Rheological Methods to Simulate Adhesive Properties for the Wood Working Industry

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ABSTRACT

Rheological methods to simulate testing of adhesive systems for the interior and structural wood industry has been developed and shown to be a useful tool for predicting production parameters. The rheological methods are used to test the reactivity and flow properties during development of new adhesive systems and to ensure that the adhesive system is optimized for the specific production line and application equipment. The reactivity measured by a parallel plate oscillation method shows if the system fulfills the requirement with respect to production efficiency both with respect to pressing time and when the final glued specimen can be handled before further processing, packing and shipment. Optimal pressing temperature profile can be predicted for the glue systems which are cured at elevated temperatures. In addition, the rheological methods can be used to make guidelines for technical properties like assembly time. The assembly time, which is the longest time between application of the glue until the product is in press, is important for how fast the customer can produce. The assembly time is decided by glue spread, temperature, moisture in the wood and the reactivity of the new system. For systems that are mixed before applied on the wood specimen the pot life is of importance and this property can also be estimated from rheological measurements. The rheological methods show good correlation to more traditional and time consuming testing on wood, and for some properties it tells us more about the adhesive systems. Rheological methods save both time and resources; it is a helpful tool to understand the new adhesive systems and their gluing performance.

INTRODUCTION

The wood working industry includes a wide range of different products like furniture, flooring, beams, cross laminated timber, composite board and plywood. The adhesive systems for these applications will have very different requirements.

The main adhesives for the wood industry are thermosetting phenol and amino based and thermoplastic PVAc (Polyvinylacetate) systems, both one,- and two component systems, but also EPI (Emulsion polymer isocyanate) and PU (Polyurethane) are used. The adhesive systems are both cold and hot curing and require a degree of water resistance specific for the glued element.

The wood products are manufactured with a wide range of application equipment, like string application, roller application, curtain coater and different finger joint application methods. The adhesive systems for the different products then also need to have the right reactivity and flow properties to perform well. The applications and the glue systems are constantly changing and therefore also the product range and the properties of the adhesives.

The adhesive systems have a window for how long they can be used in a production line. This window is designed by the reactivity, flow properties, stability and how the glue system is applied.

During development of new adhesive systems both chemical properties, reactivity, strength, stability, adhesion to wood and flow properties needs to be investigated. This can be done by rheological methods developed for wood adhesives, and by comparison with older systems and conventional methods their suitability can be verified.

This article describes a few of the methods used during development and testing of new adhesive systems for the wood working industry.

EXPERIMENTAL

The measurements were done on Anton Paar Rheometers model MCR 301 and MCR302.

A parallel plate system was used for all the measurements; the dimension of the upper plate was varied from 25 to 50 mm depending on the measurement and the end viscosity for the measurement.

The assembly time measurements were performed with wood as lower plate to simulate better the adhesive systems properties on wood. Testing of adhesives on wood has been done earlier both for reactivity measurements[1, 2] and tack testing[3]. For the methods described in this article, birch veneer was used and the wood discs were adjusted to 12 % moisture content at 20 °C. This is the standard conditions for conventional testing of adhesive systems on solid wood.

Figure 1 shows the instrumentation for doing the assembly time measurements. The wooden disc is attached with a metal ring to the lower plate of the rheometer.

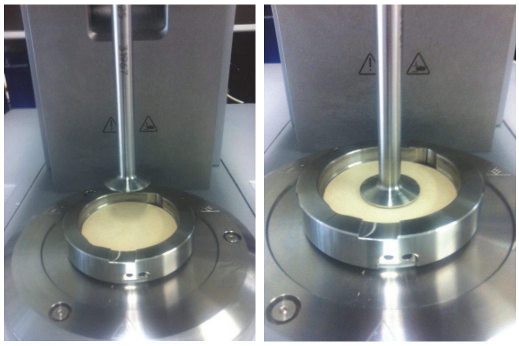

Figure 1. The assembly time measurement.

