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From the Lab to Commercial Reality with Biobased Adhesives for Wood

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ABSTRACT: Many technologies can be demonstrated in the laboratory to give products that meet performance standards, but there are many hurdles to overcome before these products are commercially viable. Demonstrating performance under simulated commercial processes conditions is the first key step to be accomplished through using appropriate adhesive application, furnish selection, and bonding conditions. Then, after evaluation of economic feasibility, comes the need to scale up adhesive production on a consistent basis. With this accomplished, the adhesive needs to be applied and bonded in a commercial scale trial in a customers' plant. If the adhesive passes all these steps, then comes all the logistic, plant modification and economic factors along with the customer's real desire for taking on the risk of change. It is always harder to do a technology push than a market pull and the technology needs to match the window of opportunity for consumer demands.

KEYWORDS: Biobased, adhesives, wood composites, commercialization

1 INTRODUCTION

The field of wood adhesives is varied and complex, [1-4], which makes the development of new adhesives very challenging. Considering just making panel and structural products shown in Figure 1, the adhesives may be used for laminating or as binders. Thus, wood adhesives are often developed with a certain use in mind.

Figure 1: *Examples of wood products, all made with biobased adhesives.*

In exploring new adhesives, research often stops at the stage of an initial laboratory proof of concept. This is attributable to the fact that often for academics and entrepreneurs the most important aspect of new product development is the proof of concept which can take substantial investment of time and other resources to accomplish. On the road to commercialization however, this is the starting point, not the end point.

For many researchers, the path toward commercialization is not well defined. Laboratory work is typically focused on meeting specific performance metrics as defined by an industry standard. Materials are carefully selected and conditions optimized to obtain samples with maximum performance and reproducibility. While this is necessary and valuable in development of new adhesive systems, it provides limited information on how the adhesive will perform in a commercial setting. Before presenting the proof of concept to an industrial collaborator or potential customer for commercialization trials, it is necessary to consider the many factors required for a new product to be successfully commercialized. Better yet is to do this in the early stages of product development.

There are three areas to consider: scale-up, process conditions and performance deliverables. In scale-up, there must be confidence in the reproducibility of the product as well as the robustness of the make down procedure. The environmental, health and safety (EH&S) of the raw materials and the final product must be considered as well as the cost and availability of the raw materials. If you have access to a process engineer, involve them early on to be able to scale-up your process. Process conditions include factors such as the mixing, pumping and application of the adhesive, the shelf life and/or pot life of the adhesive, the balance of moisture in the system, the variability of the adhesive ingredients and substrate(s) and EH&S concerns for storage, use and

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disposal of unused material to name just a few of the most important ones. Performance deliverables may be prioritized differently by different customers or extend beyond those mentioned in the standards for a given market including unofficial tests that are specific to a given customer or even an individual mill. The more information that can be gathered before the proof of concept and certainly before attempts at commercialization, the greater the chances are for moving beyond this stage.

There is currently a large effort to make new bio-based adhesives to replace those derived from fossil fuels. In order to increase the chances for success of this endeavour, it is important to understand the steps from the laboratory proof of concept to commercial utility. Although each product has its own tortuous path, the information contained herein is intended to provide some insights based upon our experiences in tracking the development and commercialization of new adhesive products.

2 LABORATORY TESTING

Developing a new adhesive requires a great deal of creativity and diligent work. However, demonstrating that a new adhesive is commercially viable requires much more than laboratory demonstration.

2.1 PRODUCT REPEATABILITY AND PROCESS ROBUSTNESS

Development and optimization of a new adhesive formulation is a complex iterative process well suited to laboratory studies. Making controlled changes to one variable or additive at a time is detailed work that requires flexibility and freedom from other constraints in order to identify the maximum potential of a new adhesive system.

