HEMIC-OAT

Conversion of hemicellulose to coatings

36th CORNET call

D2.3

Report of potential adhesive properties of the oligomers and feasibility tests

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Disclaimer

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The views expressed in this HemiCoat Deliverable *D2.3 Report of potential adhesive properties of the oligomers and feasibility tests,* which concern the possibility to use oligomers solutions as ingredients to obtain wood adhesives, are the sole responsibility of the author and do not necessarily reflect the views of the industries of the User Committee. The User Committee is not responsible for any use that may be made of the information contained therein. The author does not accept any liability for any direct or indirect damage resulting from the use of this Deliverable to its content or parts of it. The results achieved, conclusions made, and recommendations given by the author should not be interpreted as a political or legal signal that the European Commission or any other political or legal institution intends to take a given action.

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1. Introduction

The purpose of this report is to evaluate the use of oligomers, derived from the main process, as an adhesive for wood. Various tests were conducted during the project using commercial xylan to determine several parameters:

- The manufacturing method
- The compatible additives
- Optimal ratio determination
- Impact of wood type on results

Next, the oligomer solution provided by the partners was tested using the optimized parameters determined during the preliminary tests.

2. Tests with commercial Xylan

In this part, all the tests were performed with commercial xylan, derived from corn. The commercial reference for the wood adhesive is a vinyl wood glue, found in a store.

2.1. Method Description

First of all, the manufacturing method was determined by varying different parameters like drying time; drying temperature; weight during drying; surface preparation, dry content for the adhesive.

The established method for the tests described in the following report is as follows:

1. The wood samples are first cut to the correct length to allow for the adhesion test.
2. The Xylan/additive mixture, or the Oligo/additives mixture, is then applied to the designated bonding area. The two strips are then brought into contact, and a 2-kg weight is placed on top of them for the duration of the drying process.
3. Drying step takes place at room temperature for at least 16 hours.
4. The samples are then retrieved and tested on a test bench to collect data on the adhesive's failure.

Most of the tests were conducted on fir wood, which is easier to obtain, but in the section 2.4 other types of wood were also tested as substrates.

Preliminary tests have shown that a concentration of at least 50% is necessary to ensure there is enough product between the wood panels after drying.

2.2. Choice of Additives

According to the literature, it is possible to produce a satisfactory wood adhesive by combining molecules with nitrogen-containing functional groups with oligomers found in wood hemicellulose.

With a view to preserving the bio-based nature of this adhesive, various options were considered and tested:

- Chitin nanofibers in solution
- Chitosan solution
- And polyethyleneimine as comparative

The results obtained with these various additives are shown in Figure 1 below.

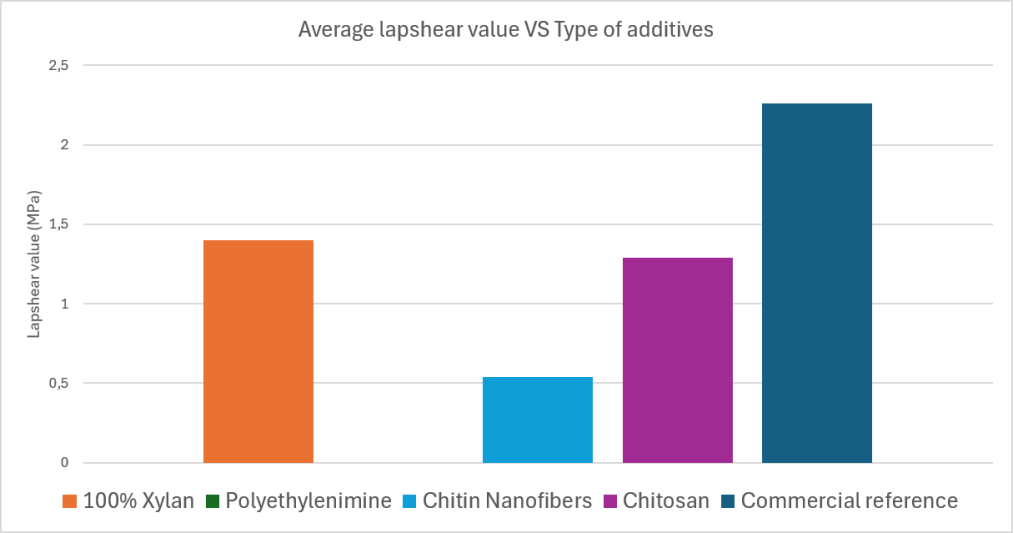

Figure 1: Adhesive properties of different additives with commercial xylan with the same ratio.

The results show that, for the same mixing ratio, chitosan appears to be the additive most compatible with oligomer-type molecules such as xylan. We also note that the adhesion value obtained is comparable to that of xylan without an additive; however, this demonstrates that the mixing ratio needs to be optimized. The chitosan solution was therefore selected for further testing.

