

# Formaldehyde Free Wood-Based Composites Produced Through a Reactive Extrusion Process

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# Issues Concerning Wood Composites

- ❑ Wood composite plants emit hazardous air pollutants (HAPs) to the environment.
- ❑ Formaldehyde is released over time from glued products.
- ❑ Public concerns about these issues cause new regulations to be proposed.

# Recent Regulations

- US EPA enacted new rules limiting emissions of HAPs from composite wood manufacturing facilities.
- This is likely to spur new research on alternative adhesives.

# Alternatives to Synthetic Adhesives

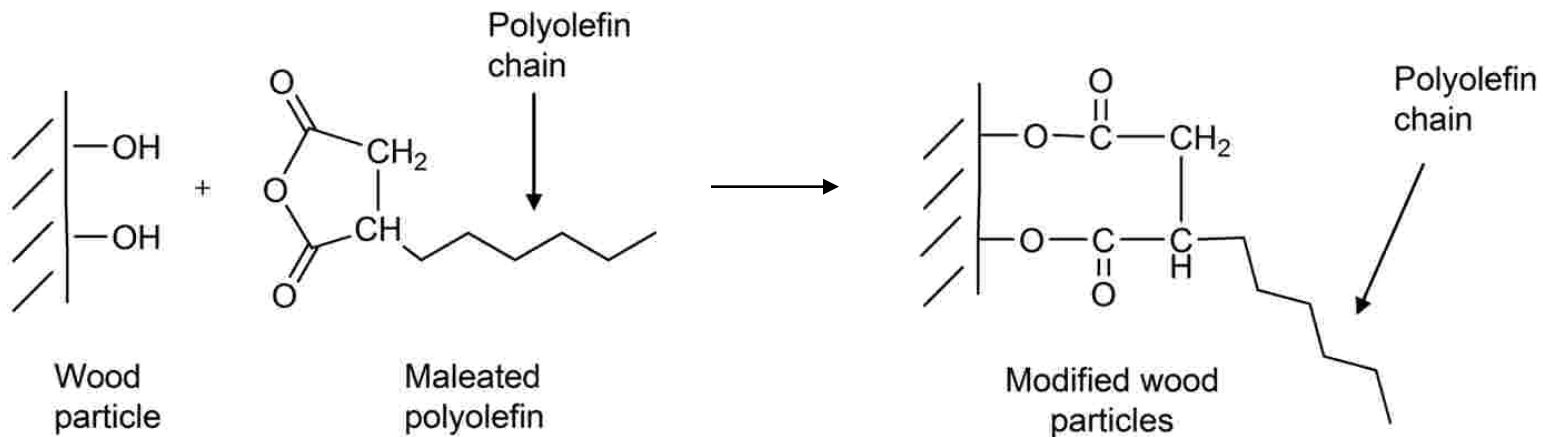
- Environmentally friendly wood adhesives
  - Low formaldehyde-to-urea molar ratio adhesives
  - Adhesives based on natural products
  - Additives to reduce formaldehyde release
  
- Binderless fiberboard
  
- Some alternatives have not been commercialized because of cost or property drawbacks.

# Wood Modification

- ❑ Modification of wood particles with anhydride compounds for wood-plastic or composite applications has been demonstrated.
- ❑ Most prior approaches to modify wood particles used solvent processes.
- ❑ Solvents such as DMF, xylene, etc. are harmful to workers and environment.
- ❑ Solvent processes are inefficient, costly and not industrially viable.