Table 1: *Some generalized product adhesive criteria with DPW= decorative plywood, PB=particleboard, MDF= Medium density fibreboard, OSB=oriented strandboard, LVL=laminated veneer lumber, viscosity in Pa.s, and WR= water resistant*

	% solids	Viscosity*	Other
DPW	30-50	1000-3000	Bond a variety of wood species, no bleed through, Moderate WR
PB	50+	200-500	Distribute well in blender, moderate WR
MDF	40+	200-500	No spotting,, moderate WR
OSB	50+	200-500	Atomize well, good WR
LVL	50+	1500	High WR, fire and heat resistance

*These values are reported for Newtonian liquids. For non-Newtonian materials such as protein based adhesives, these values will vary greatly depending on the shear associated with the viscosity measurement.

While it is important to know the best conditions for making an adhesive, it is equally critical to be able to make the same polymer blend or formulation repeatedly, as determined by viscosity, molecular weight or some other set of observables. It is important to realize that the targets very much depend upon the specific application, as illustrated in Table 1. This is also a good point to plan in detail how to overcome some major hurdles that the technology needs to address; an example in Table 2 lists these that were addressed in developing soy adhesives.

Table 2: *Some generalized product challenges that improved soy adhesives need to address.*

Past Soy Problem	Solution
1) Biologically unstable	By proper denaturing and copolymerizing with small amounts of reactant, the product is believed to be rendered biologically stable. We are continuing to work in this area.
2) Low Solids	By developing a new method for denaturing and copolymerizing with viable crosslinking agents, the soy resins are offered at solids ranging from 30-45%
3) Slow Press Times	By copolymerizing with reactive crosslinking agents, slow press times are not an issue as the cure rate can easily be tailored to meet a variety of applications
4) Poor Water Resistance (durability)	By copolymerizing with reactive crosslinking agents, the soy resin actually becomes a water resistant thermoset resin
5) Very short pot life	By innovative processing, the soy resin's shelf lives range from 2 weeks, like PF resins, to 1 year.

Upon completion of the laboratory proof of concept, it is essential to take the next step and investigate the robustness of the process in anticipation of commercial application. While the specifics of this process will vary, there are several aspects that will be consistent regardless of the end use. For a formulated adhesive system, one should evaluate the effect that order of addition has on resultant properties and performance. How sensitive is the process to changes including mixing conditions, temperatures, times, ratios of components, etc.? This is especially true when products will be formulated by a toll manufacturer or by a customer directly prior to use. The dynamics of all these change from a laboratory to a scaled up process given the change in surface to volume ratios and different localized mixing. It is important to know what method(s) are to be used to monitor the adhesive formulations' progress.

Additives may interact with one another in the absence of other ingredients and this should be evaluated and understood. Review the procedure and consider how this process will go **when completed by an operator who does not know as much as you do.** For example, in a particular soy-based adhesive system, the process for making the end product involved charging water, polymer, viscosity modifier and soy flour followed by several other additives. On one particular shift, the operator charged the water, polymer and viscosity modifier before going to lunch. The operator figured this would save time when returning from lunch and the remainder of the ingredients could be added without issue. Unfortunately, in the absence of the soy flour, the viscosity modifier reacted with the polymer resulting in greatly reduced performance of that batch of adhesive material. This happened on numerous occasions and proved to be a difficult issue to define. It was only after a fairly rigorous investigation that the change in normal operating procedure was discovered and the procedure was modified to prevent this from happening in the future. It will also be necessary to understand the effect of over or under charging of each component as related to the properties and performance of the material. Not only will this provide a better understanding of the system, but it will also provide an excellent base for troubleshooting down the line as the commercialization efforts continue. Much of this information comes naturally from the optimization stage of the formulation, however considering the data specifically in light of commercial application will add value to the project and increase the chances for commercial success.

In a perfect world, the recipe will always be followed, but in reality changes are sometimes made. Knowing ahead of time what particular changes look like adds credibility and prevents the need to stall commercial activities in order to do this analysis. While it is difficult to anticipate everything that may go wrong, it is a small task to proactively anticipate common mistakes and prevent the need for reactive analysis.