2.3. Choice of ratio

Once the additive was selected, it was necessary to determine the correct mixing ratio to ensure that the mixture's adhesion properties were optimized. Various ratios were tested, and the results are shown in the graph below (Figure 2). Each ratio corresponds to the weight equivalent of the additive relative to the amount of xylan. For example, a 1:25 ratio corresponds to 1 g of dry chitosan per 25 g of dry xylan. Of course, the mixtures are prepared as solutions so they can be applied to wood.

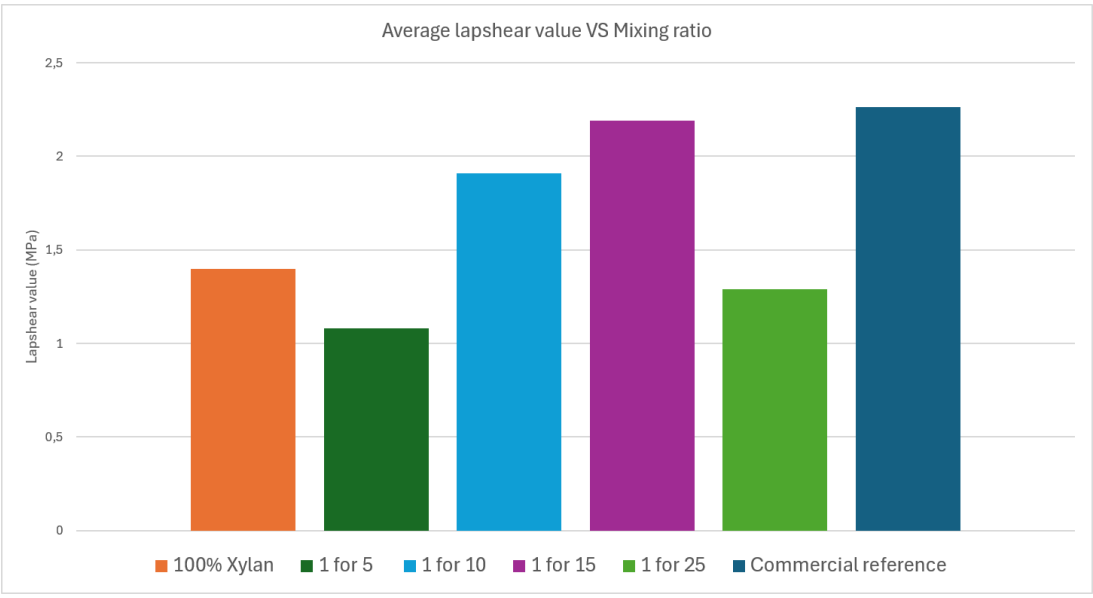

Figure 2: Adhesive properties of different ratios of xylan with chitosan

Tests conducted with different ratios using chitosan show that two mixing ratios clearly stand out from the rest and yield good adhesion properties: ratios of 1:10 and 1:15. These ratios will be used for the rest of the tests.

2.4. Tests on different woods

Since it is difficult to obtain the bond strength values of commercial adhesives with fir wood without it breaking, and because testing standards for wood adhesives are based solely on beech wood, tests using wood species other than fir have been performed.

The results are summarized in this graph below (Figure 3): the light-colored bars represent a 100% xylan solution for each wood species; the dark-colored bars represent a 10:1 xylan-to-chitosan mixture for each wood species; and finally, the brightly colored bars represent the commercial reference for each wood species.

