

Preparation of Thermoset Composites from Natural Fibres and Acrylate Modified Soybean Oil Resins

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ABSTRACT: Structural composites with a high content of renewable material were produced from natural fibres and an acrylated epoxidized soybean oil resin. Composites were prepared by spray impregnation followed by compression moulding at elevated temperature. The resulting composites had good mechanical properties in terms of tensile strength and flexural strength. Tensile testing as well as dynamical mechanical thermal analysis showed that increasing the fibre content, increased the mechanical properties. The resin can be reinforced with up to 70 wt % fibre without sacrifice in processability. The tensile modulus ranged between 5.8

and 9.7 GPa depending on the type of fibre mat. The study of the adhesion by low vacuum scanning electron microscopy shows that the fibres are well impregnated in the matrix. The aging properties were finally evaluated. This study shows that composites with a very high content of renewable constituents can be produced from soy bean oil resins and natural fibres. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 114: 2502–2508, 2009

Key words: soybean oil; natural fibres; thermosets; compression moulding

INTRODUCTION

As environmental concern becomes a major issue, the use of petroleum derived materials is getting more and more questioned and many researchers have consequently turned their attention to alternatives. This is also the case for structural composite materials. These are currently reinforced with glass fibres, aramid fibres or carbon fibres, which are of nonrenewable origin, and composites made from biobased raw materials are therefore targeted. Natural fibres such as flax, hemp, wood or sisal have many documented advantages such as biodegradability, low weight and less abrasive to the production equipment. Natural fibres are generally not as strong as glass fibres but the specific strength of natural fibres can be well compared to glass fibres. The use and properties of composites reinforced with natural fibres have been reviewed by many authors, for example by Mohanty et al.¹

Both traditional petroleum based resins^{2–4} as well as biobased resins^{5–8} have been combined with the natural fibres. Unsaturated polyester and epoxy resins are today very versatile and can be adapted for a

wide range of technical applications. However, the use of these resins with natural fibres will create composites which are only partially based on renewable constituents. Obviously, it is wishful to use resins which are synthesized from renewable raw materials, and vegetable oils, such as soybean and linseed oils are such candidates. Vegetable oils are cheap, abundant and renewable. They have a long use in the society both as food and for technical use. These oils are chemically triglycerides, i.e. esters of glycerol and fatty acids. The fatty acids can be either saturated or unsaturated. The latter contains reactive double bonds which can be utilized in various ways to obtain technically usable resins. The double bonds undergo slow oxidative cross-linking, which has been utilized in drying paints since centuries, but the reaction is too slow for modern industrial purposes. The double bonds in the fatty acid does not for example readily cross-link upon radical initiation, which is a very common method today for resin crosslinking in a composite.

Chemical modification of the triglyceride molecule is needed to obtain reactivity, and there are several routes for this.⁹ Epoxidization of the double bonds in unsaturated fatty acid is especially common, both epoxidized soybean oil (ESO) and epoxidized linseed oil (ELO) have attracted researchers attention. For example, Boquillon produced composites from hemp fibres and an anhydride cured ELO.¹⁰ Evaluating the resulting composites, the author found that the flexural modulus increased up to 5.6 GPa when

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reinforcing the resin with 28 vol % fibre. Crivello prepared composites from glass fibre and epoxidized vegetable oils, using UV-curing.¹¹ Nanocomposites from epoxidized vegetable oils has been studied as well.^{12,13}

The epoxidized natural oils can be further modified by the reaction of acrylic acid to form acrylated epoxidized oils. These oils contain pendant acrylic double bonds which will readily undergo an addition reaction and are very suitable for producing structural thermoset composites. Acrylated epoxidized soybean oil (AESO) is commercially available and AESO resins have been used for the preparation of composites. For example, Scala and Wool studied the acrylation of various natural oils at different levels of acrylation,¹⁴ Hong and Wool prepared composites from avian fibres and an AESO resin.¹⁵ Dweib et al. produced by vacuum-assisted resin transfer moulding composites from various fibre mats, AESO resin and styrene.¹⁶ In another study, Thielemans and Wool studied also composites prepared from kraft lignin.¹⁷ Hybrid composite between glass/flax fibre and AESO resin was reported by Morye and Wool.¹⁸ Nanocomposites have also been prepared from AESO resins. For example Thielemans et al. prepared carbon nanotube reinforced AESO composites¹⁹ and composites produced from AESO and layered silica has been reported as well.²⁰

