SUSBIND

DEVELOPMENT AND PILOT PRODUCTION OF SUSTAINABLE BIO-BINDER SYSTEMS FOR WOOD-BASED PANELS

D3.5 Pilot-scale evaluation of the final epoxy lipid

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1 Introduction

The deliverable D3.5., "*Pilot-scale evaluation of the final epoxy lipid*" is a result of the task T3.5: "*Epoxy lipid selected as from T3.3 and, eventually, the necessary chemical to improve their performances will be submitted to WP4.*"

Indeed, between the deliverable of the task T3.3 (leading to *D3.3: Laboratory-scale evaluation of epoxidized lipids as bio-binder ingredient*) and the D3.5 there is an important task, T3.4, related to the scale up of the most promising epoxide which deliverable, D3.4, is about: *"Scaling-up lipid epoxidation with the selected enzymes"*.

It is then clearer that the scope of D3.5 was to guarantee that all necessary chemicals from WP3 would be available for demo production of wood board as expected in the WP4. In other words, deliverable D3.5 was set to mitigate project risk: in case there was not enough epoxides delivered from WP3, Cargill would have to provide commercial grade vegetable oil epoxides for the project.

Considering the good results of T3.4, the deliverable D3.5 could then considered redundant because the epoxides from WP3 were in fact available at demo scale and they could be delivered to WP4 both for Particle Board (PB) and Medium Density Fibreboard (MDF) pilot production.

With this argument, D3.5 could have been delivered far before the expected deadline with a simple "*nothing to report*"; however, the team decided instead to stretch the challenge and extended the expectation i.e.:

a) What commercial product could match the properties of the epoxides delivered from T3.4?
b) Beyond the functionality as hydrophobic agent, in what chemistry could the epoxides be used as bio-based binder for wood board production?

D3.5 has been then split in two chapters:

- **Commercial match to bio-based epoxides**
- **Alternative use of bio-based epoxides as co-Binder for MDF**

This extension of the scope of task inevitably affected the deadline of D3.5 but it makes sense to leverage the talents across the consortium rather than respect the deadline without anything to report.

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2 Commercial match to bio-based epoxides

The objective of WP3 is to develop a new enzymatic technology for the epoxidation of unsaturated plant fatty acids and oils to be used as bio-based ingredients for binders in the production of wood-based panels.

The double bonds present in the vegetable oil are converted to epoxy rings, which can then be opened in the process and react to form a thermoset network resulting in a strong and water-resistant composite of wood chips or fibres and binder.

Since epoxy rings can only be created at the site of the double bond, it can hence be deducted that the higher the degree of unsaturation of a fatty acid or oil, the higher the reactivity of the final product will be.

In the selection of the most suitable vegetable oil as a feedstock for developing the SUSBIND binder, the degree of unsaturation is therefore considered an important criterium.

The Table 1 below indicates the average composition of the oils considered in the SUSBIND project:

Table 1: SUSBIND oil composition

	SOYBEAN	RAPESEED	SUNFLOWER SEED	LINSEED	MAIZE GERM
Caprilic	0,0	0,0	0,0	0,0	0,0
Capric	0,0	0,0	0,0	0,0	0,0
Lauric	0,0	0,0	0,0	0,0	0,0
Myristic	0,0	0,5	0,0	0,2	0,0
Plamitic	11,3	4,5	6,2	6,0	6,5
Palmitoleic	0,1	0,0	0,1	0,1	0,6
Stearic	3,6	1,5	3,7	4,2	1,6
Oleic	24,9	60,0	25,2	20,7	65,6
Linoleic	53,0	20,0	63,1	15,4	25,2
Linolenic	6,1	10,0	0,2	52,5	0,1
Arachidic	0,3	0,5	0,3	0,3	0,1
Gedoleic	0,3	1,5	0,2	0,2	0,1
Behenic	0,0	0,5	0,7	0,2	0,0
Erucic	0,3	1,0	0,1	0,1	0,1
Lignoceric	0,1	0,0	0,2	0,1	0,1
Nervonic	0,0	0,0	0,0	0,0	0,0
Saturated	15,3	7,5	11,1	10,5	8,1
Monounsaturated	25,6	62,5	25,6	21,6	66,5
Polyunsaturated	59,1	30,0	63,3	67,9	25,4

This table already gives a good idea about the unsaturation of the different oils, but there is a further simple index to directly compare different oils according to their degree of unsaturation: the **iodine value**.

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The iodine value is expressed in grams of iodine which react with 100 g of the respective sample when formally adding iodine to the double bonds as seen in Table 2.

Table 2: Iodine Values

	SOYBEAN	RAPESEED	SUNFLOWER SEED	LINSEED	MAIZE GERM
Iodine Value	130-170	100-130	130-170	>170	100-130

Considering the fatty acid composition of the 5 major oil crops, **linseed oil** would be the **technically** most promising feedstock. Indeed, epoxidized linseed oil, obtained by chemical conversion on linseed oil, is a common chemical in the industry; one example is **Vikoflex® 7190**:

Vikoflex® 7190, is an epoxidized linseed oil produced by Cargill. It is recommended for PVC homopolymer and copolymer plasticization and stabilization in a host of rigid, semi- rigid, flexible extruded, calendered, and moulded compounds. Vikoflex® 7190 is well suited for formulations requiring maximum oxirane content and efficient acid scavenging.

Table 3:Vikoflex® properties

PHYSICAL PROPERTIES

% Oxirane Content	9.2%
Viscosity at 25°C (cps)	800
Specific Gravity	1.03
APHA Color	175
EEW	174
Moisture Content (%)	< 0.05
Acid Value (mg KOH/g)	0.2
Iodine Value	2.5

The challenge, presented to Fraunhofer, was to convert linseed oil, via enzymatic route, and deliver a product as close as possible to the Vikoflex® 7190 which is a very highly epoxidized oil.