2.2 RAW MATERIALS

The chemicals used in a process are generally consistent if they are small molecules. However, large molecules, especially bio-based, can vary considerably from lot-to-lot depending on season, location, and storage age and conditions, vendor and other aspects; understanding these variations is critical to success. For economic reasons, commercial operations will use the lowest grade available; thus, it is important to understand the differences in quality one may encounter and where possible, use commercial chemicals rather than laboratory grade. Long-term testing of different raw material lots is very important to know that the material is consistent and reliable. In the event one discovers the material does exhibit variation affecting properties or performance, work can begin to understand the quality parameters that maybe used to qualify raw material lots and vendors. This issue becomes even more important for oligomeric or polymeric raw materials because they tend to be less uniform. If the variation is not easily controlled or eliminated, then at least it can be anticipated and/or managed before it is discovered by the customer or commercial collaborator.

2.3 PERFORMANCE TESTING

Performance testing is more complicated for wood than it is for other substrates due to the fact that wood is porous and does not have a well-defined surface. Porosity not only varies with species, but also between samples and across a surface. A surface too dense limits adhesive penetration which leads to decreased mechanical interlock whereas a surface too porous can lead to over-penetration and a starved bondline. The viscosity and composition of an adhesive has a big effect on penetration, but they are not the only drivers. The other aspects involve the variable surface of the wood substrate.

Table 3: Examples of some differences in veneers between laboratory studies and commercial testing for interior plywood.

	Laboratory	Commercial
Species	Yellow Poplar	Douglas Fir
Grade	High	Low
Wood Moisture (%)	6	4
Adhesive Load (g/in^2)	0.25	0.13
Press Temp. (°C)	200	170
Press Pressure (psi)	200	100
Press Time (s)	600	300

In designing laboratory tests to evaluate adhesive performance, the commercial process must be considered. As an example, consider the two conditions shown in Table 3. Although somewhat exaggerated, it is not uncommon for laboratory test procedures to be significantly different than those of a commercial process. It is not always possible to do the proof of concept work using a defined commercial process, however once a commercial application is identified, it is always necessary to evaluate a new product under commercially relevant conditions. The materials used to produce samples must be of similar quality and composition to those that are used commercially. In preparing a sample for testing, the adhesive must be applied at a commercially acceptable level and be bonded at a time, pressure and temperature that are commercially appropriate. Knowing the influence of curing conditions can help to anticipate how changes in the process can affect performance. If, for example you are making a thicker product, where the time at a given temperature is less, then you know how to alter press time in order to obtain full cure. There is equipment available to measure strength development under different times and temperatures to understand cure rates. Additionally, because of the scale, commercial equipment entraps more moisture due to the less edge space for moisture to leave the panel.

2.4 ECONOMICS

A complete material balance flow sheet should be used for calculating the economics. By-products are usually a significant detriment because they take valuable raw material and convert it to a low cost or even a negative value if it has to be disposed. It is important to make sure the process makes sound economic sense before proceeding. Marginal economic advantage and the lack of a strong market driver make it very unlikely that any process will go beyond proof of concept. Before proceeding further, a more complete market assessment must be done and it is necessary to find a suitable potential customer for commercial trial and assessment of market conditions. A commercial partner or advisor is important because they have already been through many problems and failures. Ask lots of questions about problems they foresee because this will increase your knowledge and help you to improve your chances of success by being able to address them early in the process.

3 ADHESIVE SCALE UP

3.1 PRODUCT

Before doing the scale-up chemistry, it is necessary to define the most suitable product for the trial at a potential customer. Many factors need to be considered as listed in Tables 4 and 5.

Table 4: *Some examples of process deliverables that need to be addressed.*

	Process Deliverables				
	HWPW	EWF	MDF	PB	OSB
Adhesive Cost	✓	✓	✓	✓	✓
Pot Life	✓	✓			
Storage Stability			✓	✓	✓
Sprayability			✓	✓	✓
Spreadability	✓	✓			
Distribution			✓	✓	✓
Open/Stand Time	✓	✓			
Press Press	✓	✓	✓	✓	
Tack	✓	✓	✓	✓	✓
Dry-out	✓	✓			✓
Pre-Cure			✓		
Press Time	✓	✓	✓	✓	✓
Press Sticking			✓	✓	✓
Clean-up	✓	✓	✓	✓	✓

Table 5: *Some examples of performance deliverables of the product that need to be addressed.*