AESO can also serve as a starting material for further synthesis. For example Wool et al. reacted an AESO resin with maleic anhydride. Such a resin can be used for sheet moulding compound applications.^{21,22}

Commercial available synthetic resins are frequently diluted with reactive comonomer, such as styrene. There are several reasons for doing so. First, blending the resin with a reactive comonomer will lower the viscosity. This will enhance the processing, allowing the use of closed moulding techniques as vacuum bag infusion or resin transfer moulding. Secondly, copolymerizing the resin with a compound like styrene will generally have a positive effect on the mechanical properties. The introduction of the stiff aromatic ring in the structure will influence both the mechanical properties as well as glass transition temperature. Reactive comonomers have also been utilized together with natural oil resins. Wool and coworkers examined the influence of blending the AESO resin with different levels of styrene by dynamical mechanical thermal analysis (DMTA).²³ They found that both the storage modulus and the glass transition temperature increased with an increase level of styrene. However, adding styrene will lower the content of renewable material in the final composite. Additionally, styrene is a very volatile compound which easily evaporates during processing in open moulds, and it is there-

TABLE I
Characteristics for Used Natural Fibre Mats

Natural fibre mats	Surface weight
Air laid flax mat. Supplied by Linapellava Oy, Finland	450 g/m ²
Air laid hybrid mat with 90% flax and 10% polyester. Supplied by Linapellava Oy, Finland	460 g/m ²
Sheets of dissolving pulp from Domsjö Fabriker AB, Sweden	940 g/m ²
Air laid hemp mat, supplied by Laroche SA, France	400 g/m ²
Coarse hemp mat, containing residual stem parts. Supplied by Laroche SA, France	900 g/m ²

fore an advantage if styrene is not used as a comonomer.

In the present study, we have examined the possibility to produce structural composites from natural fibres and an AESO resin without the addition of any reactive comonomers. The resulting composites will therefore be of high renewable raw material content, which is targeted today. To be of technical use, such a composite must however be able to compete with current commercial composite materials in terms of mechanical properties, processing, price and availability. These aspects were especially studied and evaluated. A commercial AESO resin was used and various natural fibre mats were spray impregnated and subsequently cured by compression moulding. Producing structural composites from natural fibre and an AESO resin without any use of styrene has to our knowledge not been done before.

EXPERIMENTAL

Materials

AESO, Tribest S 350-01, was obtained from Cognis Deutschland GmbH & Co. The initiator was 1,1-di-(*tert*-butylperoxy)-cyclohexane (Peroxan PK 122 W, Pergan), and 1 wt % of the peroxide was blended with the Tribest resin. Sheets and fluff of dissolving pulp were delivered by Domsjö Fabriker AB in Sweden. Air laid hemp mats were obtained from Laroche SA in France and air laid flax mats were obtained from Linapellava Oy in Finland. The characteristics of the fibre mats are given in Table I. A glass fibre reinforced unsaturated polyester composite with 40 wt % randomly oriented chopped glass fibres was used a reference. This composite was produced and delivered by Plastic Technology Composites AB in Sweden.

Impregnation

Composites were prepared by a spray impregnation method. The fibre mats were cut either to the size 15 × 15 cm for the flexural and impact test laminates or to the size 20 × 30 cm for the tensile test laminates. All fibre mats were dried in a vacuum oven for 1 h at 105°C at roughly 40 mbar before use. A conventional air driven, hand held, spray gun for spraying paints was utilized. To reduce the viscosity and to facilitate the spray impregnation, the resin was kept in a water bath at 50°C. The lamination was done on a tray, placed on a balance. The cut fibre mats were put on the tray, and then the resin was sprayed on the fibre mat surface with the spray equipment. The weight of each fibre mat was recorded before and after impregnation to control the fibre content. Two or three fibre mats were assembled and the impregnated fibre mats were then compression moulded at 160°C for 6 min, by using a hydraulic hot plate press. This gave laminates with the thickness of about 3 mm. The applied pressure was between 1 and 4 MPa. For the composites with 70 wt % fibres, the pressure was 4 MPa. For composites with lower fibre ratios the pressure was decreased in order not to squeeze out the resin. The fibre content varied between 30 and 70 wt %.

Characterization

DMTA (Perkin Elmer) was performed on the composites. DMTA was run in the dual cantilever bending mode and the sample dimensions were: thickness 1–2 mm, length 50 mm and width 8 mm. The temperature interval was –10°C to 150°C with a heating rate of 5°C/min and using a frequency of 1 Hz and a strain of 0.1%.

