UD composites, 98 and 110 kJ/m^2 for BD composites and 17 and 20 kJ/m^2 for NW composites. These results show that the composite has comparable mechanical properties with many commercial petroleum based GFR polyester composites.

Ageing experiments showed that during the chosen conditions, ageing deteriorates the mechanical properties of the glycerol-lactic acid-viscose composites but the addition of styrene somewhat counteracts the degradation. The composites absorb considerable amount of water which is also responsible for their ageing properties. Sealing the composite with a water proof coating could prevent deterioration of their mechanical properties in exposure to moist conditions. However, the composites should mainly be used for indoor applications.

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