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Wood Fibre Composites with High Fibre Content

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WOOD FIBRE COMPOSITES WITH HIGH FIBRE CONTENT

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ABSTRACT

The aim of this project has been to explore an idea concerning the production of thermo-forming wood fibre composites. They have high fibre content and are produced by applying papermaking technology. The main objectives have been to answer the following questions:

- Can these wood fibre composites be made with standard papermaking equipment?
- Can a composite produced in this way compete with the presently available natural fibre composites?

The results show that it is fully possible to make composites with 80% wood fibres by using papermaking technology and that they can compete with or excel over commercial products in terms of stiffness and tensile strength.

Keywords: Dimensional stabilization, Fibres, Latex, Mechanical properties, Natural fibres, Polymer matrix composites, Thermo-mechanical pulp fibre, Wood composite

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1. INTRODUCTION

The use of natural fibres as reinforcement in composite materials is very old. Centuries ago when natural fibres were the only available fibres they were used, for example, together with clay to make bricks. After decades of high-tech developments of artificial fibres such as carbon, glass fibres and Kevlar, the focus has now interestingly been changed back to natural fibres. This renewed interest in natural fibre composites reflects itself in several reports and literature reviews[1-6].

The driving forces to use natural fibres as reinforcement material today are environmental friendliness, lighter weights, lower costs and their interesting strength properties. These interesting attributes make natural fibre composites one of the most rapidly growing segments (estimated to approximately 25 % annually over the past several years) in the composite industry [7].

Rapid growth is always a strong driving force for research and industrial development. The Mid Sweden University is situated in a region with strong traditions in papermaking and wood fibres. If papermaking technology and wood fibres could be used to produce composites it could have a very positive influence on the industrial development in our region.

The aim of this project has been to explore an idea concerning the production of thermo-forming wood fibre composites with papermaking technology. The main objectives have been to answer the following questions:

- Can wood fibre composites be made with standard papermaking equipment?
- Can this composite compete with the presently available thermoplastic composites?

After the introduction this report summarizes the initial tests made on this composite material. Focus has been on processing, tensile strength and dimension stability of the composite material.

1.1 Composites

Most fibre composites are made of only two phases[8,9], one continuous phase called the matrix and one dispersed phase called the reinforcement. The matrix often consists of a polymer that binds together either a continuous web or dispersed short fibre as load carrying elements. In this report only natural fibre composites are considered although some comparison with glass fibre composites are made.

There are four major issues to resolve in order to make a composite successfully and cost efficiently:

- The process to make the composite
- The fibre properties
- The matrix properties
- The adhesion between fibre and matrix

Below are our choices of process, fibres and matrixes presented. The question of adhesion between fibres and matrix is not dealt with in this report, but this is studied in other projects at the Mid Sweden University[10] and it has recently been reviewed[11]. Before introducing our chose of system a short introduction of natural fibre composites will be presented.

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1.2 Natural fibre composites

The present largest application of wood fibre (i.e. mainly wood flour) composites is concentrated to outdoor applications such as decking, fencing and garden structures. They are referred as wood fibre polymer composites (WPC). These composites contain high amounts of moisture resistant matrix (usually polypopylene) to withstand the different weather conditions they will be exposed to. The fibre content is in general much lower than 50 %. The dominant manufacturing technology has been extrusion where the ability to make complex shapes is limited to the profile of the detail. Other materials containing a high wood fibre content such as particleboard, medium density fibreboard (MDF) and hardboard can also be classed as composites, but these panel structures are hard to manufacture at low grammages and complex shapes and has the low-cost construction industry as a niche[12]. WPC:s, MDF:s and particle boards are not further dealt with in this report.

This report concentrates on the current applications for natural fibre reinforced thermoplastics, mainly as a lightweight substitute for glass fibres. This type of natural fibre composites have already successfully found applications in the automobile industry for example in door trims and panels. The stiffness per weight is higher for natural fibres compared to glass fibres. Good acoustic damping and insulation properties, less wear of tooling, easier recycling and lower health risks are other benefits. The surfaces of natural fibres consist of fairly reactive polysaccharides and phenolic polymers that can be used to enhance the bond between matrix and fibre[13-15]. Natural fibres have rough surfaces that promote adhesion whereas glass fibres have a smooth and less reactive surface making the bonding to the matrix more difficult. Drawbacks with natural fibres are their sensitivity to moisture, non-uniform fibre sizes and length and that they are not suitable for high temperature applications.

The commercial natural fibre mat thermoplastics (NMT) have not used wood fibres to any large extent. Consequently earlier investigations [16-22] had the focus been on plant, leaf and fruit fibres such as hemp, flax, jute, kenaf, coir and coconut and not much attention has been paid to wood fibres[23]. In this investigation, wood fibre has been used as natural fibre reinforcement. Wood fibres have advantages over other natural fibres: The price is relative low and stable and the fibres are available in larger quantities. The production of wood fibres was in 1999 about 300 million tons whereas the production of other natural fibres such as silk, ramie, flax, hemp, jute, sisal and coir all together was about 5 million tons the same year[24]. The quality of wood fibres is reasonably constant since paper makers have developed the production of quality fibres from wood for centuries. Plant fibres however must often undergo so-called retting to separate the bast fibres from the rest of the stalk. The process is time consuming and weather dependent. The process must be monitored closely to reduce loss in quality[25]. In addition, wood fibres are available in a lot of semi-finished materials such various durable fibre mats and different types of paper.

The major drawbacks using wood fibres are their swelling tendency in moist conditions and their short fibre length[8,9]. These are the major reasons for their restricted use as reinforcement in composites today. The plant fibres mentioned above have longer fibres than wood in general or fibre bundles are used, but analogous to wood fibres, these fibres are also sensitive to moisture. The swelling of fibres influence the dimension stability negatively and the fibre length is believed to be an essential property to achieve the required strength of the final composite. On the other hand short fibres are often more homogeneously distributed in the composite structure and wood fibres have a high aspect ratio, which is positive.

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1.3 The processes to make composites

There exists many different techniques to processing fibre composites. Filament winding, extrusion blow moulding, injection blow moulding, resin transfer moulding and compression moulding are few examples. However, most of the present technologies can use only less than 50 % of fibre content by weight[26]. For thermoplastics solution impregnation, powder impregnation/pultrusion and film stacking are the most commonly used tehchnologies[2]. Hot pressing to produce a prepreg usually follows these processes.

We have chosen impregnations of a fibre web with a size press and follow by drying and compression moulding in one or several layers. Size presses are standard papermaking equipment and has since long been used in the industry. Making composites with a size press is not a new idea. What makes our approach different from previous investigation is our choice of fibre and matrix material.

Impregnation of fibre web is used commercially in for example flooring and decorative laminates. It has recently been used to investigate wood fibre composites with high fibre contents in a Ph.D. thesis[27]. They found that strong composites could be made with this technique, but they could not reach fibre content above 70%. They also had problems obtaining fully impregnated paper even though the paper was impregnated up to 24 seconds. Others[28] have also impregnated natural fibres and come to the conclusion that it is of utmost importance to have viscosity of the impregnating liquid that is sufficiently low.

Impregnation of fibre webs in a size press is an efficient and well establish process. As mentioned above it does put some restraints on the fibre material and the matrix you can use. The fibre web needs to absorb the liquid evenly and quickly, evenly to get best possible material properties and quickly in order to get production efficiency. The web needs to be strong enough to pass through the press and the matrix needs to be of sufficiently low viscosity.

1.4 Fibre properties

The main reason to add fibres to composites is to improve their mechanical properties. Accordingly they need to be stiff and strong. Kraft fibres are considered to be the strongest wood based fibres[29] and are more or less exclusively used in paper product developed for their strength, such liner and sack paper. Figure 1 reveals that mechanical pulps tend to form lower density sheets and even though the figure shows the strength of sheets rather than the strength of the individual fibre it can be argued that at the same density mechanical pulps can have comparable strength properties to kraft pulps.

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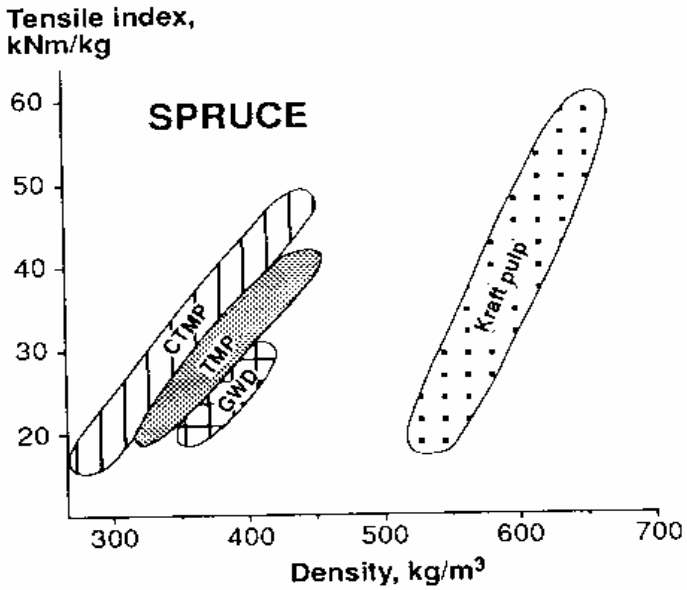

Figure 1. Tensile index of mechanical pulps compared to kraft pulp. Strength increases with density and if the mechanical pulps were to be compared at the same density as the kraft pulp similar tensile strength can be expected. Figure taken from[30]

The strength of different wood pulps in vinyl ester composites was investigated[31]. Kraft pulps gave the stronger composites than mechanical pulps. However, the relative difference in composites strength between kraft pulp and mechanical pulps were much less than their fibre strengths. This indicates that mechanical pulps have a good potential as reinforcement in composites.

The second important property for the fibre web is its absorbency. The DRF material we have chosen is specially designed to absorb liquids in hygiene products and it is available in reels. Compared to kraft paper the difference in absorption is very large, see below.

Other benefits of chemo thermo mechanical pulp (CTMP) compared to kraft pulp are the price which is lower and that the kraft pulp fibres are more slender and have large elongation, which make them less suited in composites. In addition, mechanical pulps are more affected by heat treatment and will become a part in the thermo-forming process.

The properties of composites are usually well described by the rule of mixtures[8]. That is their properties are linearly dependent on the properties of its constituents and their weight ratio. Paper often contains small amounts of polymers and can be considered as a composite. However, paper does have a third phase: Air (voids) and it does not follow the rule of mixtures until the voids have been filled[32]. This is illustrated in figure 2. It can be seen that at approximately 20% to 100% of resin (matrix) there is a linear dependence of the strength properties, but below that there is a large deviation.

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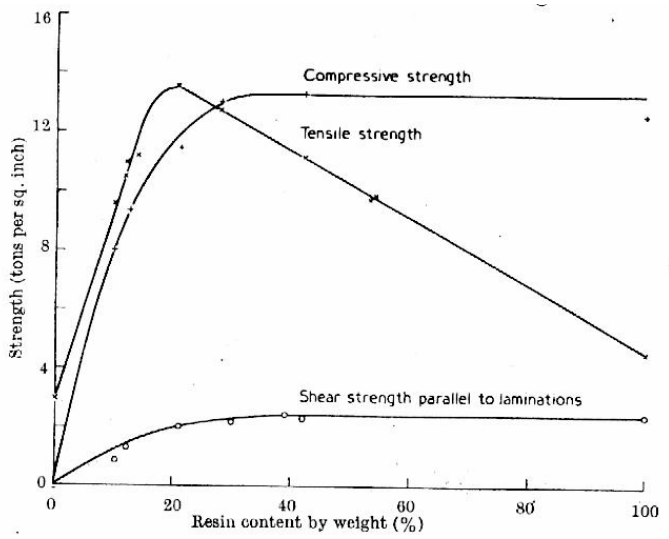

FIG. 3

Figure 2. At fibre contents up to approximately 80 %, the strength properties increase. To efficiently utilize the fibre properties a fibre content of approximately 80 seems adequate. Figure taken from [32].

In the composites made in present study 80% fibre is mostly used. In this way the fibres are, according to figure 2, used efficiently for strength. The cost of the raw materials can be low (fibre material is less expensive the matrix material) and the composite can be seen as more environmental friendly by the higher use of renewable material.

1.5 Matrix properties

The type of matrix used in this study is not conventional; instead dispersion polymers (latexes) were used. Dispersion polymers are known to be an effective binder for paper[33] and they do not only give "strength" but also ductility. The polymer is dispersed in water and no harmful solvents are used. Since the fibres are well wetted by water latexes can be used in a size press. The water adsorbing properties of the fibre mats are thus utilized by absorbing the matrix as a dispersion. In this way the matrix can be spread more evenly and deeper in to the fibre mat making it possible to use such high fibre contents as 80 % and the polymer does not need to be melted at high temperatures to penetrate the fibre mat. The properties of the latex used can be varied in a very wide range, giving a ridged and stiff composite material or a ductile and more flexible one.

In theory, to utilize properties from both fibre and matrix, all fibres must be completely surrounded by the matrix to optimal transfer the load between the matrix and the fibre[26]. The theoretical maximum volume fraction of perfectly aligned fibres is 90 %. The maximum volume fraction is lower for randomly aligned fibres such as those used in the present trials.

To summarise: We have chosen a size press, CTMP pulp webs and dispersion polymers to make wood fibre composites. Our rationales for this are:

- The size press is a well-proven and efficient production unit.
- Composites with high fibre content can be produced and the fibre content can be varied from less than 50% up to 100%.
- High fibre content means environmental friendliness and better economy: Less petroleum base products (matrix) are used and fibres are less expensive than matrix materials.

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- High fibre content means that the fibre strength is utilized efficiently.
- CTMP fibres are highly absorbent thus the matrix (latex) is evenly distributed in the fibre web.
- CTMP fibres are used because they are strong and commercially available in large even qualities.
- Dispersion polymers were chosen because they have low viscosity and because they have water as "solvent".
- Dispersion polymers are thermoplastic and their properties can easily be varied and they are commercially available in a variety of formulations.
- This process can produce thin composites that easily can be made into layered products.
- Complex structures can be made from the material.