The gap for the different measurements varied from 0,2-1mm. For the measurement performed with a wood lower disc 0,2mm gap was used to simulate the typical glue line thickness. For the normal reactivity and flow measurements 1mm gap was used.

The strain was varied during the reactivity and assembly time measurements, because of increasing viscosity of the adhesive mix, starting from 0,3% going down to 0,01% depending on the measurement and the end viscosity.

To avoid skin formation when the adhesive system is in contact with air for a long time, the adhesive surface was covered with silicon oil during the measurements.

The complex viscosity (η*) shows the adhesive mix properties during the reactivity measurements.

The storage modulus (G') and loss modulus (G'') shows the gel point of glue mix (if this occurs).

The G' and G'' also describes the gel strength of the adhesive mix, this could also be related to the stability of fillers in the adhesive system.

The correlation between the η*, G' and G'' can be described with equation 1:

$$\eta^* = ((G'^2 + G''^2)^{1/2})/\gamma \qquad (1)$$

REACTIVITY MEASUREMENTS

The reactivity of an adhesive system can be measured by a parallel plate, constant strain method. The general method for the reactivity measurements is described in Table 1.

Table 1. Method for the reactivity measurements.

Strain	0,1-0,3 %
Frequency	1,6 Hz
Temperature	15-80 °C
Gap	0,2-1mm
Plate	PP50, PP25

Small variations in the method can give valuable information about the properties described below:

Pot life

Pot life is the cold curing properties of a two-component adhesive system, usually measured between 15 and 25°C depending on production parameters. It gives information about how long a glue mix can be used before the viscosity reaches a too high level which means that the glue mixture cannot be used in industrial application. The pot life/cold curing properties is often measured as the time the adhesive system uses to reach a certain level of viscosity. Figure 2 shows a comparison of the pot life measured for 3 different adhesive systems at 20 °C.

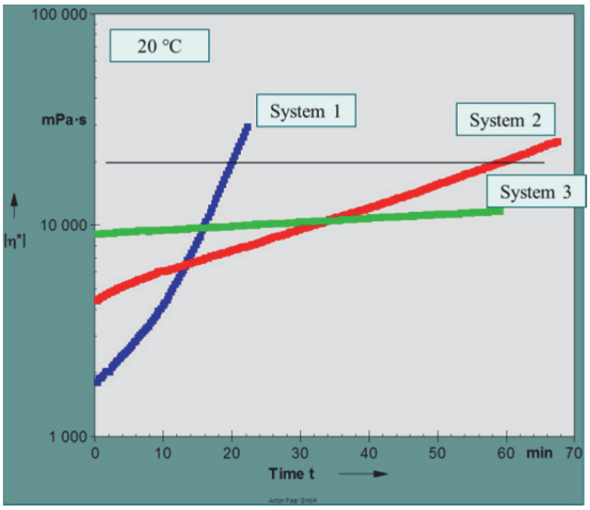

Figure 2. Comparison between 3 different adhesive systems with different cold curing properties.

System 1: 20 min pot life
System 2: 60 min pot life
System 3: Very long pot life, the system needs a higher temperature to cure

Gel time

The gel time in the wood working industry is the high-temperature curing properties of an adhesive system. It gives an indication of the pressing time for the system. The gel time is usually measured between 30 and 130 °C depending on pressing parameters used in the industrial application.

Figure 3 shows the impact of the temperature for a two-component system using different ratio between the components and different temperature. The figure shows that for this system the pressing time can be significantly reduced at all ratios going from 30 to 50 °C.