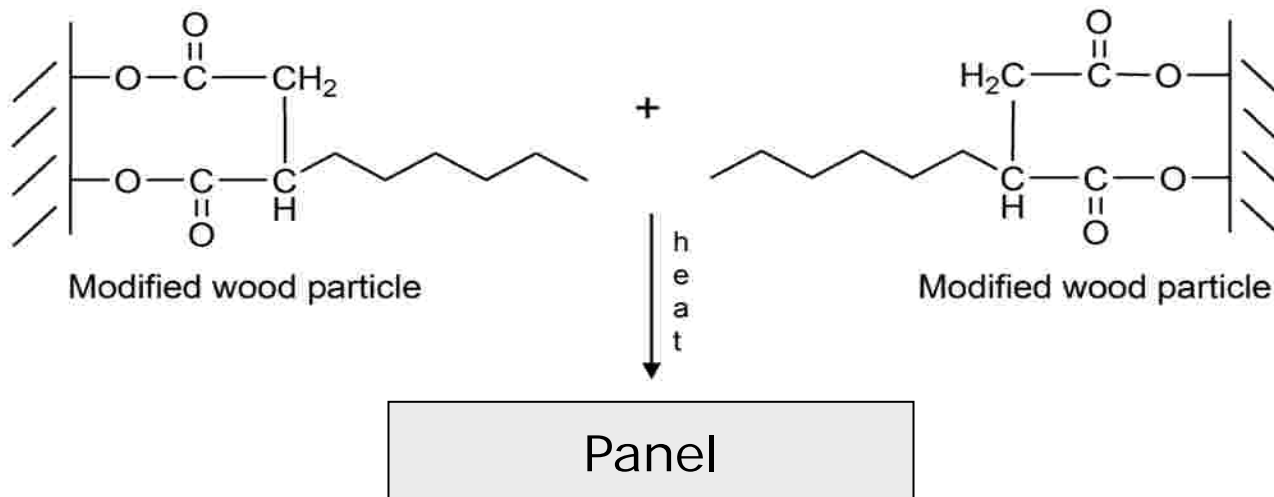
# Hypothesis

- Maleated polyolefins will react with wood particles in a solvent-free reactive extrusion process.



# Hypothesis (cont'd)

- Modified wood particles will bond together to form formaldehyde-free panels with no added adhesive.



# Objective

- Use a reactive extrusion process to produce a formaldehyde-free binding system for wood composite products.