2. MATERIALS

2.1 Fibres

In this work the reinforcement fibres are chemo thermo mechanical pulp (CTMP) sheets made from dry formed reels (DFR). The DFR is a low density fibre mat that can be seen as a semi-manufactured component to the final composite material. The DFR material has been kindly donated by SCA Östrand. The making of a dry formed reel is conceptually a fairly simple process. Dry CTMP fibres fall onto a conveyor wire and the fibres orientation is relatively isotropic having similar properties in the machine and cross direction. However, an anisotropic pattern is calendered on to it to improve strength properties. The DFR material is developed to take up as much fluid as possible when exposed and is presently exclusively used as an absorber in hygiene products.

2.2 Matrixes

Three styrene acrylate copolymer latexes with different Tg:s (glas transtion temperatures) and one vinyl acetate homopolymer latex were used as matrixes. These latexes are commercially available and were kindly donated by Celanese AB. Some properties of the latexes are shown in table 1. The latexes all contained a cross linking agent that is activated at temperatures above 100°C.

Table 1. Polymer matrix properties

Brand name	Polymer base	Tg* [°C]	pH	MFFT** [°C]	Particle size [µm]
Mowilith TA 2210 S	Styrene acrylate copolymer	-20	3 – 5	0	0,15 – 0,25
Mowilith TA 2250 S	Styrene acrylate copolymer	20	3 – 5	20	0,15 – 0,25
Mowilith TA 2270 S	Styrene acrylate copolymer	60	4 – 5	59	0,15 – 0,25
Mowilith TH 210 S	Vinyl acetate homopolymer	32	3 – 4	16	0,10 – 0,30

* Glass transition temperature
**MMFT, Minimum Filming Form Temperature

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3 METHODS

3.1 Composite preparation

The composites were prepared in two steps. First the matrix was applied to the fibre web in a size press, secondly the impregnated web was thermoformed, that is pressed under heat.

3.1.1 Matrix application

The latex solution was first diluted from the original 50% solid content to approximately 25%. The diluted latex was less viscous than undiluted enhancing its ability to penetrate the reinforcing fibre mat and to spread more evenly. The latex was applied to the DFR by dipping 30 x 36 cm sheets in the centre of a size press, where the latex solution had been added. The size press consists simply of two rolls (700 mm long and 100 mm in radius) with its axis in horizontal position. Pressure is applied and the rolls are squeezed together along the axis. In the space between the rolls, at the upper side, the impregnation fluid (latex) is added. A motor is spinning one of the rolls and the contact pressure makes the other roll to spin in the opposite direction. When sheets are placed between the rolls the fluid penetrate the fibre web and the pressure of the rolls controls the uptake. Each sheet was run twice through the size press. The process is schematically illustrated in figure 3. This technique could be used to continually impregnate large amounts of reeled DFR in a process application. The sheets where dried at room temperature. The latex uptake was calculated by weighing the sheets before and after the latex application procedure. The latex content after impregnation was always 20 % if nothing else is stated.

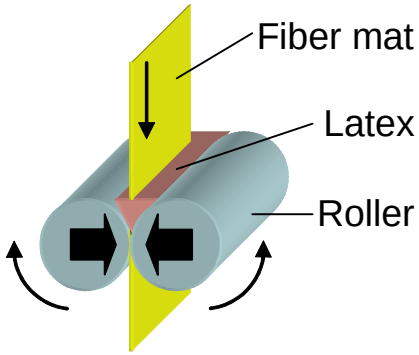

Figure 3. Schematic picture of the latex impregnation process

The pinch roller pressure and rotation speed was parameters that were adjustable in the size press. The pressure was set to 7 bar and the speed was set to 12 Hz (equal to 5 RPM or 3 m/min). The water content in the DFR sheets was around 40 % after the two impregnation steps in the size press.

3.1.2 Pressing

The impregnated sheets were pressed in two layers in a planar press. In the press the composites were compressed from about 3.25 mm in original thickness to about 0.7-1.0 mm depending on settings of the press. The influence of press settings on tensile strength was investigated by varying temperature, dwell time and pressure. The temperature and time of the press could easily be adjusted without limitation in the interesting region. The pressure was however not adjustable. In those trials in which the pressure had been altered, steel plates with different contact area were

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used or the hydraulic motor was halted at the desired pressure. The different press settings used in this work is displayed in table 2.

Table 2. Press settings

Variable varied	Temperature [°C]	Time [min]	Pressure [bar]	Latex type	Tg of latex [°C]
Temperature	20, 40, 70, 100, 130, 200	5	90	Stryrene acrylate Copolymer, no latex	-20
Time	130	0.5, 1, 3, 5, 7, 10, 15, 20, 30	90	Vinyl acetate homopolymer	32
Pressure	130	5	30, 40, 50, 65, 80, 90, 180	Vinyl acetate homopolymer	32
All variables	130, 200	5, 15	90, 180	Stryrene acrylate copolymer	-20, 20, 60

The time was started when the desired pressure had just been reached.

3.2 Tensile strength tests

3.2.1 Composite and DFR strength

Composite tensile strength was measured with a Lorentzen & Wettre tensile strength tester commonly used testing paper in the paper industry. 15 mm wide samples were cut from the manufactured composites. The samples were subjected to 50% RH and 23°C for at least 48 hours before testing. 10 test strips of each sample were measured. The span between the holding clamps was 100 mm and the pull rate was 12 mm/min. The same apparatus and method were used testing the DFR sheets without matrix.

3.2.2 Polymer matrix strength

Test strips, 10 x 80 mm, of the three styrene acrylate copolymer latexes were made (Tg -20, 20 and 60). The Tg 20 and Tg 60 latexes were manufactured by firstly drying the latex in room temperature. Then they were crushed to a fine powder and passed through a sieve with 0.5 mm holes. The powder was poured into 10 x 80 mm open tracks of a copper plate. Beneath and above the copper plate were heat stable polyester plastic films. This package was then subjected to 130 °C and 30 bar in a press for 10 minutes. The plastic film was then removed and the moulded latex test strips were cut out or broken loose from the copper plate. The latex with Tg -20 could not be crushed to powder in a practical way so this latex was manufactured by pouring directly in to the copper tracks and dried to a film before pressing. This method did not give as smooth a texture as using powdered latex, but was satisfactory. All latex test strips were tested on a MTS tensile testing machine. The span between the holding clamps was set to 20 mm and the pull rate was 1 mm/min. Due to high elongation of the Tg -20 latex, the span between the holding clamps was lowered to 5 mm so the test specimen could break before the maximum clamp distance was reached in the testing machine. Also the pull rate was increased to 10 mm/min at this Tg to save time. The tensile tests were performed at 23 °C and 50 % RH.

3.2.3 Composite strength at elevated temperatures

The tensile strengths of the composites were tested at elevated temperatures. These tests were made on a MTS tensile testing machine with 80 mm span between the clamps and the composite

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test strips were 15 mm wide. The load rate was 12 mm/min. A heating oven that surrounded the clamping area was used to increase the temperature of the composite specimens during tensile load. The temperatures were set from 20 to 130 °C with 20 °C steps. The humidity could not be measured. The composite test strips were subjected to the selected heat at least 10 minutes before testing. The composites used in this trial were manufactured somewhat differently than before. The DFR sheets were not passed through the size press, but instead dipped by hand in 25 % latex and then dried at room temperature. The latex used was the styrene acrylate copolymer type with Tg 60. The composites were manufactured by pressing at 90 bar, 130 °C and 5 minutes.

3.3 Curing

The effect on strength by curing the composite test strips (15 x 150 mm) was investigated. The curing was made in a regular oven. First the cure temperatures were set at three levels, from 105 to 180 °C while keeping the cure time constant at 30 minutes. Secondly, the cure time was altered from 10 to 120 minutes at a temperature where the largest effect on the tensile strength was obtained in the first trial. The composites used had been pressed for 5 minutes and 130°C at 90 bar. Styrene acrylate copolymer latexes were used in the trial.

3.4 Latex mixtures

Trials with mixtures of latexes were preformed using the styrene acrylate copolymer with Tg -20 and 60 respectively. The Tg 60 latex was mixed with the Tg -20 latex and the tensile strengths were tested. The DFR/latex sheets where pressed to composites at 130°C and 90 bar for 5 minutes.

3.5 Water uptake

Test strips (215x23 mm) were cut from different composites containing styrene acrylate latex with Tg -20, 20 and 60 °C. The dimensions were measured after the test strips were exposed to five different conditions in the following order: 1. Room temperature and ambient moisture; 2. Oven 105°C for two hours; 3. 23°C and 50% RH for 24 hrs; 4. 23°C and 90% RH for 24 hrs and 5. soaking in water for 24 hours. The weight, length and thickness were measured at all conditions. The weight was measured by weighing on a standard laboratory scale with four decimals, length was measured with a ruler and thickness was measured with an L&W thickness test apparatus. The water uptake of pure latex films was also tested. Weights of latex test strips were measured before and after soaking in water at regular intervals for a couple of months. The latex test strips were manufactured according to 3.2.2.

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4. RESULTS AND DISCUSSION

All data shown below can be found tabulated in Appendix 1.

4.1 Polymer matrix and fibre tensile strength

Large differences in tensile strength of the pure styrene acrylate latexes were observed. The glass transition temperatures (Tg) of the latexes seem to determine the strength properties. Low Tg gives a weak and rubbery polymer whereas high gives a stiffer and brittle one. The results of the tensile tests of the latexes are found in table 3. The test specimens were manufactured as described in 3.2.2.

Table 3. Tensile strength of styrene acrylate latex films with different Tg

Tg [°C]	Density [kg/m^3]	Tensile strength [MPa]	Modulus [MPa]	Strain [%]
60	975 ± 16	9.43 ± 1.11	1260 ± 80	1.1 ± 0.2
20	944 ± 24	5.55 ± 0.36	11.5 ± 2.5	231 ± 9
-20	1063± 52	0.54 ± 0.13	0.19 ± 0.02	429 ± 189

A hard and brittle polymer with Tg well above the temperature of testing, such as polystyrene, poly(methyl methaacrylate) and many phenolformaldehyde resins have a typical modulus of about 3500 MPa and tensile strength of about 70 MPa. The typical value for strain is about 2 %. Soft and tough polymeric materials such as plasticized PVC and rubber (elastomers) have low modulus and moderate tensile strength and very high elongation ranging from 20 to 1000 %[34]. These values give a indication that our strength measurements on the styrene acrylate polymer are in the region that can be expected.

The tensile strength of the DFR fibre mat without matrix was tested. The results are shown below in table 4.

Table 4. Tensile strength of CTMP fibre mats

Fibre mat	Density [kg/m^3]	Tensile strength [MPa]	Modulus [MPa]	Strain [%]
DFR, unpressed	506	1.5 ± 0.1	196 ± 23	2.0 ± 0.2
DFR, pressed*	613	2.0 ± 0.1	233 ± 26	2.2 ± 0.5

** pressed at 130 °C, 90 bar, 5 min*

The tensile strength of the fibre mat does however describe the bonding strength of the fibre web rather than strength of the individual fibre. If the fibres in the composite material were completely

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covered with matrix, the strength of the individual fibres would better describe the strength contribution. These data could also be used to, for example, stiffness predictions of the composite at different fibre loadings by the "rule of mixture" approach. However, at 80 % fibre content as used in the present study, all fibres are probably not covered with matrix and some fibre-fibre bonding is likely present and thus these calculations could be misleading. Nevertheless, the fibre mat strength should at least give some information of matrix performance. See also figure 5.

The glass transition temperature of the latex used as matrix has rather a large influence on the properties of the final composite strength. The effects are seen below in figure 4.

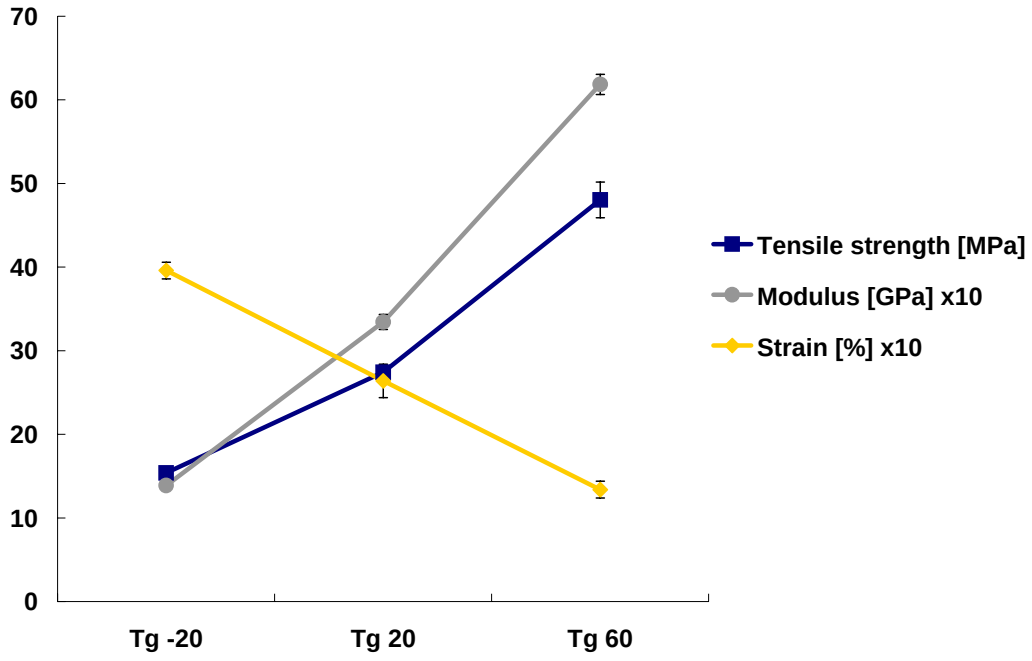

Figure 4. Mechanical properties of composites containing 20% of different styrene acrylate copolymers as matrix. (press settings: 200 °C, 15 min, 180 bar)

Comparing the strength of the composite with the strength of the matrix and fibre, it is clear that the fibres act as reinforcement to the matrix and the properties of the latex influences the strength of the composite. Alternatively it can be described that the matrix reinforces the fibre web to increase elongation. It has been claimed that the elongation of the matrix should be equal to the elongation of the fibre[18]. Consequently the Tg60 have the right order of magnitude and the Tg20 and Tg-20 too much strain (cf. table 3 and 4).