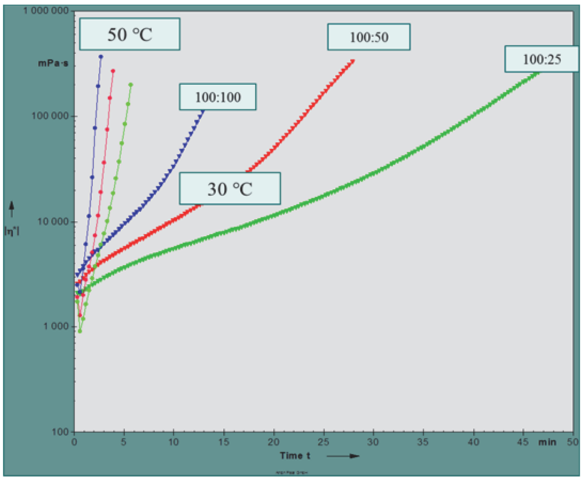

Figure 3. Two-component adhesive system, curing at 30 and 50°C with variations in the component ratio.

The geltime is defined on the rheometer as the time until the adhesive system has reached a viscosity of 100 Pa s[4].

Glue mix viscosity and curing pattern

The start complex viscosity and the curing pattern (viscosity development and the ratio between the storage and loss modulus) of a two component adhesive system can give valuable information about the performance both in the application equipment and the gluing properties.

Experience has shown that an adhesive system with too high start viscosity, can give low adhesion to the wood and bad gluing properties, even if the reactivity is similar to other systems.

An example of this is shown in Figure 4 and Figure 5. The figures show the start viscosity and the curing pattern of two different adhesive systems with the same gel time. Figure 4 shows that system 1 has got a higher start glue mix viscosity than system two, but that the two systems will have the same gel time (time to 100000mPa s).

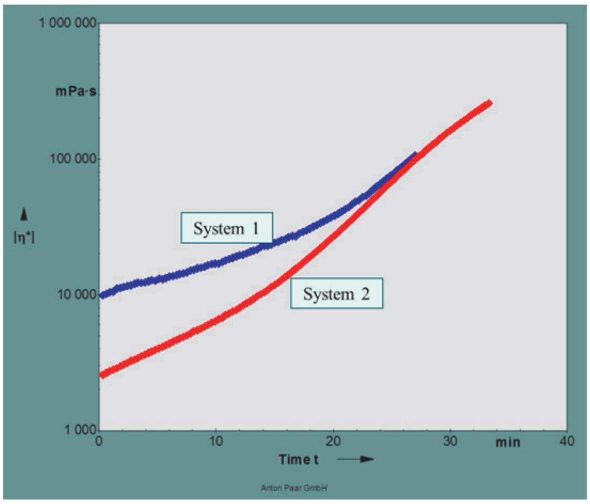

Figure 4. Viscosity development for two different adhesive systems with the same geltime.

Figure 5 shows the ratio between the storage and loss modulus of the same two systems as shown in Figure 4.

System 1 has from the start a higher level for the storage modulus than the loss modulus. System 2 has got a higher level of the loss modulus than the storage modulus, and it has a gel point after ca 20 minutes.

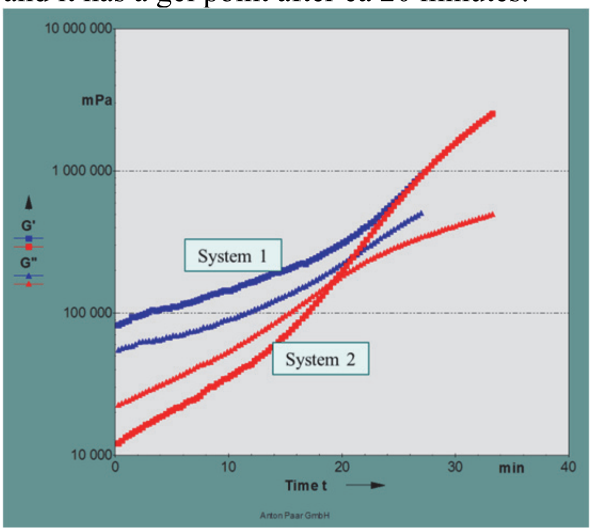

Figure 5. Ratio of storage and loss modulus of two adhesive systems.