The tensile properties were evaluated in accordance to ISO 527. The dumbbell shaped test bodies were 150 mm long, and were milled with a cutter. The test bodies were tested on a MTS 20/M, using a crosshead speed of 50 mm/min. A minimum of five test bodies were tested for each material.

The composites were characterized by three-point bending tests with a MTS 20/M. A crosshead speed of 10 mm/min and a distance between the holders of 40 mm was used. A minimum of five test bodies were tested for each material.

The Charpy impact strength of un-notched specimens was evaluated in accordance with ISO 179 using a Zwick test instrument. The test pieces were tested edge wise. A total of 10 test pieces were tested to determine the mean impact resistance.

To study the adhesion between the fibre and the AESO matrix, test bodies for fibre pull-out test were prepared by casting the resin and fibres in 150 mm long dumbbell shaped Teflon moulds. The test

TABLE II
Tensile Properties at Different Fibre Ratios

Fibre mat	Fibre content (wt %)	Tensile strength (MPa)	Modulus (GPa)
Air laid flax	70	78 (±14)	9.7 (±0.9)
Air laid flax	60	71 (±26)	9.7 (±2.8)
Air laid flax	50	61 (±7.8)	6.3 (±1.2)
Air laid flax	40	50 (±6.4)	5.0 (±0.5)

The standard deviation is given in the brackets.

bodies had a fibre content of roughly 10 wt %. Two fibres were used, flax roving and unbonded fluff of dissolving pulp, these corresponded to the reinforcements used in the composites. The specimens were then broken in the tensile strength testing machine, and the obtained fracture surfaces were studied in a low vacuum scanning electron microscope (Hitachi S300N).

The durability of the composites was evaluated by a climate conditioning test. Test bodies for tensile testing were placed in a climate chamber at 38°C and 95% relative humidity for 1000 h. The tensile properties were evaluated before the test, directly after the test and after 24 h reconditioning at room temperature. The tensile tests were performed in accordance to ISO 527 as described above.

RESULTS

Tensile testing

The tensile properties were measured for the laminates containing air laid flax mats with a fibre content ranging from 40 to 70 wt %. The air laid flax mat was chosen, as it was assumed that this reinforcement would be the most difficult to impregnate. The results are shown in Table II. Both the tensile strength and the tensile modulus increased with an increased fibre content, which can be assigned to a very good adhesion between the fibre and the matrix. The composite with 70 wt % fibre content gave a high stiffness, 9.7 GPa, which was expected. Higher fibre content gave composites with the fibres unevenly impregnated, these were therefore discarded without any further investigation. Too high fibre content will create voids between the fibres, resulting in poor mechanical strength, due to insufficient stress transfer to the fibres.

The tensile properties of composites made from the various fibre mats with the maximum fibre content were then evaluated, see Table III. Two different flax mats, one hemp mat and the dissolving cellulose sheets were evaluated. The air laid flax mat gave the best mechanical properties of all tested fibre mats. The air laid hybrid mat gave significantly lower values. This mat contains a polyester fibre binder which

TABLE III
Tensile Properties for Composites Prepared From Different Fibre Mats

Fibre mat	Fibre content (wt %)	Tensile strength (MPa)	Modulus (GPa)
Air laid flax	70	78 (± 14)	9.7 (± 0.9)
Coarse hemp	70	41 (± 8)	5.8 (± 1)
Air laid flax-PET hybrid	70	51 (± 9)	6.0 (± 0.7)
Dissolving pulp sheet	70	65 (± 4.8)	6.7 (± 0.5)

The standard deviation is given in the brackets.

mechanically glues the flax fibres together into a rigid structure. In the cured composite, the polyester fibre was observed as hard porous parts. A possible explanation for the lower tensile strength is that the polyester fibre creates fracture sites in the composite, hence causing lower values. Both the tensile strength and the tensile modulus of the hemp mat were significantly lower in comparison to the best flax mat. The hemp mat is a rather coarse, low priced mat which contains rough parts from the stem of the plants. This can cause fracture sites, explaining the lower values. Also, the composites made from sheets of dissolving pulp gave relatively high values, which are probably due to the dense pulp sheet structure, which it self is rather strongly bonded together.