4.2 Effects of press settings

The most appropriate pressing conditions are when the viscosity of the matrix polymer is sufficiently reduced without thermally degrading the composite. In general, insufficient heat, pressure and time will result in poor spreading of the matrix and a high void content in the final material. The temperature, time and pressure are believed to affect composite formation mainly in the three ways

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1. Softening of the latex matrix and the reinforcing CTMP fibres
2. Filling of cavities in the composite structure by fibre densification and matrix diffusion
3. Cross-linking and film forming of the latex and cross-linking matrix-fibre molecules

Soft and low viscous latex matrixes are more efficiently spread in the porous fibre web during compression moulding. Heat is believed to be most important parameter to make the latex soft and viscous. This is desired since the best transfer of load between reinforcing fibres and matrix is achieved when all the fibres are covered with matrix and no fibre-fibre bonds are present when subjected to stress. The temperatures used are all well above the glass transition temperature of the matrix, exactly how viscous these films are when subjected to heat is however un clear.

The fibres becomes softer at higher temperatures, the lignin and hemicelluloses are plasticized at temperatures above 90 °C. This increases the molecular chain mobility and decreases the volume fraction of voids in the composite material together with the fact that soft fibres are easier to compress compared to stiff fibres. A small trial was made to examine the modulus on DFR fibre mat without matrix. The outcome of this trial is shown in figure 5

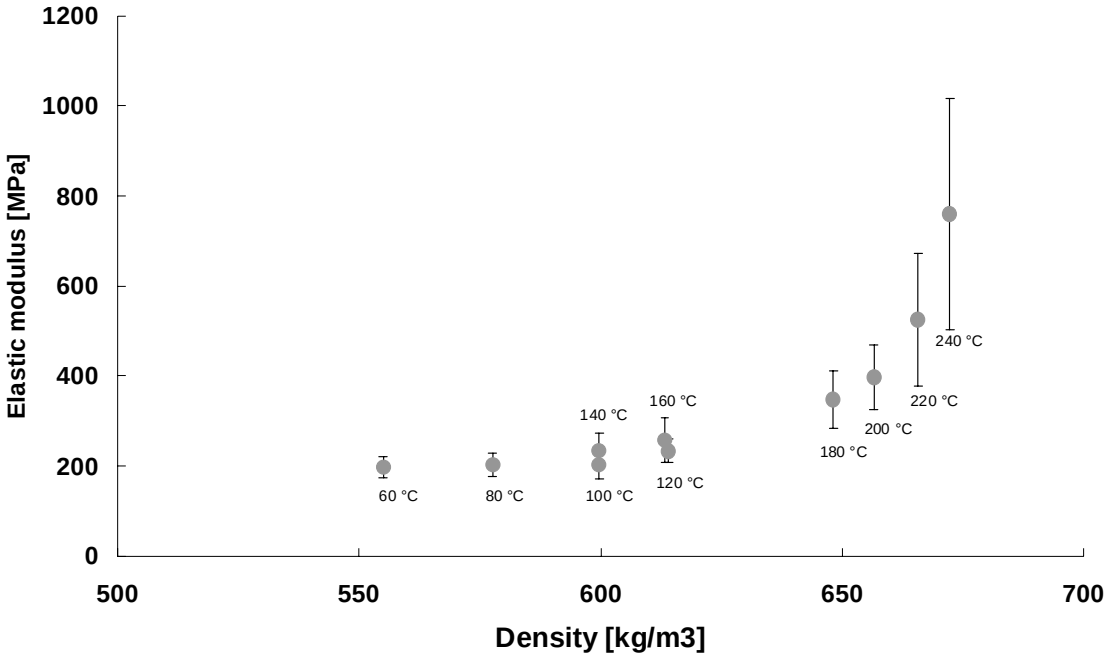

Figure 5. The elastic modulus of CTMP fibre web (DFR) pressed at 90 bar and 5 minutes at different temperatures

It is clear that the heat in the press affects the fibres themselves. Pressing at higher temperatures densifies the fibre web and increases its modulus. The fibre web gets darker at temperatures above 200 °C due to thermal degradation, however it does seem to affect modulus negatively.

The pressure is believed to be important for spreading of the matrix by squeezing it into small pores and voids and into the less accessibility areas. The compression of the fibres is controlled by the temperature and the pressure and it is time dependent. Since the softening of the matrix and fibres as well as the spreading of the matrix are time dependent, the time is an over all important factor.

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The latexes contain a cross-linking agent, which has the ability to cross link at temperatures above 100 °C. The cross-linking agent bridges the latex molecules together or cross link with the fibre surface making the matrix and composite more rigid and less flexible (at least in theory). These reactions are influenced by heat and time and should affect strength until a plateau level is reached. A cross-linked polymer obtains higher strength and stiffness, but becomes less flexible.

4.2.1 Temperature

From a production point of view it is desirable to be able to press at lower temperatures. Start-up heating time of the press, energy required to maintain the heat and thus money could be saved during composite manufacturing.

The first press setting trial was performed by varying press temperature. The main idea was to investigate if there was a minimum press temperature that could be used to obtain a sustainable composite product. Pressing at very low temperatures was not sufficient; the composite material delaminated when pressing at 20 and 40°C. At 70°C and above, the heat was enough to make a sustainable composite. The tensile results from this trial are shown in figure 4.

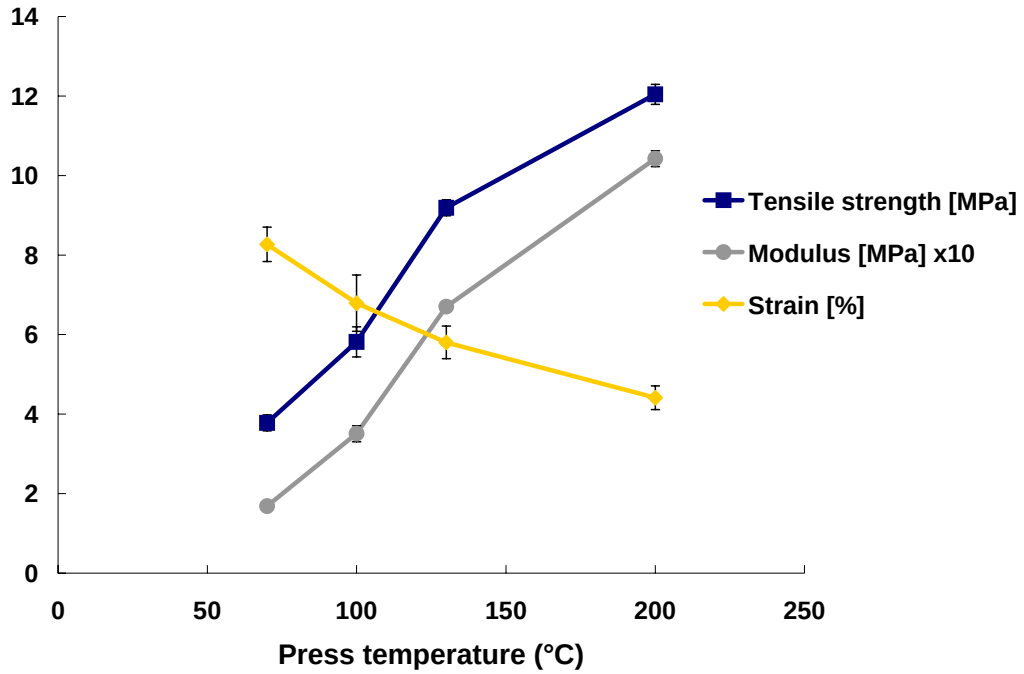

Figure 6. Composite tensile strengths at different press temperatures (Styrene acrylate, Tg -20 as matrix, pressed for 90 bar in 5 minutes)

As expected the press temperature showed to be an important variable when making composites by this type of compression moulding. The results showed that temperature has a rather huge influence on the strength of the final composite. The tensile strength increases more than 200 %, stiffness with more than 500 % and elongation decreases about 50 % at 200°C compared to 70°C.

Looking at figure 6 it seems tempting to press at even higher temperature than 200 °C to increase the strength even further. However, at too high applied heat, thermal degradation of the fibres will occur and we have avoided pressing at temperatures above 200 °C.

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The influence of press temperature on composites made with styrene acrylate, Tg -20 latex have been compared with those used above. As expected, the strength change is analogous with temperature for these latexes. The moduli for the different composites are shown in figure 7.

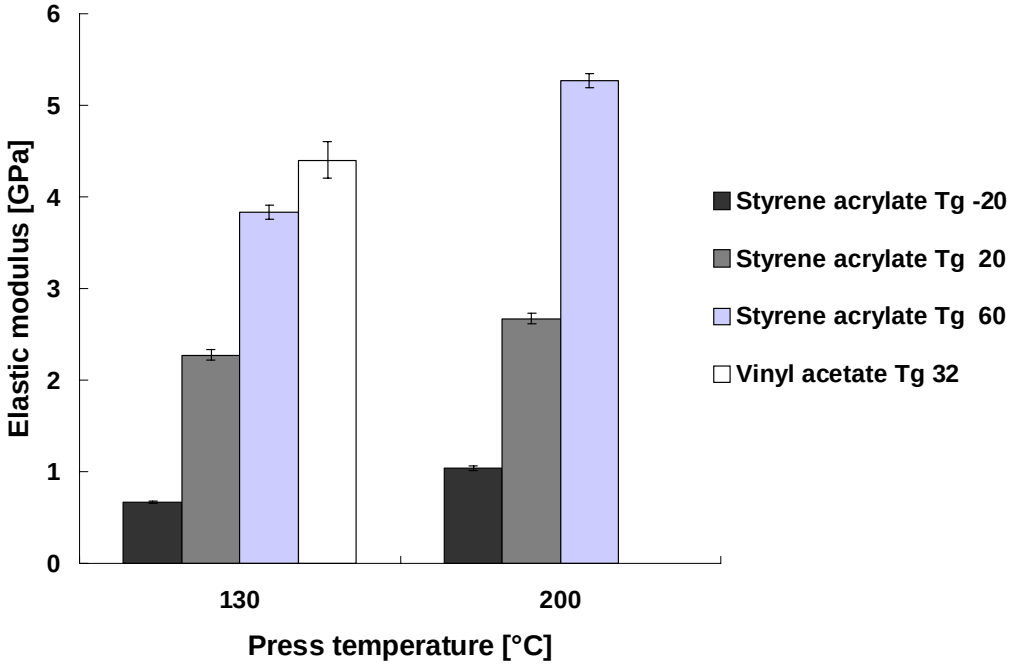

Figure 7. Composite elastic modulus with different matrixes and press temperatures. Composite consolidated at 90 bar and 5 minutes

It was not possible to manufacture a composite made of latexes with Tg 20, 32 and 60 at 70 °C as for Tg -20 probably because of high latex viscosity and poor fibre wetting at these temperatures of the latexes. It was also not possible to make a composite out of vinyl acetate at 200 °C due to thermal degradation of the latex.

4.2.2. Time

A fast rate of manufacturing is interesting to the composite producers. The time it takes to press the material should be kept short so it is possible to make large volumes in short times. In that way the cost of the end product will be lower. In this press trial, composites were pressed at different dwell times. Longer press times increased strength and stiffness to some extent as shown in figure 7, but decreased elongation.

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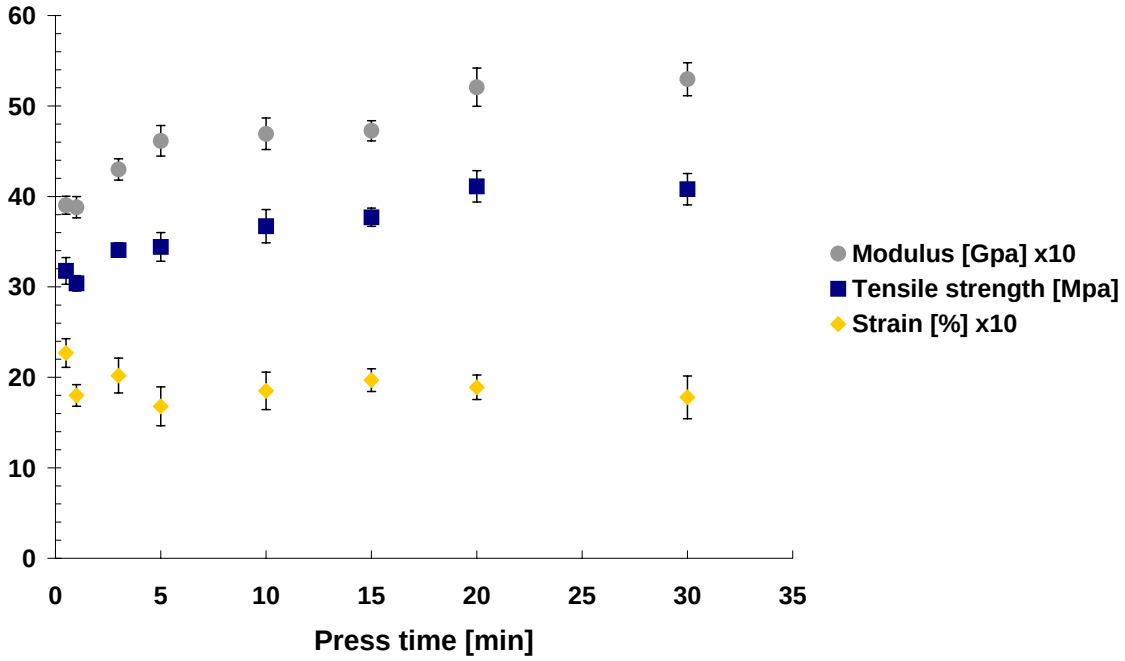

Figure 8. Mechanical properties at different press times. (Vinyl acetate, Tg 32, 130° C, 90 bar)

Interestingly, it was possible to press a sturdy composite in 30 seconds. The tensile strength of the composite pressed for 30 seconds was 31.8 MPa, elongation was 2.3 % and E-modulus 3.7 GPa. At a press time of 30 minutes, the strength was measured to 40.8 MPa, elongation 1.8% and stiffness 5.3 GPa. Longer times in the press increase the tensile strength and stiffness of the composite material, but decreases elongation.