System 2 showed better gluing properties than system 1.

With the rheological reactivity measurement, one measurement gives information about both the reactivity, the viscosity of the glue mix and the curing pattern.

Collecting this data for systems used for different applications, can be a good help when developing new systems.

ASSEMBLY TIME

Maximum assembly time is the longest time between application of glue until the product is in press, with a good performing glue line as the result. This is an important parameter in the wood working industry, because of general handling and unexpected stops in the production from the application of the adhesive until the product are in the press.

The assembly time is decided by the following parameters:

- Glue spread (amount of glue mix/m^2)
- Temperature of the adhesive, in the production area and in the wood.
- Wood moisture content
- Reactivity of the adhesive system.

An estimation of the assembly time is done using a parallel plate, oscillation method using wood disc as lower plate. By using a wood disc as a lower plate the interaction between the adhesive system and the wood in addition to the reactivity will be monitored.

Table 2. General method for the assembly time measurements.

Strain	0,01-0,3 %
Frequency	1,6 Hz
Temperature	15-30 °C
Plate	PP50, PP25
Wood disc	Diameter 58mm Thichness 2mm Birch 12 % MC

Examples of measurements estimating the assembly time with different temperatures, glue spread, ratio of two-component system and comparison of different systems is shown below.

Based on experience from more traditional measurements, the assembly time of a adhesive system on the rheometer could be set to when the glue mix on wood has reached a certain complex viscosity illustrated by a line in the figures below.

Figure 6 shows the estimation of the assembly time for a two component adhesive system by varying the ratio of the components. Changing the ratio of the two component system will change the assembly time.

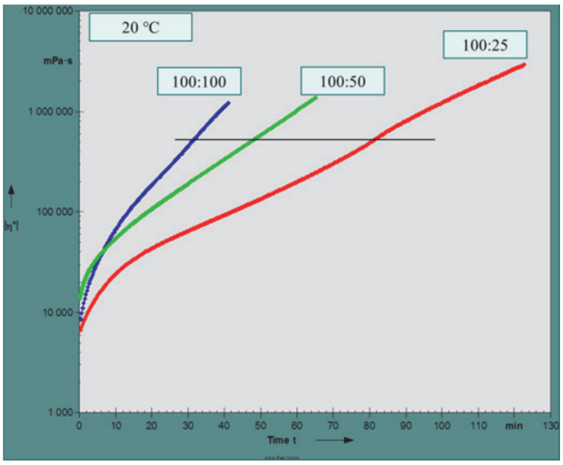

Figure 6. The estimation of assembly time for a two component adhesive system with different ratios of the components.

Figure 7 shows the estimation of assembly time of 3 different adhesive systems at 20 °C. The systems have different composition and reactivity.

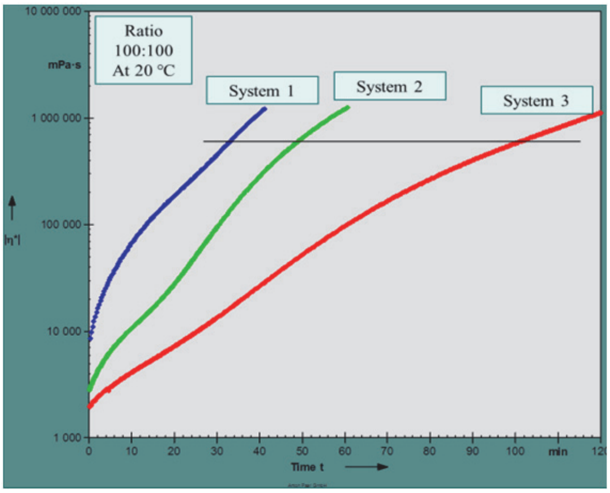

Figure 7. The assembly time of 3 different adhesive systems.