	Performance Deliverables				
	HWPW	EWF	MDF	PB	OSB
MOR			✓	✓	✓
MOE			✓	✓	✓
Internal Bond			✓	✓	✓
Thickness Swell			✓		✓
Linear Expansion			✓	✓	✓
3-Cycle Soak	✓	✓			
Bleed-Through	✓				
Dry Wood Pull	✓	✓			
Final Panel % MC	✓	✓			
Surface Hardness			✓	✓	✓
Standard Source	HPVA/ANSI	HPVA/ANSI	CPA/ANSI	CPA/ANSI	USDC/CSA
Standard Number	HP-1-2004	EF-2002	A208.2.2002	A208.1.1999	PS2/CSA437

This involves formulating the adhesive system so that it bonds with all the substrates (wood species, veneer vs. composites, etc.) a given customer uses and doing so under commercially relevant conditions. Furthermore, the formulation must also be compatible with the application equipment. If, for example, the application is going to be sprayed, then one must understand the viscosity requirements, nozzle specifications (aperture size, pressure/flow limits, etc.) and pump requirements for a given adhesive formulation. If one is using a water-based system in a process that uses heat and pressure to cure the product, it is important to understand the moisture limits of the process, both upper and lower, to prevent excessive steam generation, which may lead to steam blows, or insufficient heat transfer, which may lead to product delamination. One must consider the effect process conditions may have on viscosity, density and stability. For a non-Newtonian system, such as proteins, it is important to consider how the sheer forces on the adhesive may change throughout the process thereby affecting the viscosity at various stages within the process. There may be an aspect of the process that entrains air into the system, thereby reducing the density and changing the control one has on application rate. Understanding the temperature at each stage of the process is another important aspect of formulating the product to have appropriate stability throughout the process. Other process variables, such as process time(s) and green strength (tack) must also be considered when selecting a product and formulating for end use.

3.2 PROCESS

Scaling up can create many issues, but heat transfer, mixing, and cycle time are the most common. Heat transfer, either into or out of the reaction, is much more difficult at commercial quantities since the surface area scales as a square function while the volume scales as a cubic function. In addition, the process on a larger scale may have additional variables to overcome due to hang up on internals, poorer vertical mixing, residue left from the prior reaction, etc. The decreased surface to volume ratio makes it hard to get added components and the temperature uniformly distributed as rapidly as can be accomplished in the laboratory, especially as the viscosity of the reaction mixture increases. Figure 2 shows an example of the size difference between a laboratory and pilot reactor.

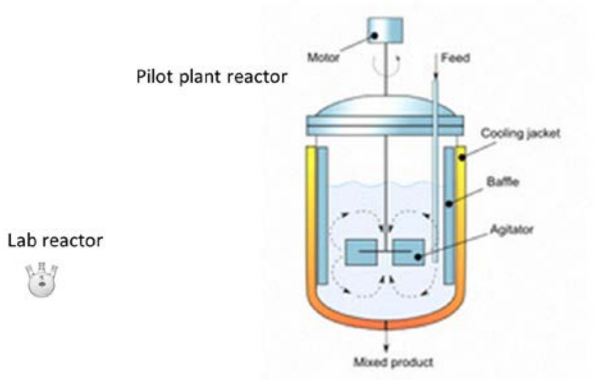

Figure 2: Illustration of size and shape difference between a laboratory flask on the left and a pilot scale reactor on the right.

Heating and cooling times are longer in larger reactors making exotherms and endotherms more difficult to predict and control. For reactions that are sensitive to temperature, one must consider the effect of longer heating and cooling times and the effect they may have on the final properties of the adhesive system. Considering the challenges of scale up early in the process will increase the chances for commercial success and decrease the time required for commercialization.

3.3 COMMERCIAL TRIALS

No matter how much effort is put into generating commercially relevant data in the laboratory, a technology is not considered viable without commercial demonstration. Plant trials are costly for a variety of reasons. Equipment may need to be brought in and time taken to set up, test and validate its operation. The materials to be used, both the adhesive system as well as the substrate, add cost, especially if the trial produces unusable or downgraded product. There will be a cost associated with the lost production time in setting up, switching over and switching back to normal operations. Finally, there is the cost and time of travel for individuals from the supplier as well as the customer to attend the trial. The better that one understands how the commercial process works and how to control it, the more likely one is to be successful in the plant trial.