Flexural testing

The flexural properties were evaluated using several different fibre mats, see Table IV. The sheets of dissolving pulp gave clearly the best results. This material differs from the plant fibre mats as it consists of rather dens sheets of strongly bonded dissolving pulp. The cured composite will therefore resemble a layer-like laminate, with cellulose sheets bonded together by the resin. When performing the tensile testing, these layers in the laminate delaminated, causing lower tensile values. In the flexural mode, the fracture behaviour is obviously different, and

TABLE IV
Flexural Properties For Composites Prepared From Different Fibre Mats

Fibre mat	Fibre content (%)	Stress at peak (MPa)	Modulus (GPa)
Air laid flax	70	98 (± 5.3)	6.9 (± 0.8)
Air laid flax-PET hybrid	70	72 (± 24)	5.3 (± 2.0)
Coarse hemp	70	46 (± 8.2)	3.7 (± 0.5)
Air laid hemp	70	58 (± 21)	3.7 (± 1.0)
Dissolving pulp sheet	70	140 (± 10.2)	7.2 (± 0.8)
Glass fibre	40	207 (± 17.7)	5.5 (± 0.7)

The standard deviation is given in the brackets.

good flexural strength is obtained. The air laid flax composite gave a very good flexural strength while the flax-PET hybrid mat gave poorer strength. This corresponds well with the results from the tensile testing.

The hemp mats gave slightly lower values than the flax mats which also was the case for the tensile testing. The glass fibre reference composite had as expected the best flexural strength compared to any of the natural fibre composites. The flexural modulus of the natural fibre composites can however be compared with the modulus of the glass fibre composite. In some cases the modulus of the natural fibre composites was even higher. It should also be noted that the fibre content of the glass fibre composite is lower than for the natural fibre composites. Obviously, such a comparison can only give a very rough picture of the performance of these natural fibre composites in comparison to glass fibre composites.

Dynamical mechanical thermal testing

Dynamic mechanical thermal analysis is a technique to apply a stress to a material and measure its response. Since polymers are viscoelastic to their nature, the analysis will give information about the storage modulus and the loss modulus. As the storage modulus is conceptually equivalent to the modulus of traditional mechanical testing, it gives a measurement of the stiffness of the material.

Composites for DMTA analysis were produced from the air laid flax mat, with fibre ratios ranging from about 30 to 70 wt %. This reinforcement was chosen for the DMTA analysis as it gave the best tensile strength. The results are shown in Figures 1 and 2 and tabulated in Table V. The storage modulus increased from 3.2 GPa for the composite with 30 wt % fibre up to 7.4 GPa for the composite with 70 wt % fibre. This strong increase of the modulus

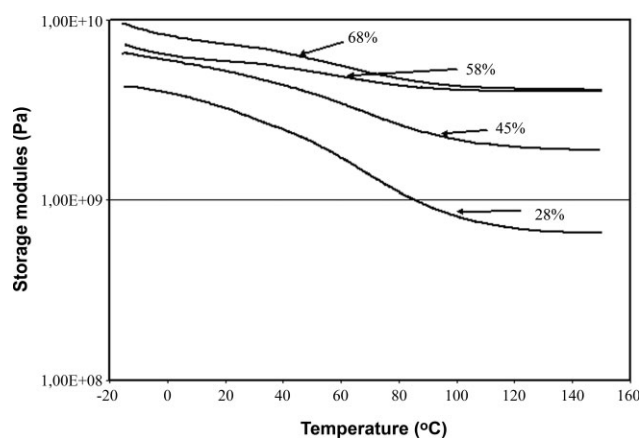


Figure 1 Storage modulus at different fiber contents.

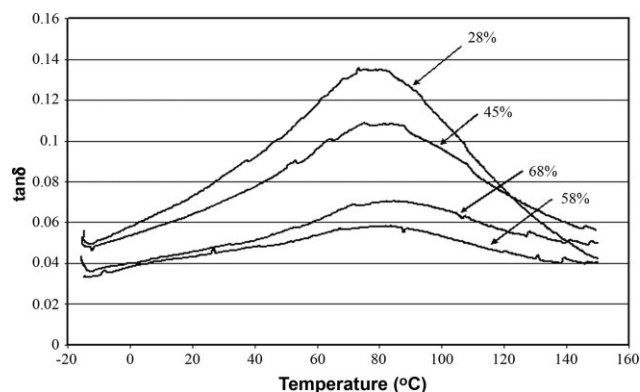


Figure 2 Tan δ at different fibre ratios.

can be interpreted as a good adhesion between the fibre and the matrix.