Figure 8 shows the impact of the temperature on the assembly time for the same adhesive system, and how this quite dramatically decreases when the temperature is increased.

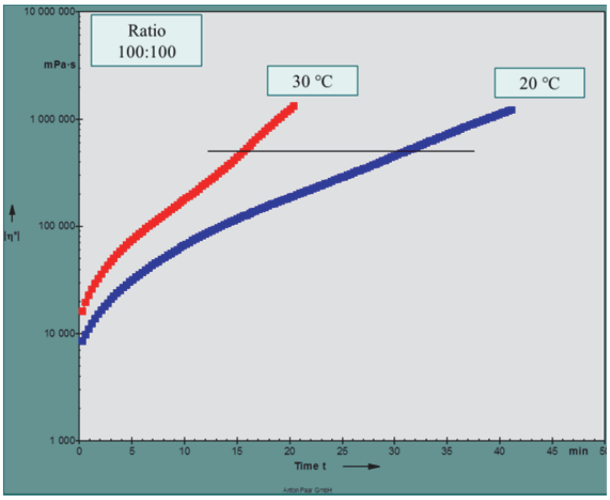

Figure 8. Estimating the assembly time of an adhesive system at different temperatures.

Figure 9 shows that the assembly time can be increased by having higher glue spread (apply more glue). This is simulated by using a larger gap in measurement on the rheometer.

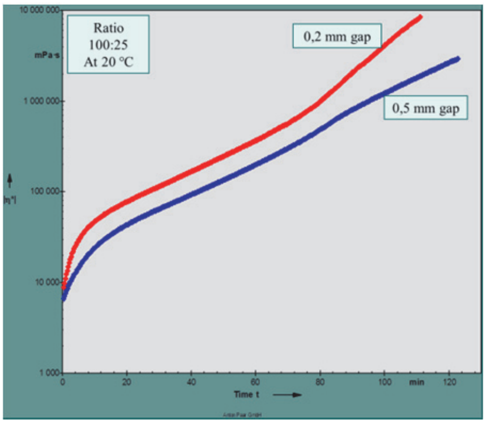

Figure 9. The assembly time of an adhesive system using different glue spread (changing the gap).

The method described above shows good correlation with the traditional methods for measuring the assembly time. Measuring the assembly time on the rheometer is less time consuming than traditional methods.

The test method has so far been used on two-component MUF systems for the wood working industry.

Because of water transfer into the wood, the complex viscosity of the glue mix starts at a higher level than measuring the reactivity without the wood.

The correlation of the complex viscosity of the applied glue mix with the assembly time is dependent on the solid content in the glue mix.

FLOW PROPERTIES

The flow properties of an adhesive system are measured by a rotational method with increasing shear rate.

Table 3. Method details for measuring the flow properties.

Shear rate	0,001-1000 1/s
Time	Depending on shear rate area
Temperature	20-25 °C
Plate	PP50
Gap	1 mm

The method is used for observing the following properties:

- ➢ Performance in application equipment,
handling before press, sagging
- ➢ ID, production reproducibility
- ➢ Storage stability, sedimentation during storage

Figure 10 shows the shear rate areas correlated to the different properties listed above.

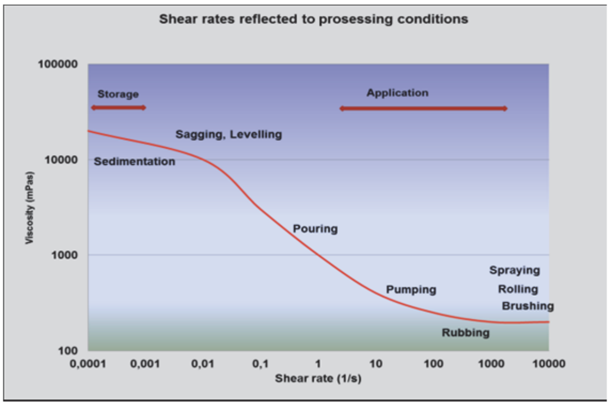

Figure 10. Shear rate areas for measuring different properties of an adhesive.