Comparing the press time results obtained with the vinyl acetate latex with the styrene acrylate latexes in figure 9, it is clear that all the latexes behave in the same way regarding modulus of the composite.

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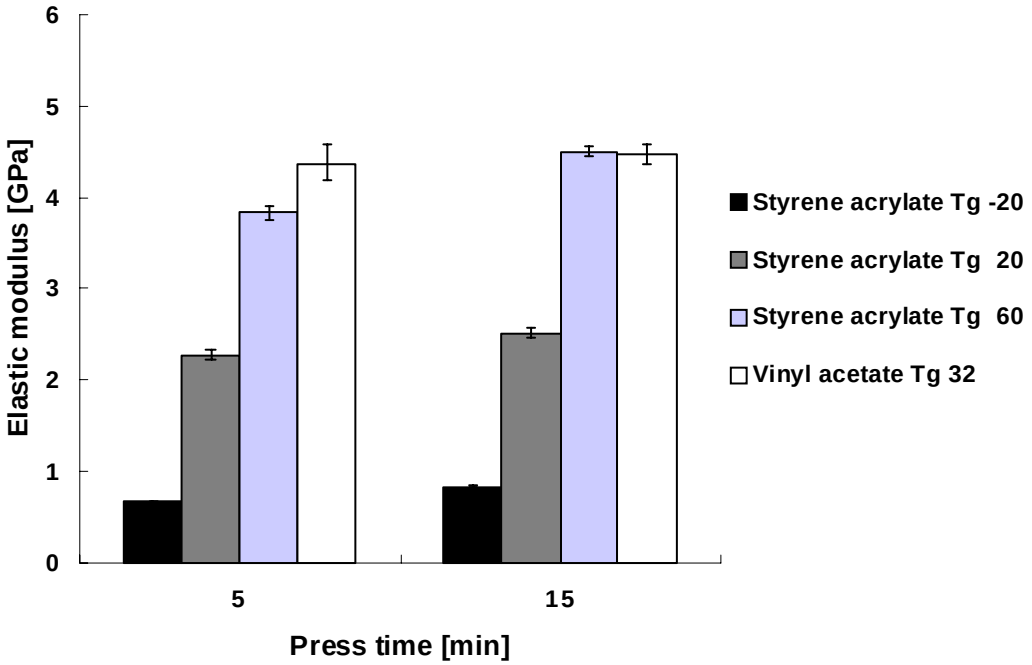

Figure 9. Elastic modulus of composites made of different latexes as matrix pressed at 5 and 15 minutes at 90 bar and 130 °C

Pressing 15 minutes compared to 5 minutes also increased the density slightly; 0,5 % for vinyl acetate and 1.5 % for Tg -20 °C, 4 % for Tg 20 °C and 4,5 % for Tg 60 °C for styrene acrylate latexes. This suggests that styene acrylate latex with high Tg need longer time to spread in the fibre network compared to styrene acrylate latex with lower Tg. The relatively low increase in density for the vinyl acetate matrix might be due to that this latex are of less viscous nature at 130 °C compared to the styrene acrylates making it easier to spread even though this polymer has a Tg of 32°C. This statement was however never verified by testing. Film forming rate can naturally also be a part of the results.

4.2.3 Pressure

The tensile properties of the composites were measured after pressing at different pressures. Interestingly, a relatively strong composite was achieved at pressures far below the standard pressure of 90 bar. Tensile strength and tensile stiffness index increased 10 % and 6 % respectively when pressing at 90 bar instead of 30. Strain at break decreased 20 %. The tensile strengths are presented in table 4.

Table 4. Tensile strength of wood fibre composites press at different pressures (vinyl acetate Tg 32 as matrix, pressed for 5 min. and 130 °C)

Pressure [bar]	Density [kg/m3]	Tensile strength [Mpa]	Strain at break [%]	Modulus [GPa]
30	978	28.7 ± 0.9	2.1 ± 0.2	3.5 ± 0,1
40	1006	30.9 ± 1.3	2.3 ± 0.2	3.7 ± 0,1
50	1042	31.9 ± 1.5	1.9 ± 0.2	4.0 ± 0,1
65	1051	28.7 ± 1.7	1.4 ± 0.2	4.0 ± 0,1
80	1074	34.7 ± 1.6	2.3 ± 0.2	4.1 ± 0,2
90	1077	33.7 ± 2.2	1.7 ± 0.2	4.4 ± 0,2

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It does not seem necessary to press at very high pressures to obtain a reasonably tensile strong composite. Density increases with increasing applied pressure as expected.

Styrene acrylate matrix composites were pressed at 90 and 180 bar at the same temperature and time as with the vinyl acetate matrix composite above. The outcome is shown in figure 10.

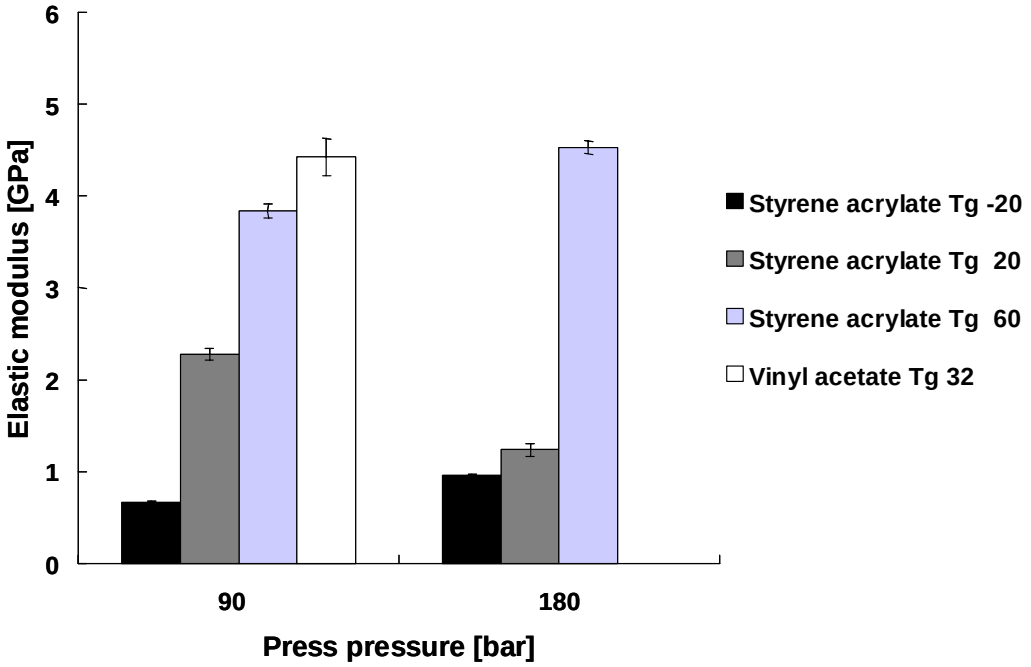

Figure 10. Elastic modulus of composites made of different latexes as matrixes pressed at 90 and 180 bar at 130 °C for 5 minutes.

4.2.4 All variables

The earlier press setting trials have all shown similar tendencies when it comes to tensile strength. Increased temperature, time and pressure all contribute to an increase in strength and stiffness whilst contributing to a decrease in elongation. A trial was performed to investigate the synergistic effect on these settings by increasing two or all variables at once. One result from this trial is found in figure 11 below where all variables have been varied.

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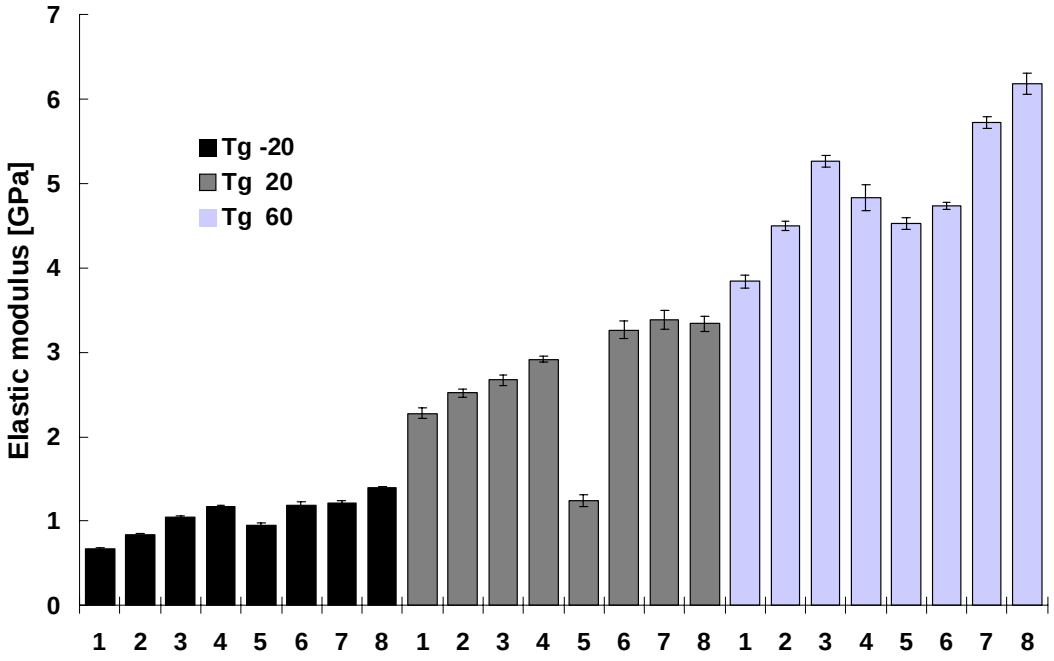

Figure 11. Change in elastic modulus when varying the press variables at two levels, alone or simultaneously, according to table 5 (left columns: Tg -20, centre: Tg 20, right columns: Tg 60.

Table 5. Press settings used in figure 9

	1	2	3	4	5	6	7	8
Temp [°C]	130	130	200	200	130	200	130	200
Pressure [bar]	90	90	90	90	180	180	180	180
Time [min]	5	15	5	15	5	15	5	15

Increasing all variables at the same time gave the best effect on tensile strength compared to changing only one parameter. Strength increased in analogy with stiffness. Elongation decreased more when varying all settings on the press. Increasing two variables at a time gave an effect that was larger than varying one, but less increasing all three.

Regarding tensile strength care should be taken when pressing the composites. Pressing at low temperature, time and pressure can give a lighter and more flexible composites compared to pressing at higher temperature, time and pressure where better strength and stiffness are obtained. However, the greatest changes in tensile strength are achieved by changing the type and Tg of latex.

To summarize the press setting trial, the effect of temperature, dwell time and pressure showed that all variables had a positive influence on tensile strength and stiffness, but that the elongation decreased. The temperature had the largest effect compared to pressure and dwell time on the composite strength in the ranges used. Increasing two variables at the same time gave a combination effect and highest strength and stiffness (and lowest elongation) was achieved by increasing all variables to their maximum tested value. It was clear that the break of the test strips

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from the tensile tester occurred almost exclusively in the checked pattern originated from the DRF sheets, this was probably due to a decrease in latex impregnation in these patterns.

4.3 Curing

Curing the composites with heat at different temperatures and times will activate the cross linking agent and create links between latex-latex and latex-fibre molecules. It will also affect the film formation of the latexes. The strength of the composite is affected by this treatment and the effects on tensile strength were investigated.

The impact on strength by curing the styrene acrylate composites after pressing was rather great. In the first trial, in which the cure time was constant and the cure temperature was altered, tensile strength and in particular elongation increased at higher temperatures. The stiffness however decreased. The relative increase in strength is shown in figure 12.

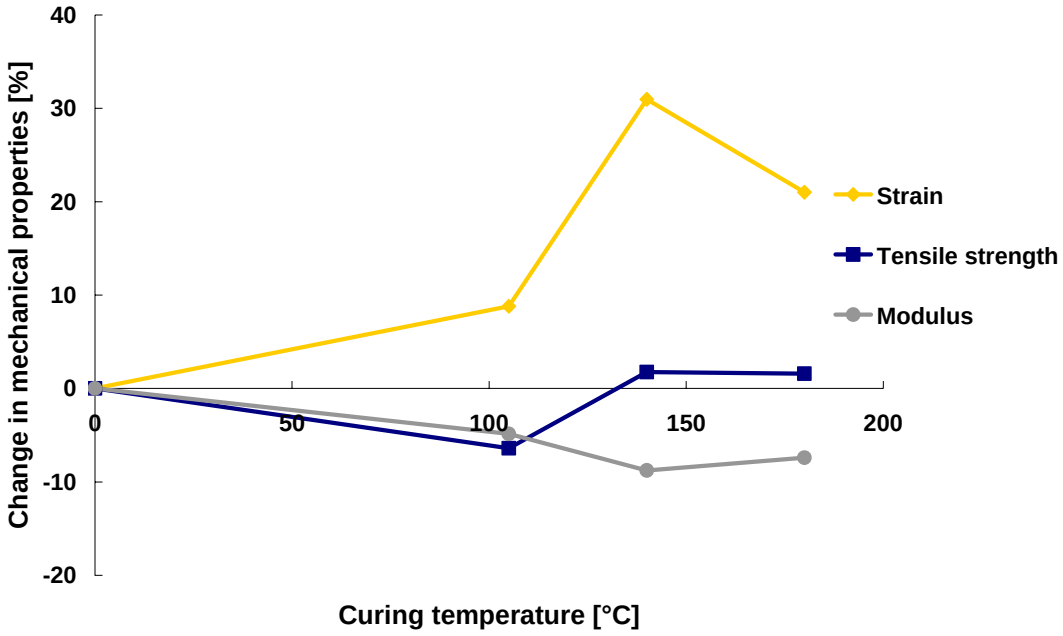

Figure 12. The relative change in mechanical properties after curing 30 minutes at different temperatures (Matrix: styrene acrylate Tg 60, 20 % content, consolidated at 130 °C, 15 min and 90 bar)

In the following trial, the cure temperature was set to 160°C and the cure time was changed. The effect of cure time is seen in figure 13.