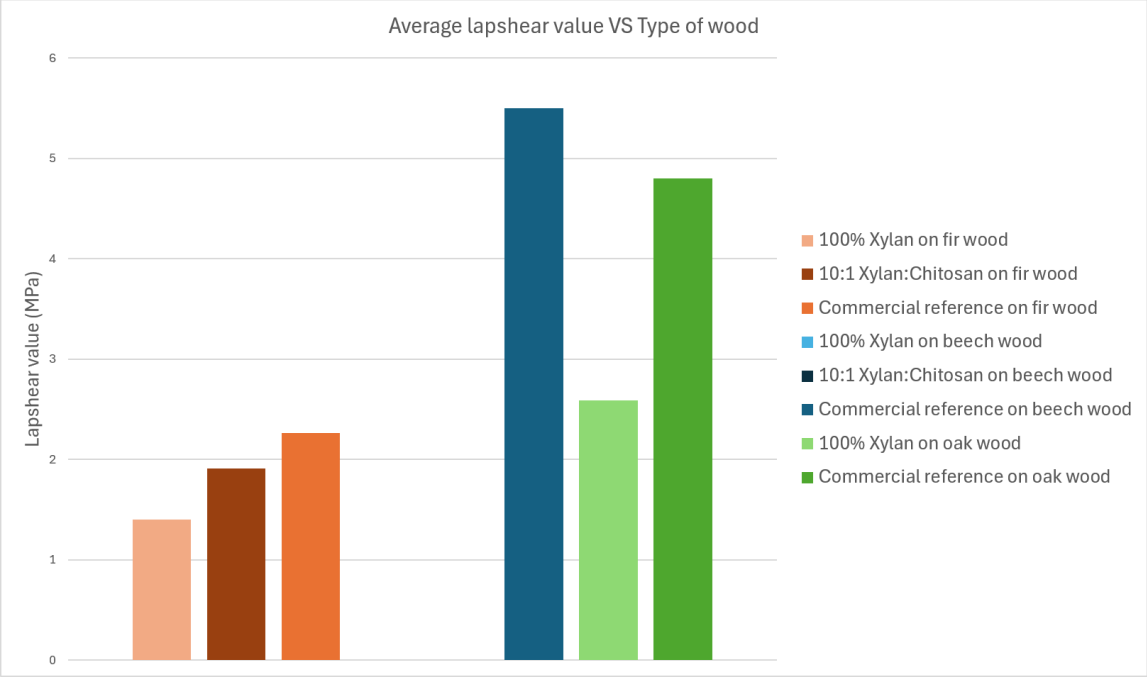

Figure 3: Adhesive properties of xylan, 10:1 xylan:chitosan and commercial reference on different types of wood: fir wood (orange); beech wood (blue); oak wood (green)

It is very difficult to obtain adhesion values for xylan—with or without additives—on beech wood: the wood test specimens broke apart when mounted on the test bench. It is quite possible that this is due to an incompatibility between commercial xylan and beech wood, as well as a difference in porosity between beech wood and the other wood species tested.

In order to continue comparing the adhesion properties of xylan with those of the oligomer solution provided by our partners, it was therefore decided to continue testing on fir wood only.

3. Tests with Oligomers solution

The oligomer solution was sent to us by our partners during the M17 of the project.

3.1. Comparative tests

First, we wanted to test the adhesive properties of the oligomer solution in combination with chitosan at the two most promising ratios determined previously: 10:1 and 15:1.

In the graph below, the results of the adhesion tests on commercial xylan and chitosan are shown in blue; the results for the oligomer solution and chitosan at the same mixing ratios are shown in green; and the results for commercial wood glue are shown in orange. All tests were conducted on fir wood.

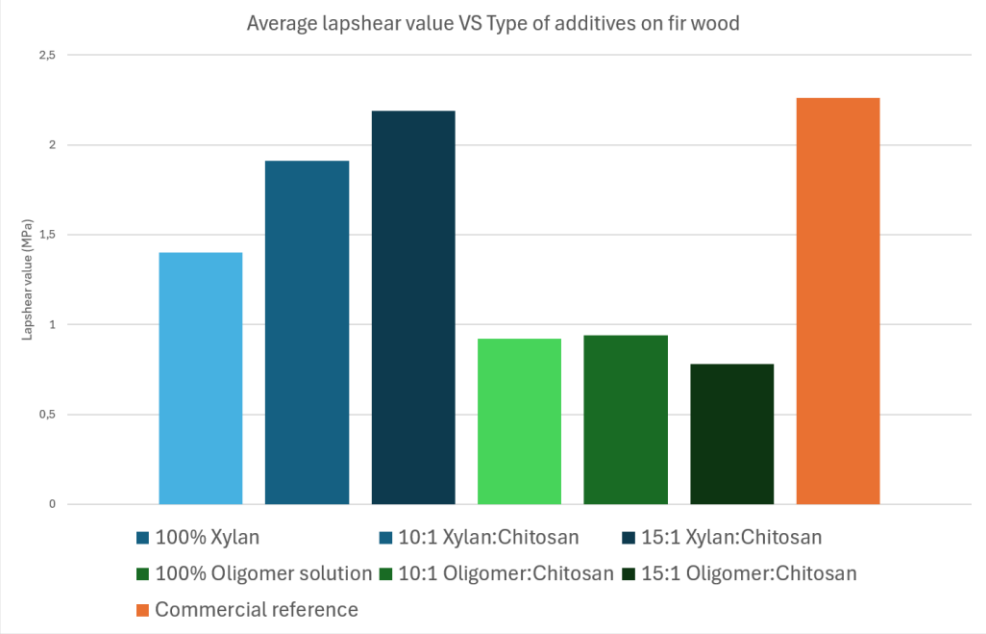

Figure 4: Adhesive properties of oligomer solution with chitosan (green) compared to commercial xylan with chitosan (blue)

As can be seen in the graph, the results with the oligomer solution are significantly lower than those obtained with commercial xylan, regardless of the mixing ratio tested. Furthermore, during manufacturing, it is very common for several test specimens to break during drying or when placed in the testing machine, thereby demonstrating weak adhesion between the wood plates.