Adhesive systems for the wood working industry often contain fillers and additives that need to be stable during storage. Observing the flow curve for the adhesive systems in the low shear area will give valuable information about this property.

The stability of fillers in the adhesive systems can also be monitored by observing the ratio between the storage modulus and the loss modulus. Generally less sedimentation is observed when the storage modulus is higher than or similar to the loss modulus. But experience has shown that this is not always true and it is also other properties, like dispersion of the fillers, which influences.

Adhesive systems for different applications need very different flow properties to perform well. This is shown in Figure 11, and described in the examples below.

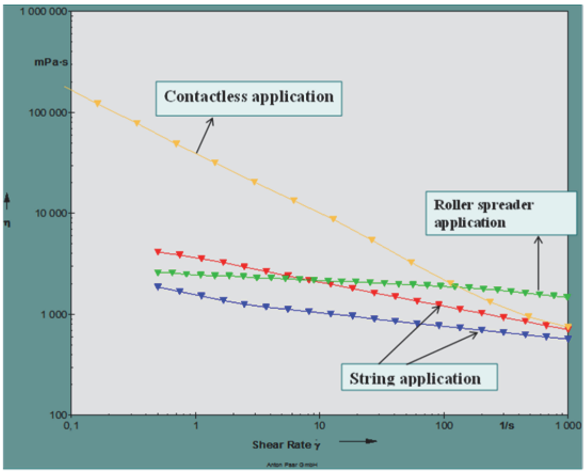

Figure 11. Flow properties for 3 different applications.

<u>Contactless application finger jointing</u>:

The adhesive system is applied at high pressure and needs to instantly hang from the finger joints after application. Very shear thinning properties is an advantage (see Figure 11, contactless application).

Contactless application of an adhesive on a finger joint is shown in Figure 12.

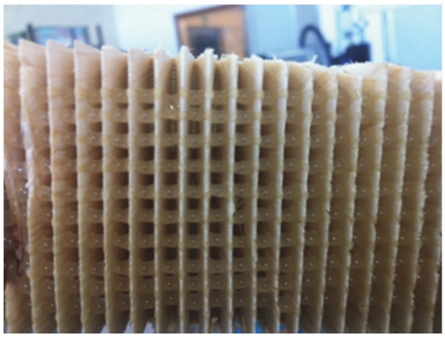

Figure 12. Contactless application.

<u>Roller spreader application</u>

Too low viscosity at high shear will give dripping and spill of adhesive (see Figure 11, roller spreader application).

<u>String application</u>

For this application equipment, it is an advantage with low viscosity at high shear. This is to ensure easy pumping, good string picture even at low application. The high viscosity at low shear is also important for the strings to stay on the wood after application and during handling (see Figure 11, string application).

String application of adhesive for production of beams is shown in Figure 13.

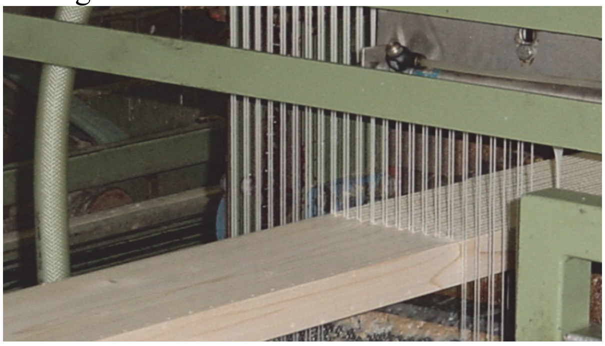

Figure 13. String application.

SUMMARY

The rheological methods show good correlation to more traditional and time consuming testing on wood, and for some properties it tells us more about the adhesive systems. Rheological methods save both time and resources; it is a helpful tool to understand the new adhesive systems and their gluing performance.

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