The dampening, or tan δ , is the ratio between the loss modulus and the storage modulus and gives information about the internal friction of the material. For a composite the molecular motion in the interface will contribute to the dampening. The dampening will consequently give information about the adhesion of the interface. There is a general trend in Figure 2 with decreased tan δ values with higher fibre ratio, though the sample of 60 wt % fibre deviates. A decreased peak of the tan δ value with an increased content of the reinforcement has been observed for various reinforcements by others authors as well.^{24–26} A possible explanation is that there is a strong interaction between the fibre and the matrix. For a composite with a low fibre ratio, the polymer chains are relatively freer to move. Increasing the fibre ratio will decrease the mobility of the polymer chains and consequently lower the dampening. This was discussed by Pothan et al.²⁶

The peak of the dampening takes place in the region of the glass transition where the material changes from a glass to a rubbery state. The glass transition temperature is often recorded at the maximum of the tan δ . Using this method of determining the glass transitions temperature, the recorded T_g of the composites ranged from 73°C for the lowest fibre ratio up to almost 85°C for the highest fibre ratio. The increasing of T_g with increasing fibre ratio is a

TABLE V
Summary of DMTA Results For Air Laid Flax Composites with Varying Fibre Content

Fibre content (%)	Storage modulus (E') at 20°C (GPa)	Loss modulus (E'') at 20°C (GPa)	T_g (°C)
68	7.4	0.34	84.6
58	5.9	0.26	81.7
45	5.2	0.34	75.0
28	3.2	0.24	73.2

TABLE VI
Impact Resistance

Fibre mat	Fibre content (%)	Impact resistance (kJ/m ²)
Air laid flax	70	19.7 (± 2.8)
Air laid flax	60	18.8 (± 3.2)
Air laid flax	50	17.0 (± 2.0)
Air laid flax	40	13.4 (± 1.4)
Dissolving pulp sheet	70	20.3 (± 2.8)
Glass fibre	50	56.0 (± 6.7)

The standard deviation is given with brackets.

rather unusual behaviour but it has been observed by other authors as well. For example Aranguren and coworkers²⁷ reinforced a linseed oil based resin with 30 wt % wood flour. A very strong increase of the glass transition temperature was observed by DMTA, this was attributed to a good adhesion between the fibre and the resin. A strong interaction between the fibre and the matrix will restrict the mobility in the of the polymer chains in the interface region.

Impact resistance

Impact resistance is the ability of a material to resist a sudden load, and is related to the toughness. For many composite applications the toughness is an important property, and it is also known that natural fibre reinforced composites have poorer toughness than glass fibre reinforced. The impact resistance was determined for air laid flax mat specimens reinforced with 40 to 70 wt % and the dissolving pulp sheet composites, see Table VI. The impact resistance increased with increased fibre content as expected, the composite made from dissolving paper pulp gave a very similar result as the flax composites. However, comparing to the reference glass fibre composite, the natural fibre composites gave a clearly lower result. It is obvious that the biocomposites cannot compete in this respect.

Low vacuum scanning electron microscopy

The fracture surfaces from the pull-out test were analysed by low vacuum scanning electron microscopy, see Figure 3. Fibres of similar quality as in the mats were used in these tests. The fracture surface is clean, with no fibre pull in the dissolving pulp composite fracture surface, and the fibre seems well distributed in the matrix. The adhesion between the cellulose fibres and the matrix is therefore very good. The fracture surfaces of the composites made from flax roving shows longer fibres with some fibre pull-outs. This indicates that the adhesion between the matrix and the fibre is not optimal.