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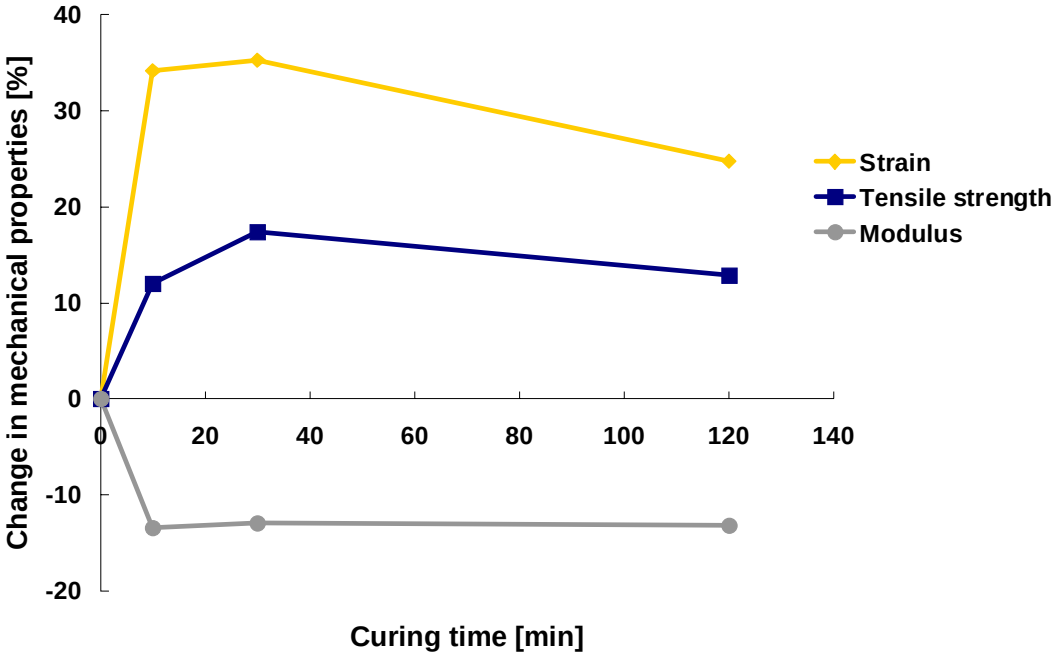

Figure 13. The relative change in mechanical properties after curing at different dwell times at 160°C (Matrix: styrene acrylate Tg 60, 20 % content, consolidated at 130 °C, 5 min and 90 bar)

Curing the composites at 160°C requires only short cure times to affect the strength. Already after 10 minutes a great change in strength is obtained. At longer cure times however, the effect declines.

To sum up, for this particular styrene acrylate composite in a 2 ply formation the largest influence occur at temperatures around 130°C and that happens pretty much initially. Elongation increases and stiffness decreases with curing. Note that in the press settings investigation the opposite was found. Elongation decreased and the stiffness increased at elevated temperatures and times during pressing. The reasons for this are not known. A relatively small decrease in density was obtained after curing due to an increase in composite thickness. This change is however too small to fully explain the change in tensile strength. It is likely that an increased film formation is part of the explanation.

4.4 Latex mixtures

Mixtures of latexes were used see if a dispersion containing one high Tg latex giving high strength and one low Tg latex giving high elongation, could be formulated. Less dusting and better durability of the dry semi-manufactured impregnated DFR sheets are other benefits by mixing latexes. A dry DFR sheet with a impregnated high Tg latex tends to dust much more than a latex with Tg below room temperature. Low Tg latexes form a film between the fibres in the DFR sheet and glue them together. High Tg latex does not form a film at room temperature and stays as powder in the loosely bonded fibre web and thus more dust are released both in form of fibres and latex. A small addition of low Tg latex to high Tg latex were therefore believed to decrease dusting while the strength properties of the higher addition where believed to dominate. Moreover, a small addition of low Tg latex could enhance the elongation of the unpressed DFR/latex sheets to some extent. This is believed to be helpful when later forming the material by compression moulding.

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A small addition (10%) of low Tg latex to a latex with high Tg gave a positive impact on the tested strength properties and the dusting was reduced (evaluated only by visual inspection). For a 50/50 mixture the strength and stiffness of the composite were closer to using only low Tg latex whereas elongation was similar to the high Tg latex. Thus using a mixture of 50/50 low/high Tg latex, only the less wanted properties from both are obtained (relative low strength and low elongation). This is illustrated in figure 14.

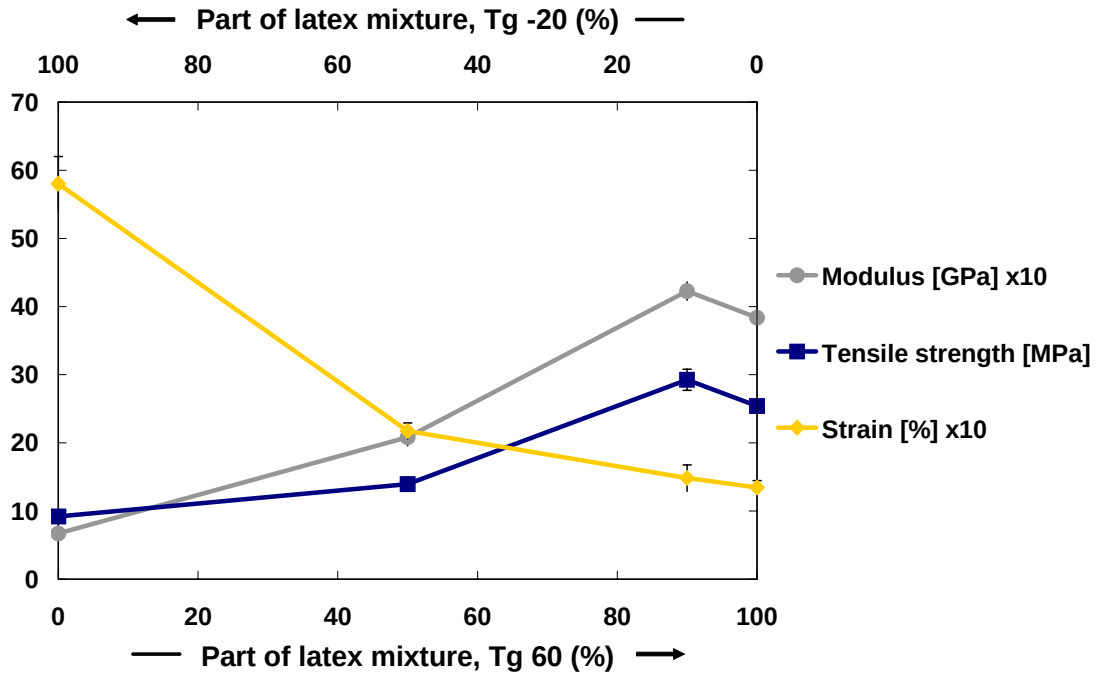

Figure 14. Strength properties of composites with mixtures of Tg –20 and Tg 60 styrene acrylate copolymer latexes (consolidated at 130 °C, 5 min and 90 bar)

The curves suggest that the latexes have a synergetic effect at low additions of the Tg -20 latex ti the high Tg latex. The results should however be investigated further before drawing to optimistic conclusions. At a 50 % portion the strengths are lower than expected i.e. a negative synergy.

4.5 Effect of water uptake

For almost all types of composites, resistance against water and moisture are important properties. The dimensional stability of the composites and the degradation by bacteria and fungi, especially for natural fibre composites, are affected by water. CTMP fibres contain much of the natural substances of trees that prohibit the attack of fungi compared to other wood fibre pulps. However, this does not help against negative effects on dimension stability caused by water. Wood fibres and air laid CTMP fibres, especially DFR fibre mats, are extremely sensitive to moisture. This sensitivity is used during the latex impregnation step to spread the latex solution evenly and readily in the fibre web. As comparison, the water uptake of the DFR fibre mat and a kraft liner (another type of wood fibre sheet) was measured by simply dipping in a water bath and measuring the weight before and after the dipping procedure. The water uptake of DFR was as high as 20 g/g fibre whereas the water uptake of the kraft liner was measured to 1.5 g/g fibre. In addition the water absorption rate is much faster for the DFR material even though the thickness and grammage of the kraft paper was lower. However, a drawback is that this sensitivity towards moisture gets harder to overcome by the finished composite product with such small use of matrix

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that has been used in these trials. Test strips made from composites were measured after being exposed to different conditions.

The latex films themselves absorb water to some extent depending on the type and the blend. A gravimetrical trial was made to examine the water uptake by films of styrene acrylate latex and the results from this experiment is seen in figure 15.

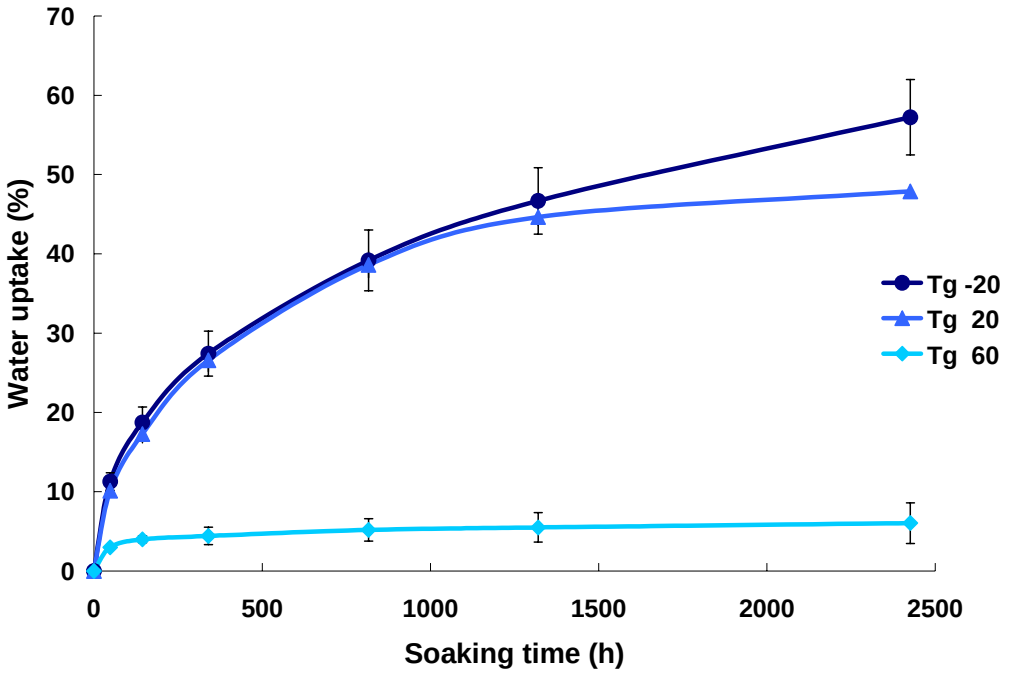

Figure 15. Water uptake by film of styrene acrylate latexes with different Tg:s after soaking in distilled water during three months.

The water uptake of the latexes did not stabilize even after 2 months of testing, especially for the TG -20 and Tg 20 latexes. After 48 hours, the latex uptake was about 10 % for the Tg -20 and Tg 20 latex whereas the latex uptake for Tg 60 was around 2 %. The styrene acrylate latexes with Tg -20 and 20 do not seem appropriate to use as a matrix to the composite regarding water uptake. The water absorption is not only physical absorption but is probably caused by cracks in the films, which were observe by SEM.

4.5.1 Weight

After the composite test strips were exposed to oven heating, the weight was reduced by about 5% as expected. This is the same kind of figure as the moisture content in regular paper at room temperature and moisture. When exposed to 50% RH, the weight was increased by water uptake and almost equal to the weight at room temperature. At 90% RH, all test strips increased about 15 % in weight. So far the water content in the test strips was very similar regardless of latex type or press settings used in the manufacturing step. However, when soaking in water there was a difference in water uptake between the different latexes and between the kinds of settings used in the press when preparing the composites. Composites made with relatively high Tg did not absorb water as much as those made with lower Tg:s. This might be explained by a larger part of acrylate in higher Tg latex as acrylate is known to be hydrophobic. Pressing warmer, longer and harder gave a better resistance against water uptake. It can also be explained by the higher stiffness and thereby resistance to fibre swelling of the Tg 60 latex. The results are presented in figure 16.