There is great reluctance to change from normal procedures for running a trial. A bad trail can make a poor and lasting impression, especially if you do not know at the time of the trail what went wrong.

3.3.1 PREPARATION

The chance of success at any trial is increased in proportion to the level of preparation. Without a decent showing, the chance of being allowed a second trial is minimal. This means understanding, in detail, the operation, production monitoring, and product specification and testing. Know what equipment and operating regimen is needed for your adhesive to have a fair evaluation.

Before any trial, visit the trial location, more than once if possible. Spend time walking the line to understand the flow and timing of the process. Consider the lab process and how it is similar to or different from what you observe in the plant. Take special note of when, where and how monitoring and quality samples are taken and evaluated as well as how this relates to the testing developed in the lab. Observe issues encountered during normal production and analyse how those issues may be magnified or diminished when using the new adhesive system. Talk to the **operators and plant personnel** to get an understanding of their priorities, observables and strategies for overcoming unexpected issues. In nearly every manufacturing plant, there is one or two individuals who can get anything done. Ideally it is the plant manager, but that may not always be the case; sometimes it is a supervisor, an electrician, a pipe-fitter or an operator who has been at the plant for a long time. Identify these people and ensure you establish a relationship with them.

After you feel you have a good understanding of the manufacturing process at a specific plant, prepare a detailed trial plan. Set the goals of the trial and manage expectations based on your analysis of the commercial process. If there is a particular aspect of the process that you are concerned with, consider running a series of smaller trials or setting your goals such that successful demonstration of that aspect of the process is a gate to continuing the trial. Get as much input on your trial plan as you can from both sides, especially the customer. Plant managers appreciate over communication and caution when putting a new chemical into the plant.

One of the most important, but often overlooked aspects of a trial plan is defining when to stop the trial. It is easy to envision a successful trial and all the accolades and sales dollars that will come in thereafter, but it is imperative to prepare for failures as well. Use the knowledge from walking the line and observing the process to identify potential issues and then include specific actions for how to deal with them. Establish a clear line of command and authority before, during and after the trial. Assign specific responsibilities, points of contact and timelines for follow up so that there is no confusion about who is responsible for managing the trial from both sides.

3.3.2 TRIAL

The day of the trial, hold a meeting with all the stake-holders to review the trial plan and discuss any last minute changes or potential issues. Review the operational condition of the equipment that day and the how the plant has been operating in the recent days. Before the trial begins, ask for a quality sample to be taken as a control for the trial. Once the trial begins, expect the unexpected; things go wrong in manufacturing more often than one would like. Adhere to the trial plan. It is tempting to make small changes on the fly, but sometimes a series of small changes can get you so far off track, it is difficult to recover. Trust that the analysis, thought and work put into the trial plan is more valuable than a decision made at the moment under stress.

Walk the production line while running the new product and make note of any changes in the process with the new adhesive system compared to what was observed previously. Talk to the operators; these are the people who run this process day in and day out. They will notice things you won’t and they likely won’t tell you if you don’t ask. Request as many samples as the customer is willing to check and take special note of when and where the samples were taken. Request extra samples for you to take back to the lab and test using your equipment and procedures. Not only will this add to your analysis of the trial, it will allow you to improve your lab process to be more like the commercial process.

Check the trial plan often. Analyse how the system is performing in comparison to your stated goals and expectations. If things are going well and you have met all your goals and expectations, ask if it is possible to run out the rest of the material to avoid disposal costs. If the trial is not running well and you have run through all the changes proposed in the trial plan, get final recommendations from the stake-holders and then stop the trial. Retreat and plan to return another day will buy you more credibility and greatly increase the likelihood of returning for a second trial than exhausting the patience of your customer or the mill personnel trying to deal with unanticipated issues.