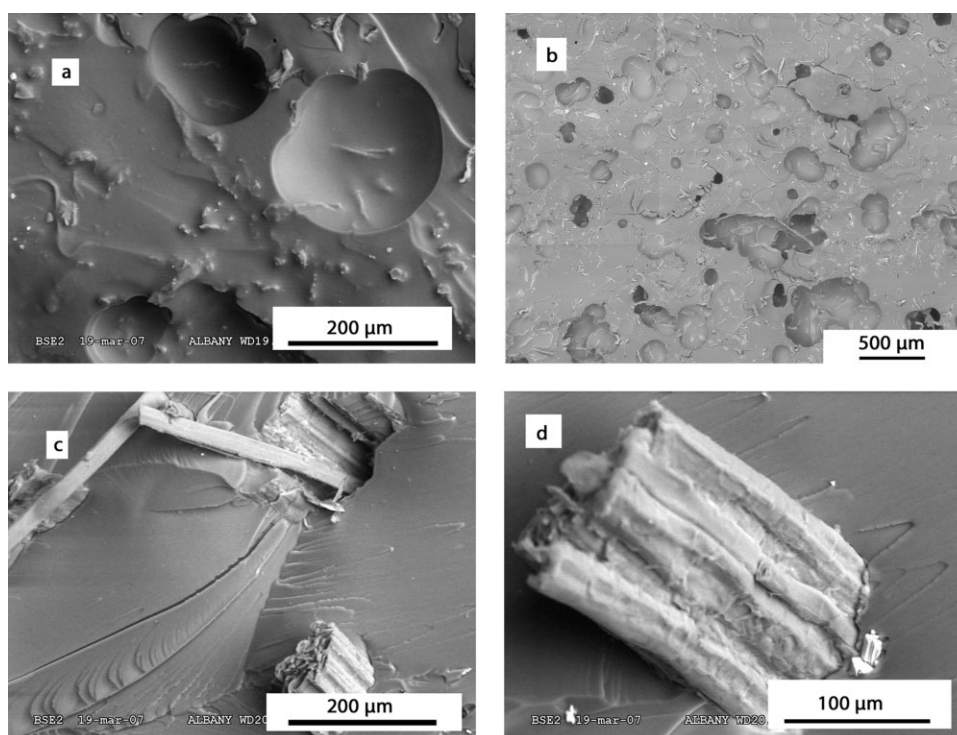


Figure 3 Low vacuum SEM micrograph of the fracture surface for composites made of dissolving pulp (a and b) and air laid flax (c and d).

Climate conditioning tests

Test bars for tensile testing were kept in a climate chamber at 38°C and 95% RH for 1000 h. The result is shown in Figure 4. Natural fibres are known to absorb moisture due to hydrogen bonds between water and the hydroxyl groups in the cellulose. The absorption of water is reflected by the mechanical properties. After 1000 h, the composites lost less than 30% of their mechanical properties. Recovering the test pieces for 24 h, the composites regained some of its mechanical strength.

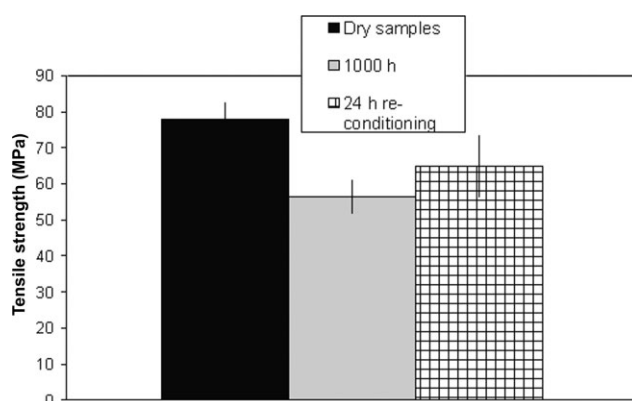


Figure 4 Aging test of flax fibre reinforced composites.

CONCLUSIONS

Structural composites from natural fibres and an acrylated epoxidized resin were produced with a high content of renewable material. The resin was applied on the natural fibre mats by spraying, and cured by heating in a hydraulic press. The results show that it is possible to produce composite with high mechanical properties without adding to the resin a reactive comonomer like styrene. Both tensile testing and DMTA show that with increasing the fibre content, increased mechanical properties are observed, as expected. This suggests that there is a good adhesion between the resin and the matrix. It is possible to use a fibre content as high as 70 wt %. This is advantageous for several reasons. First, a high fibre ratio will enable high mechanical properties as already mentioned. Secondly, natural fibres are generally low priced. Thus, high fibre ratio will lower the price of the resulting composite. Finally, since the resin contains some nonrenewable material from the resin synthesis, the higher fibre content, the higher is the amount of renewable material in the final composite.

These natural fibre composites cannot compete in terms of mechanical properties compared to a conventional glass fibre composite. Especially the impact strength is poor for the natural fibre composites, as the glass fibre composite yielded more than

the double value compared to the flax composite. However, these natural fibre composites are not so far behind when comparing the flexural properties. Natural fibre composites are known to be sensitive to humidity due to the hydrophilic nature of the fibres. The aging test verified this; therefore, the main applications will be in indoor products.

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