Fraunhofer was able to produce 350g of enzymatically epoxidated linseed oil (**LSO-TG-Epoxides**) starting from refined oil provided by Cargill. The sample was analysed by TLC and epoxide titration, resulting in a respective oxirane oxygen content (OO) of ~8.34%. The epoxide yield Y (OO) related to the amount of available double bonds in the substrate was therefore ~85% which is a little lower than the commercial benchmark but acceptable to compare the performances as co-binder.

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3 Alternative use of bio-based epoxides as co-binder for MDF

The first US patent for soya bean adhesives was issued to researcher Otis Johnson in 1923, although in fact, it was the American industrialist Irving F. Laucks who pioneered the development of soya bean-based glues in the early years of the 20th century. Laucks began selling his soya bean glue in 1923 as an improvement over the often-used casein glue systems. (*Sticking power from soya beans*, James Wescott and Charles Frihart, C&I Issue 3, 2011).

Since then, soy-based adhesives have been developed for industrial uses in wood manufacturing, particularly for interior uses. This situation continued into the 1960s, at which time the price of petrochemically based adhesives had become so low that they literally displaced protein adhesives from their traditional interior markets. Specifically, phenolic and urea–formaldehyde resins replaced blood, soybean, and starch glues in all plywood and composite wood panels; resorcinol–formaldehyde resins replaced casein glues in lumber laminating and millwork applications; and poly(vinyl acetate) and acrylic emulsion glues replaced virtually all collagen adhesives (animal and fish bone/skin derived) from furniture, musical instruments, and general interior wood assembly. (Wood Handbook: Wood as an Engineering Material, Agriculture Handbook 72, U.S. Department of Agriculture Forest Products Laboratory, Madison, Wis., 1987).

Soy flour adhesives are today discovering a second life, mainly because the opportunity to deliver an adhesive with lower GHG emission than incumbent resins. Still soy-based adhesive, once used for engineered wood board such as MDF, show always lower performances than MUF resins, both for mechanical properties, such as Flexion, Module of Elasticity, and Internal Bonding, as well as for water resistance, expressed as Swelling.

The SUSBIND project offered the opportunity to test if Epoxidized Linseed Oil (**ELO**) can close the performance gap of **soy flour adhesive** in the production of MDF. The combination of these two ingredients is interesting for sustainability arguments and it has a simple rationale: provided ELO reacts at the MDF production conditions, its hydrophobicity could reduce the sensitiveness of soy adhesive to moisture. Besides, a "reactive oil", such as ELO, could also burst the adhesive properties of soy flour because of the reactive ends of this protein.

The objective of D3.5 was then re-defined to test the opportunity to use ELO as co-binder in a soy flour adhesive system for MDF. Both the commercial ELO, **Vikoflex® 7190**, and the one synthetized by Fraunhofer, **TG-epoxide**, were sent to Valbopan where they prepared 3 different adhesives:

- SBLSOM26: soy flour dosage 15% on dry wood
- SBLSOM25: soy flour dosage 15% on dry wood + 20% on dry soy flour Tg-epoxide from Fraunhofer
- SBLSOM27: soy flour dosage 15% on dry wood + 20% on dry soy flour Vikoflex® 7190 from Cargill

The scope of the test was to compare the properties of pilot MDF panels produced with these different adhesive systems and check if ELOs could enhance the panel properties once compared to adhesive based on neat soy flour.

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The results of these very preliminary test are reported in the following table.

Table 4: Preliminary soy flour tests

Binder Type	Sample nº	Thickeness	Density	Flexion	MOE	Internal Bonding	Moisture	Swelling 24h
		mm	(≥860 kg/m^3)	(MDF≥ 50 N/mm^2)	(MDF ≥ 5000 N/mm^2)	(MDF ≥0,65 N/mm^2)	(4-11%)	(MDF≤ 35,0 %)
Soy flour 15,0%	SBLSOM26	3,5	1028	46	3987	0,64	5,84	77,98
Soy flour 15,0% and ELO (TG-epoxide) 20% on Soy	SBLSOM25	3,57	1082	33	2809	0,80	3,42	73,09
Soy flour 15,0% and Vikoflex® 7190 20%	SBLSOM27	3,57	1077	52	4486	0,99	5,64	64,43

As expected, despite the relatively high dosage of adhesive MDF obtained with neat soy flour, SBLSOM26, delivers MDF board with very poor properties.

The addition of ELO, specifically the one produced at Fraunhofer, SBLSOM25, improves the situation for internal bonding which is above the required value of the specification for this board type (0,80 Vs 0.65 N/mm^2).

Finally, very promising are the results with Vikoflex® 7190 were nearly all properties are acceptable except swelling which remains to high compared to specification.

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4 Conclusion

The preliminary results obtained by the combination of soy flour and ELO as adhesive system for MDF are very promising because nothing has been made to optimise the system and still, the measurements are not far from industrial standards.

The tests showed that ELO actively contributes to the adhesion mechanism, although the application conditions are mild, and it is very unlikely that epoxy rings open and react. Indeed, heat exchange is probably low to promote a reaction of ELO and the addition of appropriated catalyst could enhance the performance of the adhesive system.

Besides the solution, although it brings complexity to the MDF production site, it is manageable via dry dosage of soy flour into the wood chips and further spraying of the ELO on the chips/flour mix.

In conclusion, the combination of soy flour and ELO offers an interesting opportunity to develop meaningful adhesive systems for industrial production of MDF.

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