3.3.3 POST TRIAL

At the end of the trial reconvene the stake-holders to review the trial plan and goals, and then determine what could be done better or different in the next trial. Ask for a second control sample to be tested after the plant returns to normal operations as a bookend for how manufacturing was running for the day. Although the plants try to run consistently, there are so many uncontrolled variables that you need to compare your data with normal production on that day. Prepare a formal trial report detailing the trial and providing analysis where appropriate. Concentrate on the main issues to avoid them being buried by less important information. Use the knowledge gained during the trial to compare to lab data and revise procedures when appropriate for subsequent trails. A plant manager is not going to risk a major change in operations without repeated demonstration of operability.

4 COMMERCIALIZATION

Even running a highly successful trial is no guarantee that your product will be accepted. At this stage everything gets more scrutiny from the cost of using the new adhesive (chemical and manufacturing cost) to how the new product (plywood, particleboard, etc.) will be presented to the customer. Typically one of the first steps after a successful trial is a second trial with extended run time to better evaluate the operational stability of the new system. During this phase, expect that almost any problem that occurs will be linked to the new adhesive system whether it is realistic or not. While evaluating a new adhesive system, plant personnel tend to have a heightened sense of awareness, so that a small issue that would typically go unnoticed is observed even though it may have always been there. Take note of all criticisms that come up and talk to the operators to determine if it is real or perceived. This idea extends to downstream users as well. If the product is shipped to an end user and the end user knows there has been a change, then any difference, defect or difficulty observed will be linked back the new adhesive as well. Push for blind market trials if possible wherein the end user does not know there has been a change. As long as the product meets the manufacturers' quality tests, there may be no reason to alert the end user to any change in the product. If an issue comes up with an end user, it will get back to the manufacturer regardless of whether the end user knows there was a change or not. Change management should be part of any commercialization strategy and will require the buy-in of all parties to manage.

4.1 ADHESIVE MANUFACTURING

Commercial production of adhesive requires detailed knowledge on how the process will be controlled to make a consistent product and what the detailed product specifications are. Understand your raw materials and the adhesive production equipment and operations to minimize any variability that will influence the consistency of your product's performance. It would be nice if the wood bonding plant has the same wood supply of the same moisture content, and the temperature and humidity of the plant were constant, but they are not. Thus, adhesives and processes need to be adjusted for these changes. Understanding your operational window will be a crucial aspect of successful commercialization.

4.2 TECHNICAL SUPPORT

Because there is variation between plants in the equipment, how the processes are run, wood properties, environmental conditions, and products made, technical support should be anticipated. The level of technical support required will vary from customer to customer and even from plant to plant within a single customer. The amount of resources that will have to be committed to technical support will depend upon the individual customer or plant culture, personnel, and expectations. This is an example of the type of intangible cost that affects the economics of a new adhesive, but is usually not considered when the economic viability of a new adhesive is compared to an existing adhesive where these costs are already built in.

Because manufacturing is driven by getting the product out the door, there is a strong desire to live with the current product over a small improvement in economics with a new adhesive. The new adhesive may look better, but the unknown problems that may occur often prevent change. This is another place where the hard work of building trust with your commercial partner, including the plant manager and operations staff, will pay off.

4.3 SALES AND MARKETING

Sales and marketing are another cost for a new product that are often not considered, but they are a necessary and a real cost to your company. In addition, the cost of running trails and modifying equipment are costs to your customer. Thus, consider what benefits your new adhesive provides that overcome this barrier.

4.4 OTHER

Who is the **champion** of a new technology? Without support of someone in upper management in your company and your customer to overcome the many barriers, a new product will die along the way. Barriers can be hurdled, side-stepped, and barrelled down, but not without significant effort and cooperation of the entire team and the champion is needed to encourage and direct the program.

5 CONCLUSIONS

Inventions can be taken from the lab to commercial development, but there are many hurdles along the way. An appropriate analogy is the onion in that peeling away each layer makes your cry anew, but this does not stop people from eating onions. Innovation requires risk and risk requires management. When properly managed, innovation can offer disruptive changes that alter the landscape of products and profit margins. Understanding what needs to happen at each step and anticipating the next step is crucial to move a new product from proof of concept to commercial acceptance.

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