It was also noted that the drying time was much longer with the oligomer solution, and that for some test specimens, the oligomer/chitosan mixture never really dried, remaining viscous. To try to reduce drying time and improve drying, several test specimens containing an oligomer solution were placed at different temperatures as shown in Figure 5.

Figure 5: Aspect of oligomer solution after drying at 70°C (left); at 120°C (middle); at 150°C (right)

The sample placed at 70°C retained its original appearance and remained viscous after 3 days at 70°C. The samples placed at 120°C and 150°C appeared swollen, with bubbles forming before hardening. It is therefore impossible to improve drying by increasing the temperature without damaging the mixture.

This is more of a polymerization issue than a drying issue, and after discussing it with our partners, it turns out that the oligomer solution still contains a high amount of sugars: the concentration is 60% dry content, of which 40–45% is sugars. It is therefore important to determine the effect of sugar on adhesion properties.

3.1. Impact of sugar content

To determine the effect of sugar on the adhesive properties of oligomers, a solution of C5-C6 sugars obtained from the fermentation department was used to "contaminate" a xylan-chitosan mixture. The xylan-chitosan mixture was prepared in a 10:1 ratio, and the sugar solution was added afterward, varying the percentage of sugar added to determine the minimum amount at which adhesion remains possible. The results are shown in the graph below (Figure 6).

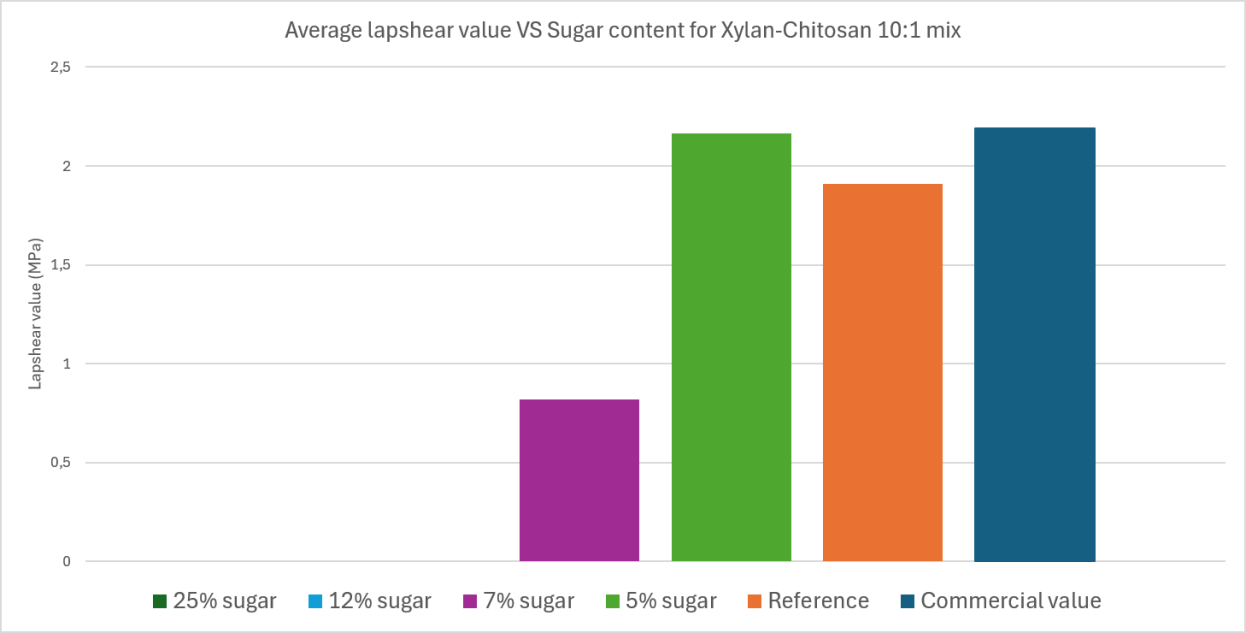

Figure 6: Adhesive properties of the Xylan/Chitosan solution as a function of sugar concentration

The results show that the sugar content must be below 10% for adhesion to begin, and to ensure that there is no impact on adhesion properties, the sugar content must not exceed 5%.

4. Conclusion

It is possible to make wood glue using an oligomer solution, provided certain criteria are met. A process must be developed to produce an oligomer solution with a concentration of at least 50% and less than 5% sugar in the mixture.

It is necessary to continue testing with a solution free of sugar contamination in order to confirm the feasibility of the adhesion of the oligomer-based adhesive. The use of certain molecules may also promote the polymerization of the oligo-chitosan solution, and this deserves further testing.