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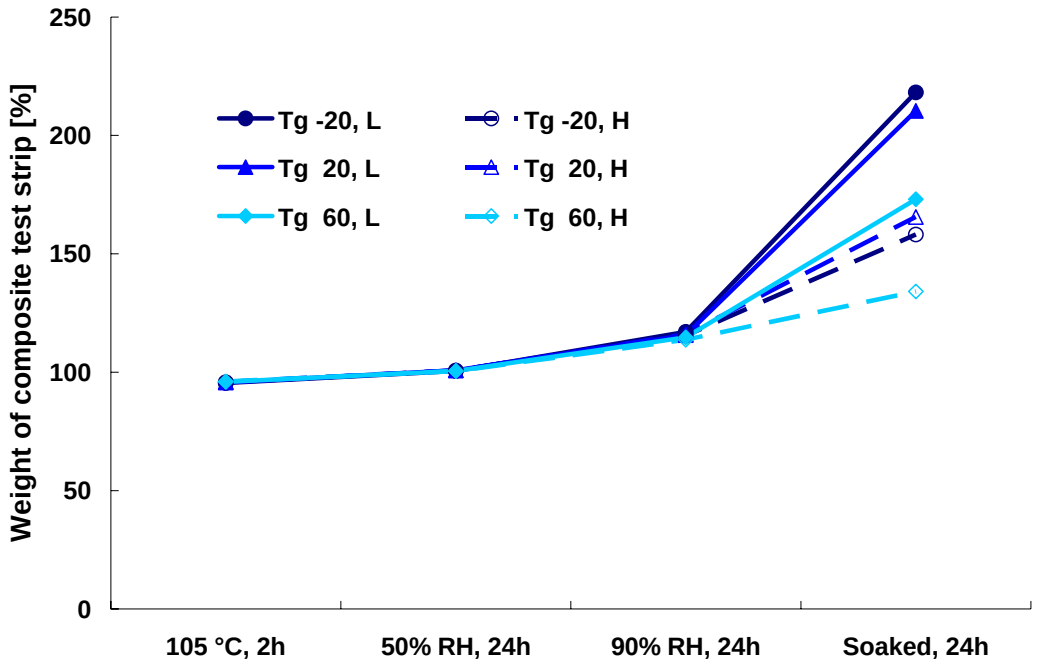

Figure 16. The weights of test strip exposed to different conditions and compared to original weight at room temperature and humidity. (L: 130°C, 5min, 90 bar, H: 200°C, 15 min, 180 bar)

There is a large difference in water uptake after soaking the composites for 24 h. Comparing low Tg latex composites pressed at low temperature, time and pressure with a high Tg latex composite pressed at high press settings. The difference between different Tg:s can be explained by the fact that the Tg of the styrene acrylate latexes are controlled by the amount of acrylate constituent units in the polymer chain making the polymer backbone less flexible. High Tg latex contain higher amount of acrylate, which is more hydrophobic. This is seen for the latex films as well, see figure 14. The differences achieved by changing press when fabricating the composites can have several explaintions. It probably softens the latex and fibres more at higher temperatures and the latex spreads more at higher pressures while the fibres gets more compacted. In this way there are fewer voids in the composite structure, making it harder for the moisture to penetrate. Higher temperatures and longer press times can increase the cross linking of the latex making the composite structure less sensitive to dimensional changes by moisture and the matrix gets stronger and stiffer making it more difficult for the fibres to swell. In conclusion: Curing improved the moisture resistance even further.

4.5.2 Length

The length of the test strips was not significantly affected by the moisture and water. Small differences could however be seen. These are shown in figure 15. Due to limited accuracy of the measurement the individual results may deviate from the actual values, but by looking at the results as a whole, it is clear that the length of the test strips are affected by the water and are more affected when more damp environments are used. The maximum increase in length after soaking was however not more than 1%.

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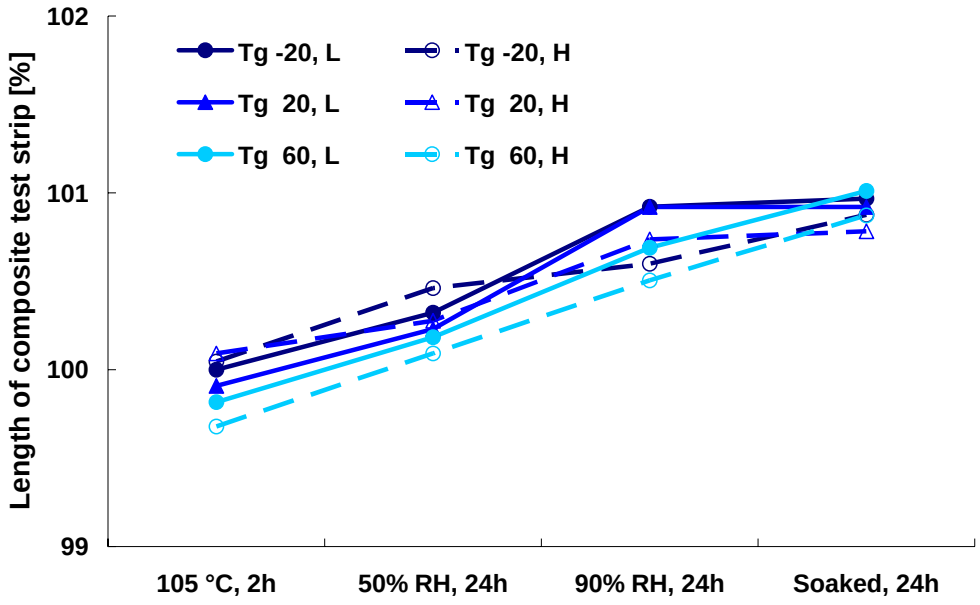

Figure 17. Changes in length when composite test strips are exposed to moisture at different conditions. L and H represent low and high settings in the press respectively (low: 130°C, 5min, 90 bar, high: 200°C, 15 min, 180 bar)

4.5.3 Thickness

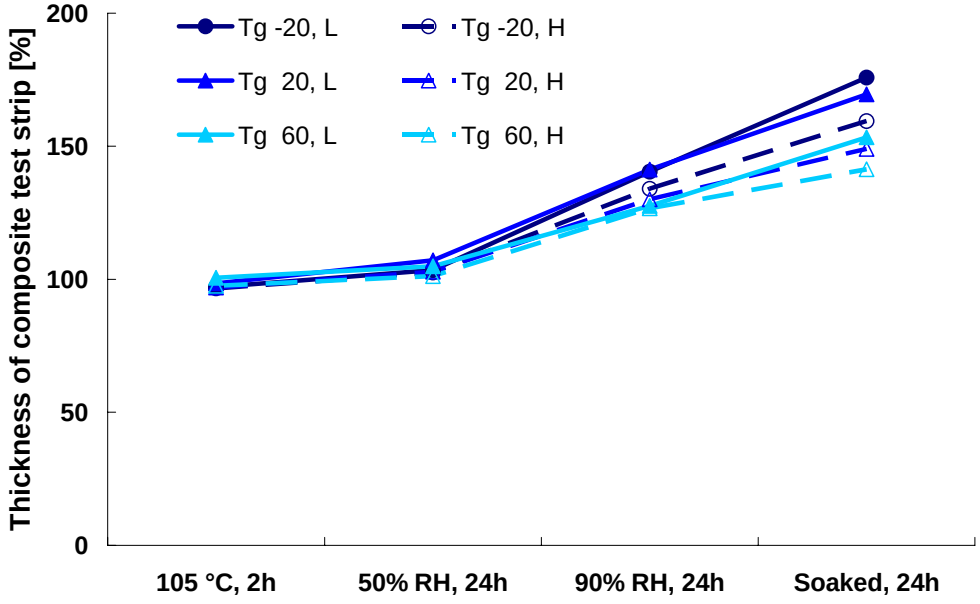

Figure 18. The thickness of composite made of styrene acrylate latexes at different Tg:s and press settings after being exposed to different humidity's. L and H represent low and high settings in the press respectively (L: 130°C, 5min, 90 bar, H: 200°C, 15 min, 180 bar)

The thickness of the test strips also increased beyond acceptable levels at increased moisture. When the composites were soaked in water, thickness increased to more than 50 % in average. The results are shown in figure 18. These results correspond well with calculated results using the increase in weight and follows a similar pattern regarding press settings and Tg of the latexes.

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Tests with composites made with the same type of latex, but at 30 % latex content instead of 20, showed an improvement in resistance against moisture and water. The increase in weight was reduced by about 30 % units and about 15 % units in decreased thickness. No decrease in length was observed. However, the water uptake was still large. On the other hand, a DFR sheet without latex and without any type of pressing has a water uptake of about 2000 % (20 g water/ g fibre)

4.6 Composite tensile strength at elevated temperatures

Good formability can allow a composite to be thermoformed into complex shapes. During forming, the material has to extend in to deep edges and joints and thereby increasing its surface area. The formability of the composite structure is believed to be connected to the elongation of the material when subjected to stress. The elongation of the composites is affected by heat so a trial in which tensile strength of the composites was measured at different temperatures was performed. The results on strain at elevated temperatures are presented in figure 19.

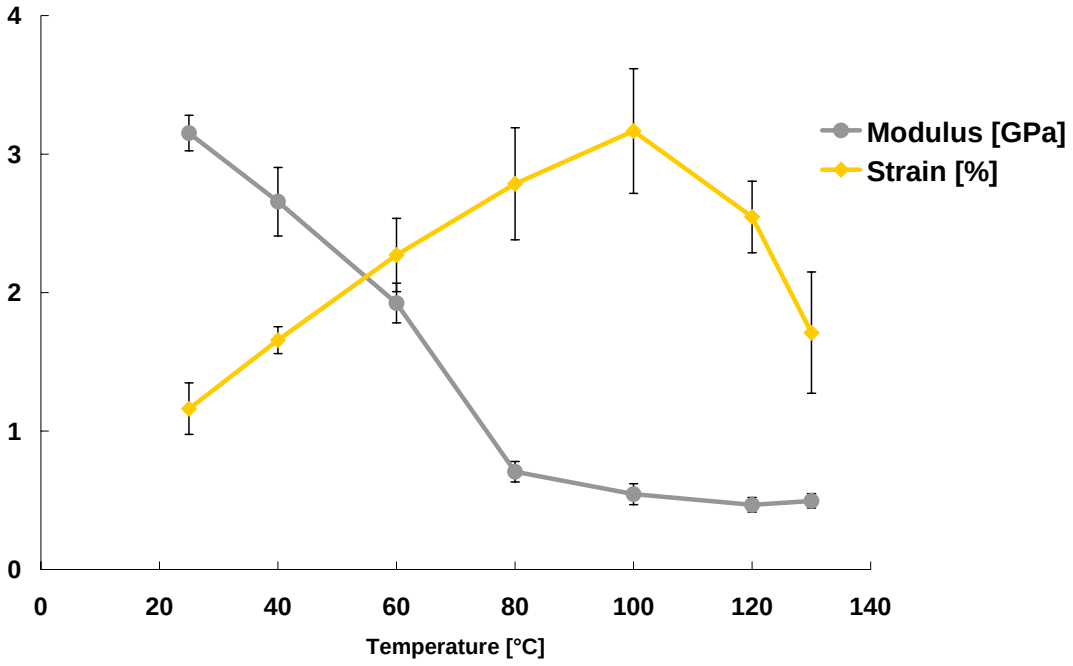

Figure 19. Strain at break and modulus for composites made with Tg 60 styrene acrylate latex as matrix at different temperatures (manufactured at 130 °C, 90 bar and 5 minutes)

The strain at break increases about 200 % at 100 °C compared to room temperature. Then the strain seems to decline at higher temperatures. The curve in figure 19 suggests that forming of the composite material containing styrene acrylate with a Tg of 60 as matrix should be performed around 100 °C to make use of highest achievable strain.

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4.7 Effect of the fibre material

When testing tensile strength it is clear that the composite test strips fail in the checked pattern originating from the DFR fibre mat. A DFR fibre mat without the checked pattern could increase the tensile strength and elongation considerably. Tests with hand-dipped sheets of smooth surface DFR sheets confirm this statement. These DFR sheets however were not suitable to use in the size press mainly because of its poor surface strength and the unevenness in matrix uptake during impregnation.

4.8 Shaping of the composite

Different tests were made to examine the formability of the composite material. Thermoforming to both simple and advanced structures by vacuum forming or compression moulding were tested. Also different tooling tests were performed such as sawing, drilling, nailing, bending, painting and corrugating the material, see figure 18 for a summarizing picture. Vacuum forming was tested on the final composite material with the styrene acrylates as well as vinyl acetate as matrix at PRplast AB, Östersund and SICOMP AB, Piteå both located in Sweden. These tests showed that vacuum forming is not suitable at the present development stage of material and processing equipment. Compression moulding was successfully tested to simple structures (i.e. trays) at SICOMP AB and Formpress AB, Färjestaden, Sweden. A more advanced structure was tested at our own lab by pressing round plugs with steep edges with good results.

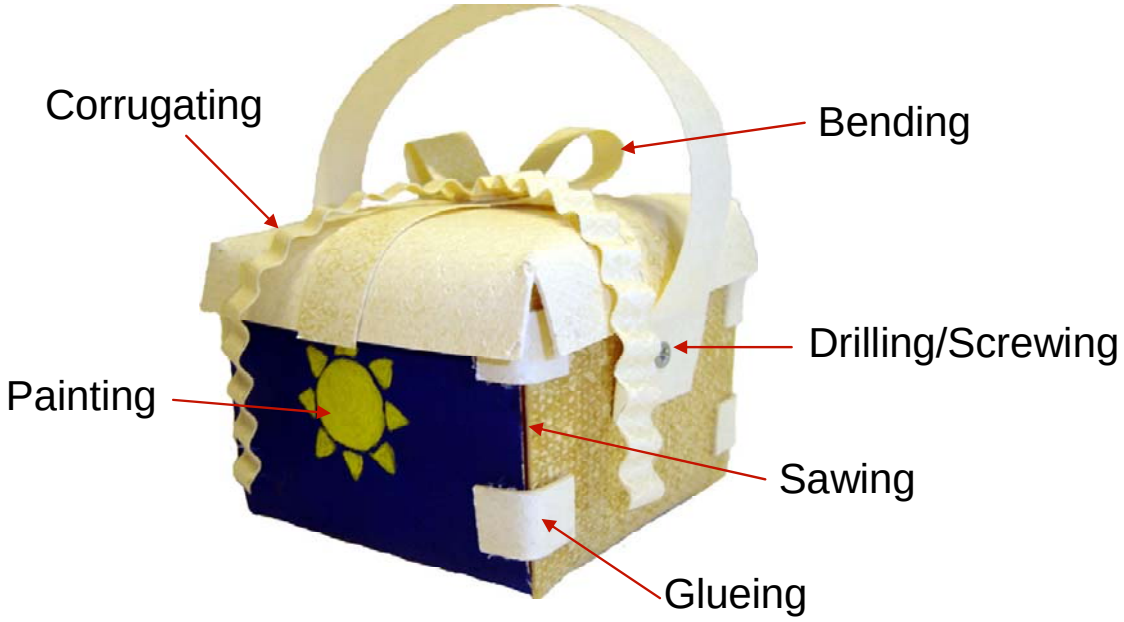

Figure 20. A basket completely made of our composite material (plus two metallic screws, glue and some paint). The figure represents some of the different tooling performed on the composite.