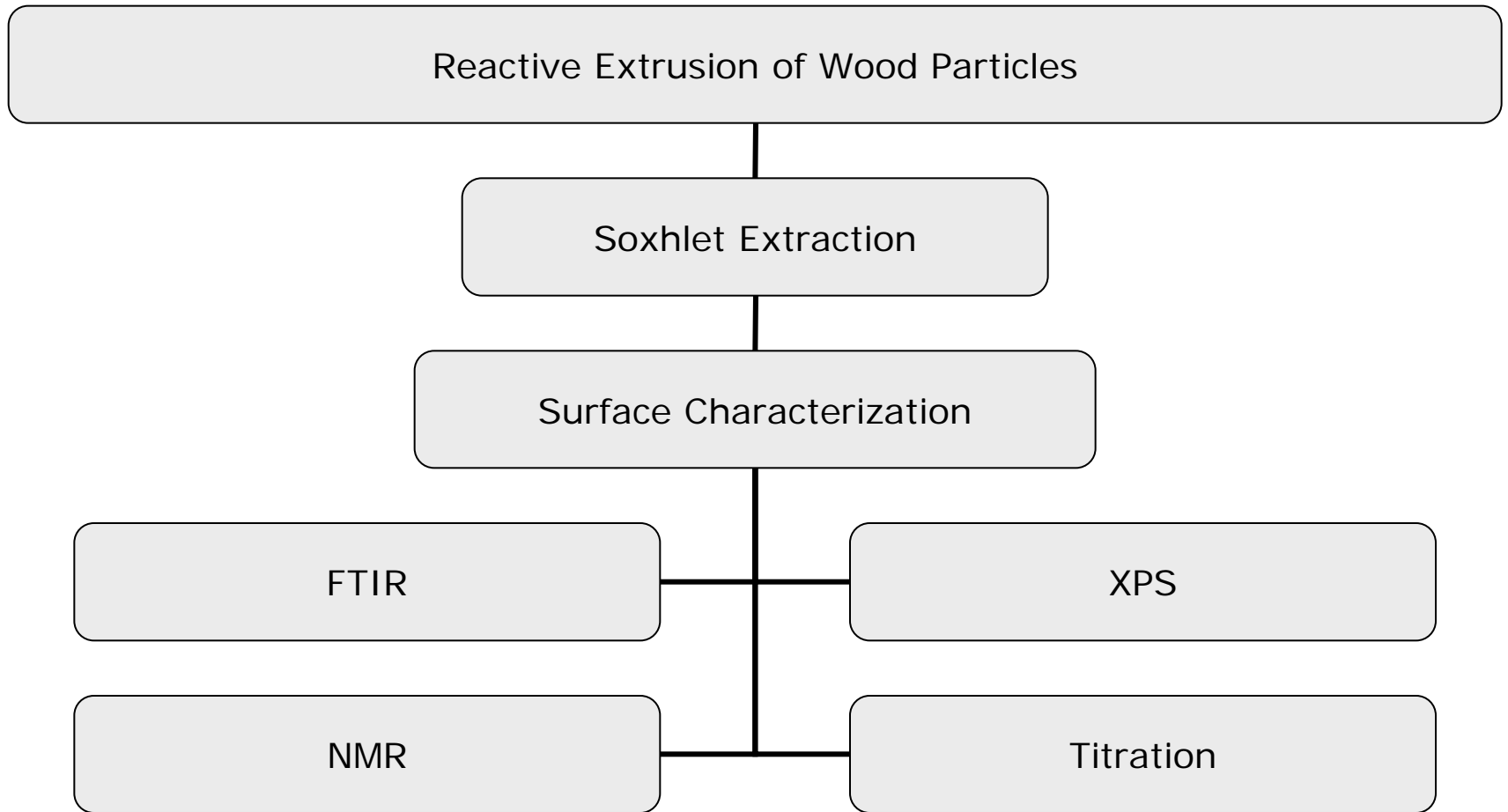
# Requirements to Achieve Objective

- ❑ Modify wood particles in reactive extrusion;
- ❑ Characterize surface composition via spectroscopic and titration techniques;
- ❑ Manufacture wood composite panels and evaluate properties;
- ❑ Comparison with standards.

# Part 1

Modification of wood particles in a reactive extrusion process and surface characterization

# Research Scheme - Part 1



# Material Composition



<b>Ingredients</b>	<b>% Total in composite</b>
Maple particles <sup>1</sup>	94 - 79
Maleated PE, PP	5 - 20
Hydrated zinc acetate	1%