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5. COMPARISON TO COMMERCIAL PRODUCTS

One of the aims of this study was to investigate if wood fibre composites can compete with commercial products. No extensive evaluation could be made but the modulus and the tensile strength was compared with commercial products. Data of commercial materials were taken from Oksman[35].

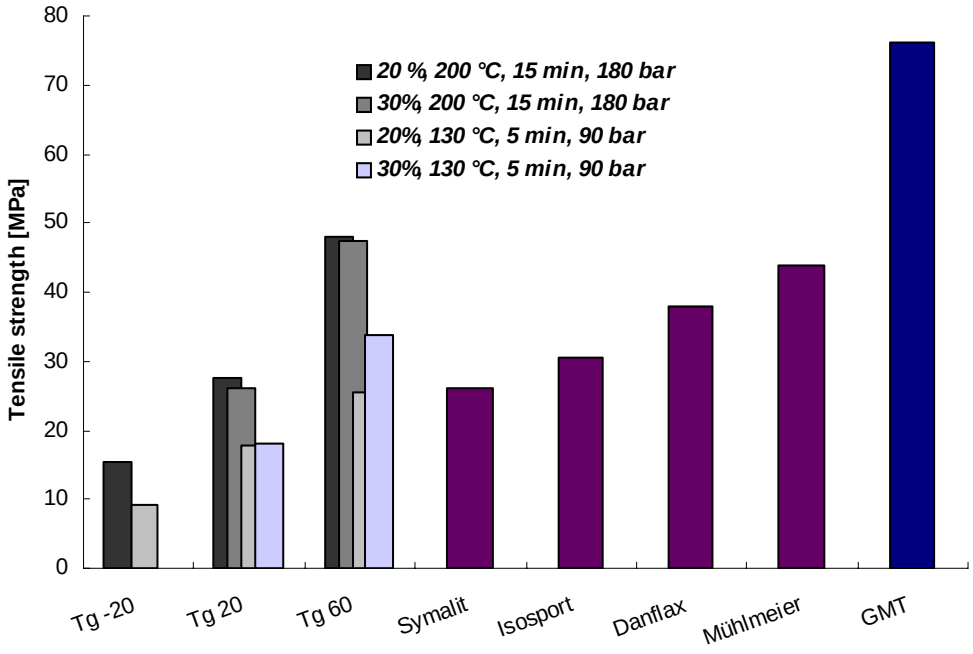

Figure 21. The tensile strength of our composite compared to commercial samples. One glass fibre product (GMT) is also included

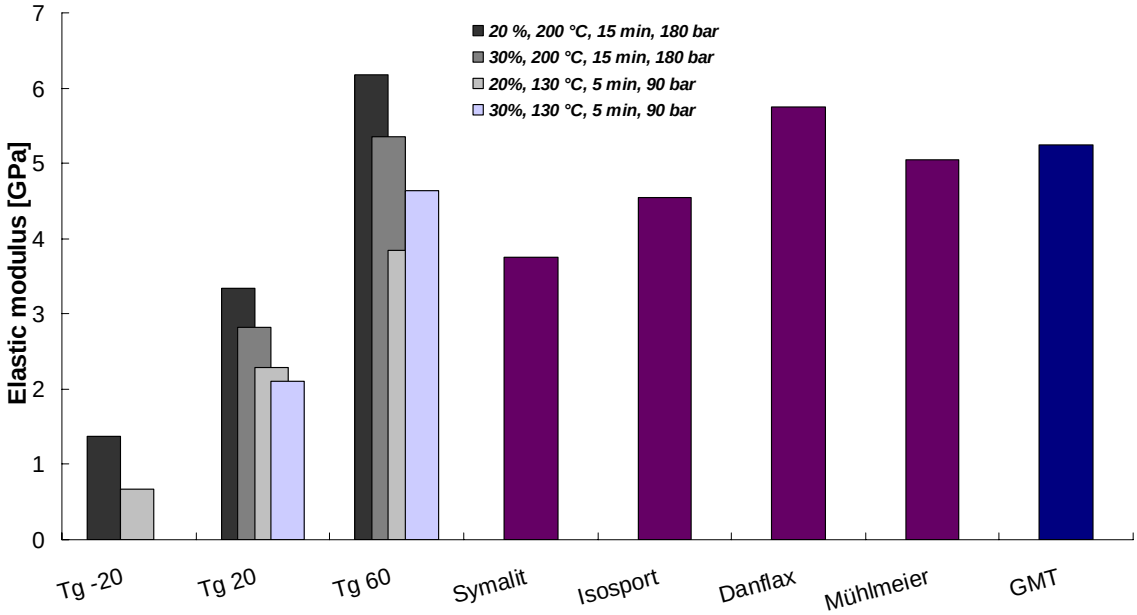

Figure 22. The elastic modulus of our composite compared to commercial samples. One glass fibre product (GMT) is also included

It can be concluded from the figures that our composite can compete with, and in some cases exceed, existing commercial products in terms of tensile strength and modulus.

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6. CONCLUDING REMARKS

The aim of this project has been to explore the possibility to produce thermo-forming wood fibre composites with high fibre content by applying papermaking technology. The key elements we have been using are a size press, a very absorbent fibre web and dispersion polymers. The results show that it is fully possible to make the composite in laboratory scale with this set-up. A logical next step would be to make the composite at pilot or production scale.

The material properties depend largely on the latex properties, but the pressing settings are also important for the final result. The material can be made with large differences in strength and water uptake, mostly depending on latex properties. A soft latex as matrix gives a flexible material whereas a stiff latex gives a stiff material. Blending latexes with the same type of copolymers, but different compositions, gives a mixture of strength properties from both types of latex used. The monomer composition (type) of the latex is also important. The conclusion is that there should be room for plenty of improvement in the composite properties by investigation of different types and amounts of latexes.

It is clear that the composite test strips fails in the checked pattern originating from the DFR fibre mat. A fibre mat without the checked pattern could increase the tensile strength and elongation considerably. Tests with smooth surface sheets DFR sheets confirm this statement. Consequently there is room form improvement in the composites properties by changing the fibre web as well.

Pressing at elevated temperatures, higher pressure and longer time increases the tensile strength and stiffness as well as the moisture resistance of the composite (in the experimental range we have been using). Press temperature needs to be kept high enough to prevent delamination and low enough to prevent the CTMP fibres and the matrix from thermal degradation. Pressure and time are not as important as the temperature in the investigated ranges.

Curing the composites after the pressing procedure changes the tensile strength and water up take. Different curing agents are thus options to improve properties in the future.

Our composite can compete with, or excel over, commercial products in terms of modulus and tensile strength. The water uptake may need to be improved and its thermo-forming properties need to be better evaluated. In order to do that demand and restraints of end-users must be understood.

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APPENDIX 1

Experimental data

The values behind the ±-sign are an interval estimate for the mean to a 95% confidence level, where available.

Table A. Data for section 4.1: Tensile strength of styrene acrylate latexes samples with different Tg. (Pressed at 30 bar, 130 °C and 5 minutes.)

Tg [°C]	Thickness [µm]	Grammage [g/m^2]	Density [kg/m^3]	Tensile strength [MPa]	E-modulus [GPa]	Strain at break [%]
-20	1090	1154	1063	0,54±0,13	1,91E-4±2,16E-5	429±189
20	960	899	944	5,55±0,36	0,0115±0,00295	231±9
60	970	940	975	9,43±1,11	1,261±0,081	1,06±0,21

Table B. data not showed in report, Dimensions of DFR fibre web, 1 ply

	Width [mm]	Length [mm]	Area [m^2]	Thickness [µm]	Grammage [g/m^2]	Density [kg/m^3]
original	301	359	0,108059	672	337,8	502,9
Run trough sizepress w. latex and dried	303	378	0,114534	1625	431,3	265,4
run, dried and pressed*				398	408,4	1026,6

*Pressed at 90 bar, 5 minutes and 130 °C

Table C. Data for section 4.2 Tensile properties of CTMP fibre web (DFR), no latex, 1 ply, influence on press temperature (5 minutes, 90 bar)

Press temp. [°C]	Thickness [µm]	Grammage [g/m^2]	Density [kg/m^3]	Tenisle strength [MPa]	E-modulus [GPa]	Strain at break [%]
60	629	349	555	1,75±0,15	0,198±0,02	2,0±0,2
80	611	353	578	1,79±0,12	0,204±0,03	2,1±0,2
100	590	354	600	1,84±0,14	0,204±0,03	2,2±0,3
120	565	347	614	1,96±0,11	0,233±0,03	2,1±0,2
140	575	345	600	2,00±0,18	0,235±0,04	2,1±0,1
160	557	342	613	2,03±0,20	0,257±0,05	2,1±0,2
180	536	347	648	2,28±0,14	0,348±0,06	2,0±0,2
200	526	345	656	2,39±0,21	0,397±0,07	1,9±0,1
220	511	340	666	2,55±0,30	0,525±0,15	1,8±0,1
240	497	334	672	3,15±0,58	0,760±0,26	1,7±0,2

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Table D. Data for section 4.2.1 Influence on tensile strength on composite by varying press temperature (Matrix: Styrene acrylate, Tg -20, 20% content. Press settings: 90 bar, 5 minutes, 2 ply)

Press temp [°C]	Thickness [µm]	Grammage [g/m^2]	Density [kg/m^3]	Tenisle strength [MPa]	E-modulus [GPa]	Strain at break [%]
70	977	772	791	3,78±0,20	0,169±0,01	8,27±0,43
100	836	770	920	5,82±0,38	0,351±0,02	6,79±0,71
130	768	770	1002	9,19±0,20	0,670±0,01	5,80±0,41
200	797	761	955	12,04±0,25	1,042±0,02	4,41±0,30

Table E. Data for section 4.2.2 Influence on tensile strength on composite by varying press time (Matrix: Vinyl acetate, Tg 32, 20 % content. Press settings: 90 bar, 130 °C, 2 ply)

Press time [min]	Thickness [µm]	Grammage [g/m^2]	Density [kg/m^3]	Tenisle strength [MPa]	E-modulus [GPa]	Strain at break [%]
0,5	797	796	998	29,9±1,4	3,7±0,1	2,3±0,2
1	784	791	1009	28,8±0,8	3,7±0,1	1,8±0,1
3	760	794	1045	33,0±0,8	4,2±0,1	2,0±0,2
5	750	797	1062	32,7±1,5	4,4±0,2	1,7±0,2
7	764	784	1025	30,0±1,7	4,2±0,2	1,5±0,2
10	742	799	1077	35,4±1,0	4,5±0,1	1,8±0,1
15	740	790	1068	35,7±1,6	4,5±0,2	2,0±0,1
20	725	804	1110	38,6±1,6	4,9±0,2	1,9±0,2
30	692	773	1117	38,7±1,8	5,0±0,2	1,8±0,3

Table F. Data for section 4.2.3 Influence on tensile strength on composite by varying press pressure (Matrix: Vinyl acetate, Tg 32, 20 % content. Press settings: 5 min, 130 °C, 2 ply)

Press pressure [bar]	Thickness [µm]	Grammage [g/m^2]	Density [kg/m^3]	Tenisle strength [MPa]	E-modulus [GPa]	Strain at break [%]
30	859	840	978	28,7±0,9	3,5±0,1	2,1±0,2
40	828	833	1006	30,9±1,3	3,7±0,1	2,3±0,2
50	792	825	1042	31,9±1,5	4,0±0,1	1,9±0,2
65	801	842	1051	28,7±1,7	4,0±0,1	1,4±0,2
80	775	833	1074	34,7±1,6	4,1±0,2	2,3±0,2
90	768	827	1077	33,7±2,2	4,4±0,2	1,7±0,2

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Table G. Data for section 4.2.4 Influence on tensile strength on composite by varying all press variables (Matrix: Styrene acrylate (last point: vinyl acetate), 2 ply)

Press settings Temp/time/pressure [°C]/[min]/[bar]	Latex Content [%]	Tg [°C]	Thickness [µm]	Grammage [g/m^2]	Density [kg/m^3]	Tenisle strength [MPa]	E-modulus [GPa]	Strain at break [%]
130/5/90	18	-20	769	740	963	8,01±0,23	0,65±0,04	5,2±0,5
130/15/90	18	-20	722	734	1017	10,13±0,22	0,90±0,02	4,3±0,3
200/5/90	18	-20	794	739	931	11,08±0,20	1,02±0,02	4,0±0,2
200/15/90	18	-20	799	740	925	12,05±0,45	1,19±0,04	3,2±0,2
130/5/90	20	-20	768	770	1003	9,19±0,20	0,67±0,01	5,8±0,4
130/15/90	20	-20	763	776	1018	10,33±0,26	0,84±0,01	5,2±0,5
200/5/90	20	-20	797	761	955	12,04±0,25	1,04±0,02	4,4±0,3
200/15/90	20	-20	774	747	965	12,47±0,21	1,17±0,02	3,4±0,2
130/5/180	18	-20	675	716	1061	10,77±0,34	0,94±0,03	4,2±0,5
130/15/180	18	-20	647	713	1102	12,69±0,39	1,13±0,04	4,1±0,3
200/5/180	18	-20	716	713	996	12,16±0,39	1,16±0,02	3,4±0,2
200/15/180	18	-20	693	708	1022	14,26±0,55	1,38±0,02	3,7±0,4
130/5/180	20	-20	662	742	1121	11,42±0,24	0,95±0,02	4,9±0,7
130/15/180	20	-20	643	730	1136	13,52±0,36	1,18±0,04	4,1±0,3
200/5/180	20	-20	696	720	1035	12,98±0,52	1,21±0,03	3,4±0,3
200/15/180	20	-20	674	731	1084	15,38±0,21	1,38±0,02	4,0±0,1
130/5/90	20	20	838	774	923	17,70±0,66	2,28±0,06	2,8±0,4
130/15/90	20	20	818	786	961	19,92±0,80	2,52±0,05	2,8±0,3
200/5/90	20	20	832	793	953	22,75±0,89	2,67±0,06	2,9±0,2
200/15/90	20	20	780	778	997	25,22±0,51	2,92±0,04	2,8±0,1
130/5/90	30	20	933	931	998	18,12±0,88	2,11±0,04	3,6±0,4
130/15/90	30	20	899	929	1033	19,84±066	2,48±0,05	3,0±0,2
200/5/90	30	20	917	936	1021	23,25±0,40	2,58±0,06	3,2±0,2
200/15/90	30	20	881	948	1076	22,39±1,23	2,69±0,06	2,6±0,3
130/5/180	20	20	1108	811	731	9,80±0,33	1,23±0,07	3,0±0,2
130/15/180	20	20	709	759	1070	24,92±0,73	3,27±0,10	2,7±0,3
200/5/180	20	20	698	749	1072	27,68±1,12	3,38±0,11	2,7±0,3
200/15/180	20	20	741	753	1017	27,49±0,86	3,34±0,09	2,6±0,2
130/5/180	30	20	846	920	1088	23,31±1,06	2,78±0,07	2,7±0,3
130/15/180	30	20	792	861	1087	25,03±1,46	2,89±0,11	2,9±0,3
200/5/180	30	20	894	885	990	24,43±1,34	2,55±0,06	3,3±0,2
200/15/180	30	20	846	883	1043	26,10±1,42	2,82±0,08	3,1±0,3
130/5/90	20	60	873	841	963	25,40±0,72	3,84±0,08	1,3±0,1
130/15/90	20	60	834	840	1007	30,40±0,51	4,50±0,05	1,3±0,1
200/5/90	20	60	791	845	1069	39,11±0,56	5,23±0,07	1,2±0,1
200/15/90	20	60	798	840	1052	28,90±0,78	4,53±0,15	1,0±0,1
130/5/90	30	60	791	832	1053	27,59±0,77	4,24±0,11	1,1±0,1
130/15/90	30	60	779	833	1069	30,41±0,97	4,58±0,05	1,1±0,1
200/5/90	30	60	726	821	1131	39,93±1,03	5,12±0,08	1,2±0,1
200/15/90	30	60	691	792	1147	42,10±1,78	5,18±0,06	1,2±0,1
130/5/180	20	60	915	965	1055	28,43±0,81	4,53±0,07	1,3±0,1
130/15/180	20	60	896	952	1063	31,68±0,68	4,73±0,04	1,4±0,1
200/5/180	20	60	863	954	1106	42,17±1,33	5,72±0,07	1,3±0,1
200/15/180	20	60	843	940	1115	48,05±2,14	6,18±0,12	1,3±0,1
130/5/180	30	60	887	967	1090	33,91±0,71	4,64±0,06	1,4±0,1
130/15/180	30	60	837	928	1109	35,39±1,13	5,04±0,07	1,1±0,1
200/5/180	30	60	807	929	1152	44,46±2,11	5,63±0,07	1,1±0,1
200/15/180	30	60	839	930	1109	47,44±0,91	5,36±0,07	1,4±0,1
130/5/90	20	32	796	817	1027	31,57±1,44	4,11±0,06	1,8±0,2

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Table H. Data for section 4.3. Influence on tensile strength on curing the composite by varying curing time and temperature. (Matrix: Styrene acrylate, 20 % content. Press settings: 5 min, 130 °C, 90 bar, 2 ply) Assuming that dimensions on composite test strips are the same before and after curing. N= 6

Tg [°C]	Curing temp [°C]	Curing time [min]	Thickness [µm]	Grammage [g/m^2]	Density [kg/m^3]	Tenisle strength [MPa]	E-modulus [GPa]	Strain at break [%]
60	0	0	873	841	963	25,40±0,72	3,84±0,08	1,3±0,1
60	160	10	873	841	963	28,44±1,62	3,32±0,07	1,8±0,3
60	160	30	873	841	963	29,82±0,52	3,34±0,02	1,8±0,1
60	160	120	873	841	963	28,66±2,27	3,33±0,05	1,7±0,3
20	0	0	838	774	923	17,70±0,66	2,28±0,06	2,8±0,4
20	105	30	838	774	923	16,07±1,04	2,00±0,03	3,1±0,5
20	140	30	838	774	923	18,14±0,67	1,86±0,05	3,7±0,4
20	180	30	838	774	923	18,10±0,68	1,91±0,04	3,4±0,3

Table I. Data for section 4.3. Influence on tensile strength on curing the composite by varying curing time and temperature. (Matrix: Styrene acrylate, Tg 60, 20 % content. Press settings: 5 min, 130 °C and 90 bar, 2 ply. Testing if changes in material dimensions affect strength at different curing temperatures. Curing time: 30 minutes. N = 4

Curing temp [°C]	Thickness Before curing [µm]	Grammage before curing [g/m^2]	Density Before curing [kg/m^3]	Thickness After curing [µm]	Grammage After curing [g/m^2]	Density After curing [kg/m^3]	Difference in thickness [%]	Difference in grammage [%]	Difference in density [%]
0	879	842	958	880	843	958	0,1	0,0	-0,1
70	855	840	984	851	838	984	-0,4	-0,3	0,1
105	830	827	997	862	826	959	3,8	-0,1	-3,8
140	831	836	1007	872	834	956	5,0	-0,3	-5,0
180	841	847	1007	894	836	935	6,3	-1,3	-7,1

Curing temp [°C]	Tenisle strength♣ [MPa]	E-modulus♣ [GPa]	Strain at break [%]	Tenisle strength♦ [MPa]	E-modulus♦ [GPa]	Change in Tenisle strength [%]	Change in E-modulus [%]
0	24,61	3,84	1,1	24,61	3,84	0,0	0,0
70	24,81	3,91	1,3	24,91	3,92	0,4	0,4
105	26,72	3,89	1,7	25,73	3,75	-3,8	-3,8
140	31,71	3,85	1,6	30,21	3,67	-5,0	-5,0
180	31,55	3,78	1,5	29,69	3,56	-6,3	-6,3

♣ calculated by using thickness before curing
♦ calculated by using thickness after curing

Table J. Data for section 4.4 Influence on tensile strength on latex mixtures as matrix (20 % total latex content in sheet. Press settings: 5 min, 130 °C, 90 bar, 2 ply)

Latex A [part, Tg, type]	Latex B [part, Tg, type]	Thickness [µm]	Grammage [g/m^2]	Density [kg/m^3]	Tenisle strength [MPa]	E-modulus [GPa]	Strain at break [%]
100 % Tg -20 SA	0 % Tg 60 SA	768	770	1003	9,19±0,20	0,67±0,01	5,8±0,4
50 % Tg -20 SA	50 % Tg 60 SA	759	844	1113	13,96±0,97	2,08±0,12	2,2±0,1
10 % Tg -20 SA	90 % Tg 60 SA	758	822	1084	29,26±1,56	4,23±0,13	1,5±0,2
0 % Tg -20 SA	100 % Tg 60 SA	873	841	963	25,40±0,72	3,84±0,08	1,3±0,1
10 % Tg -20 SA*	90 % Tg 32 VA*	753	893	1186	29,36±1,95	4,01±0,15	2,1±0,3

SA = styrene acrylate, VA = vinyl acetate, * = total latex content 24 %

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Table K. data not shown in report, Tensile strength of other matrices and fibrewebs and commercioal products (pressettings: 5 min, 130 °C, 90 bar)

Matrix polymer	Matrix content [%]	Fibre web		Thickness [µm]	Grammage [g/m^2]	Density [kg/m^3]	Tenisle strength [MPa]	E-modulus [GPa]	Strain at break [%]
Wood glue*	27	DFR standard		811	880	1085	29,71±0,79	3,96±0,04	1,7±0,1
Wood glue*	17	DFR Without checked pattern		544	693	1274	25,8±1,96	4,26±0,21	1,1±0,1
SA Tg -20	10	DFR Without checked pattern		643	637	991	11,40±0,49	0,84±0,04	3,1±0,3
VA Tg 32	19	CTMP fibre web[2]		908	915	1008	58,14±1,88	4,76±0,07	3,2±0,2
Va Tg 32	?	Brown recycled fibre web[2]		977	1037	1062	49,60±2,24	4,57±0,16	4,0±0,4
Poly-propylene	60	Flax mat[3]		2512	1935	771	23,02±0,47	2,04±0,10	2,8±0,2

*= cascol trälim ute (vinyl acetate based), 2 = home made sheets. 3 Commercial product, not pressed with standard equipment, SA = styrene acrylate, VA = vinyl acetate

Table L. Data for section 4.5: Water uptake of pure styrene acrylate latex, N= 10

soaking time [h]	Water uptake, Tg 60 [g]	Water uptake, Tg 20 [g]	Water uptake, Tg -20 [g]	Water uptake, Tg 60 [%]	Water uptake, Tg 20 [%]	Water uptake, Tg -20 [%]
0	0,4914±0,0384	0,4039±0,0704	0,4665±0,0779	0	0	0
48	0,5065±0,0393	0,4480±0,0746	0,5245±0,0837	2,99±0,17	10,11±0,76	11,28±1,12
144	0,5116±0,0382	0,4861±0,0791	0,5715±0,0877	4,00±0,43	17,27±1,09	18,72±1,97
340	0,5137±0,0374	0,5469±0,0862	0,6383±0,0921	4,42±1,09	26,59±1,45	27,41±2,84
816	0,5178±0,0370	0,6532±0,0998	0,7603±0,0996	5,18±1,41	38,62±1,62	39,17±3,82
1320	0,5195±0,0360	0,7253±0,1158	0,8677±0,1084	5,50±1,86	44,64±1,07	46,68±4,18

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Table M. Data for section 4.5.1, Effect on dimension stability by water uptake, weight of composite. Styrene acrylate as matrix,, n=1

Tg [°C]	Latex content [%]	Press temp. [°C]	Press time [min]	Press pressure [bar]	Weight at room temp and RH [g]	Weight after owen 105 oC, 2h [g]	Weight after storing at 50% RH, 24h [g]	Weight after storing at 90% RH, 24h [g]	Weight after soaking in water for 24h [g]
-20	20	130	5	90	3,8156	3,6423	3,8368	4,4650	8,3240
-20	20	200	15	180	3,5241	3,3736	3,5481	4,0680	5,5730
20	20	130	5	90	3,8382	3,6705	3,8680	4,4407	8,0735
20	30	130	5	90	4,6708	4,4766	4,7003	5,3280	8,0935
20	20	200	15	180	3,6934	3,5412	3,7211	4,2850	6,1140
20	30	200	15	180	4,3665	4,1960	4,3940	4,9790	6,6600
60	20	130	5	90	4,0104	3,8448	4,0305	4,6006	6,9393
60	30	130	5	90	4,7623	4,5777	4,7893	5,3951	6,8352
60	20	200	15	180	3,7758	3,6273	3,7918	4,2926	5,0630
60	30	200	15	180	4,4746	4,3410	4,4491	4,9891	5,4669

Table N. Data for section 4.5.2 Effect on dimension stability by water uptake, length of composite. Styrene acrylate as matrix, length, n=1

Tg [°C]	Latex content [%]	Press temp. [°C]	Press time [min]	Press pressure [bar]	Length at room temp and RH [mm]	Length after owen 105 oC, 2h [mm]	Length after storing at 50% RH, 24h [mm]	Length after storing at 90% RH, 24h [mm]	Weight after soaking in water for 24h [mm]
-20	20	130	5	90	217,2	217,2	217,9	219,2	219,3
-20	20	200	15	180	217	217,1	218	218,3	218,9
20	20	130	5	90	217,5	217,3	218	219,5	219,5
20	30	130	5	90	217	217,1	217,9	219	219,8
20	20	200	15	180	217,2	217,4	217,8	218,8	218,9
20	30	200	15	180	217,2	217,3	217,9	218,6	218,7
60	20	130	5	90	217,7	217,3	218,1	219,2	219,9
60	30	130	5	90	217,8	217,1	218	219,2	220,2
60	20	200	15	180	217,8	217,1	218	218,9	219,7
60	30	200	15	180	217,8	217,1	217,9	219	219,2

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Table O. Data for section 4.5.3 Effect on dimension stability by water uptake, thickness of composite. Styrene acrylate as matrix, thickness, n=1

Tg [°C]	Latex content [%]	Press temp. [°C]	Press time [min]	Press pressure [bar]	Thickness at room temp and RH [µm]	Thickness after owen 105 oC, 2h [µm]	Thickness after storing at 50% RH, 24h [µm]	Thickness after storing at 90% RH, 24h [µm]	Thickness after soaking in water for 24h [µm]
-20	20	130	5	90	838,2	813,4	867,2	1177,4	1473,4
-20	20	200	15	180	640,8	618,6	657,2	858,6	1021,8
20	20	130	5	90	846,4	833,6	906,2	1195,2	1434,4
20	30	130	5	90	967,6	969,2	1020,8	1279	1545,2
20	20	200	15	180	761,8	738,2	783,4	990,4	1135
20	30	200	15	180	890,2	868,4	915,4	1113	1278
60	20	130	5	90	824	828,4	864	1051,4	1263,6
60	30	130	5	90	917	928,6	948,2	1113,4	1290,6
60	20	200	15	180	652	636,2	659,6	825,8	921,2
60	30	200	15	180	760,8	747,2	766	932,4	1040,8

Table P. Data for section 4.6: Tenisle strength of composite at different testing temperatures, matrix: styrene acrylate, Tg 60, 25% content, hand dipped samples, pressed at 90 bar, 130 °C and 5 minutes. Dimensions of test strip were assumed unchanged at elevated temperatures.

Temperature [°C]	Thickness [µm]	Grammage [g/m^2]	Density [kg/m^3]	Tensile strength [MPa]	E-modulus [GPa]	Strain at break [%]
25	877	850	971	19,30±2,6	3,152±0,128	1,16±0,19
40	877	850	971	19,91±1,8	2,657±0,248	1,66±0,10
60	877	850	971	15,27±1,0	1,924±0,143	2,27±0,26
80	877	850	971	8,57±1,3	0,706±0,074	2,79±0,40
100	877	850	971	6,75±0,7	0,543±0,076	3,17±0,45
120	877	850	971	3,95±0,6	0,467±0,053	2,55±0,26
130	877	850	971	3,73±0,5	0,495±0,053	1,71±0,44