<sup>1</sup>maple of 125 micron size

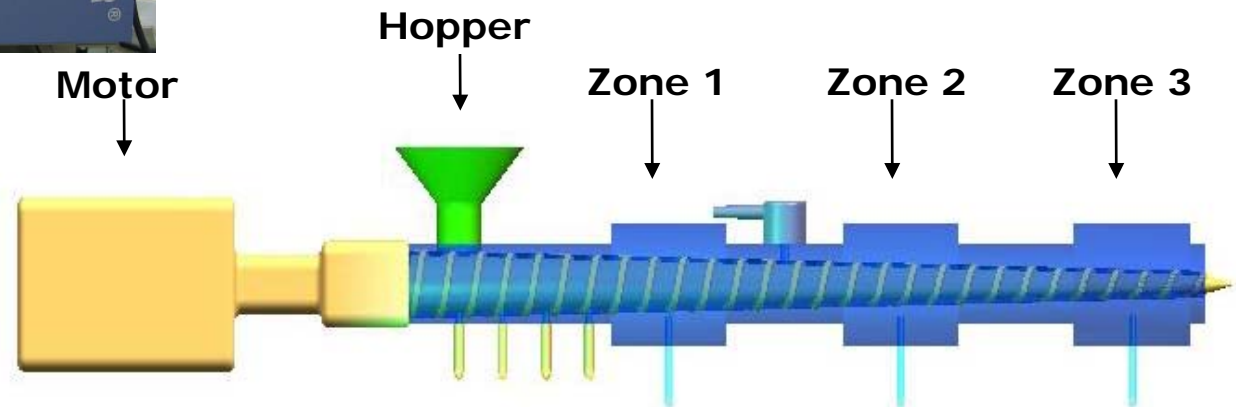
# Extrusion Processing Conditions



conical counter  
rotating twin  
screw extruder

- ❑ Rotational screw speed: 60 rpm
- ❑ Extruder barrel temperature: 160°C

## Extruder Diagram

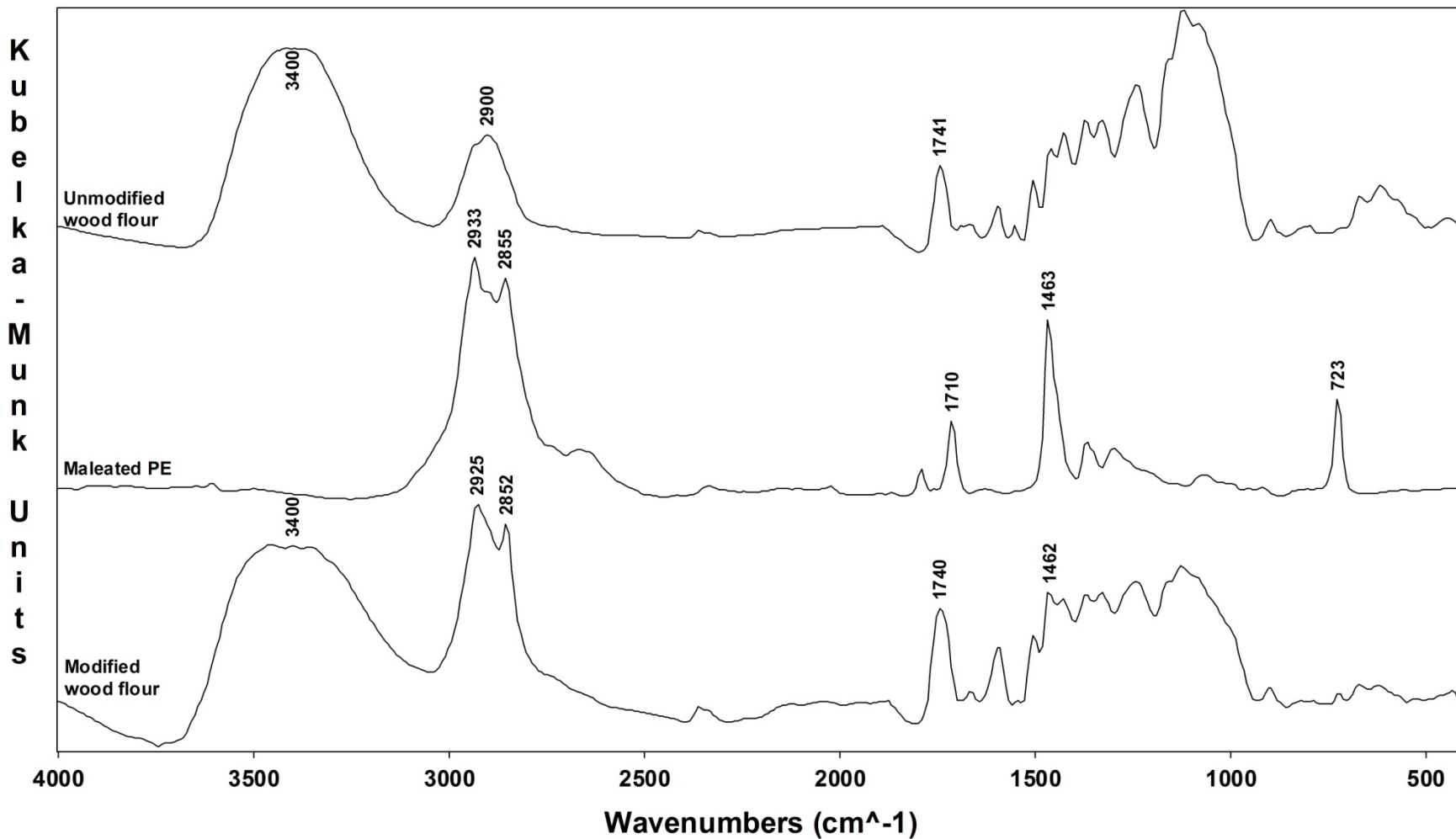


All zones maintained at a constant temperature for each test.

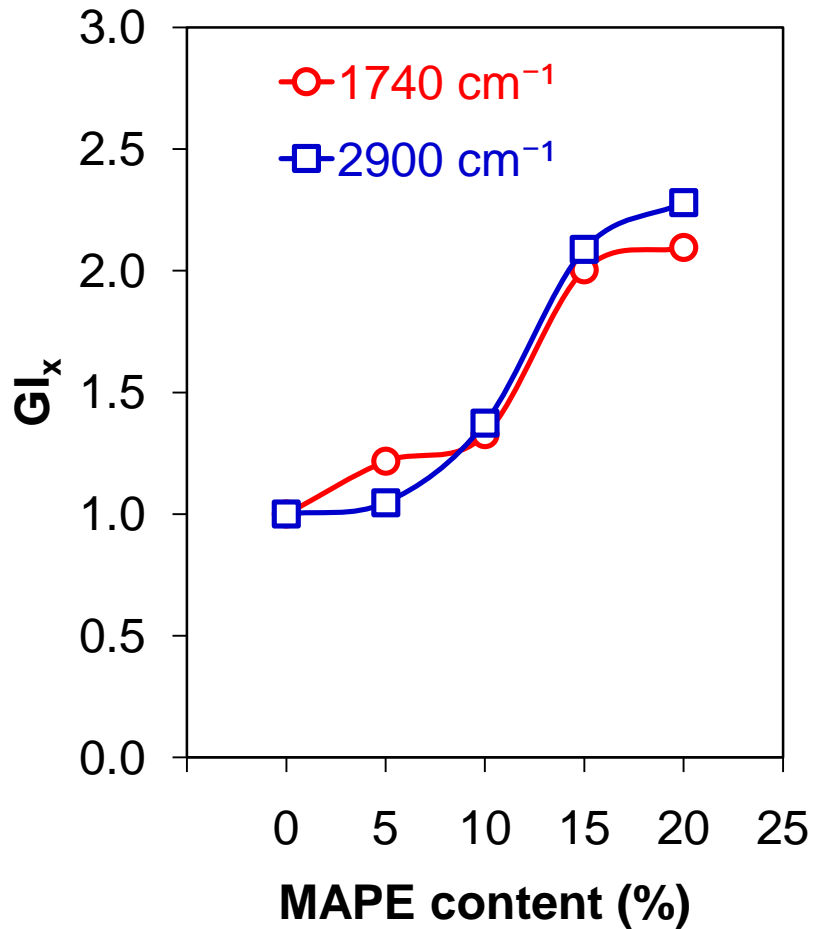
# Surface Characterization of Modified Wood Particles

- Modified wood particles were Soxhlet extracted with xylene to remove unreacted coupling agent;
- Particles were oven dried and analyzed by FTIR and XPS.

# FTIR Results



# Grafting Index ( $GI_x$ )



$$GI_x = \frac{A_{X (Modified)}}{A_{X (Unmodified)}}$$

$A_x$  : integrated area of the peak

$X$  : peak at 1740  $cm^{-1}$  or 2900  $cm^{-1}$

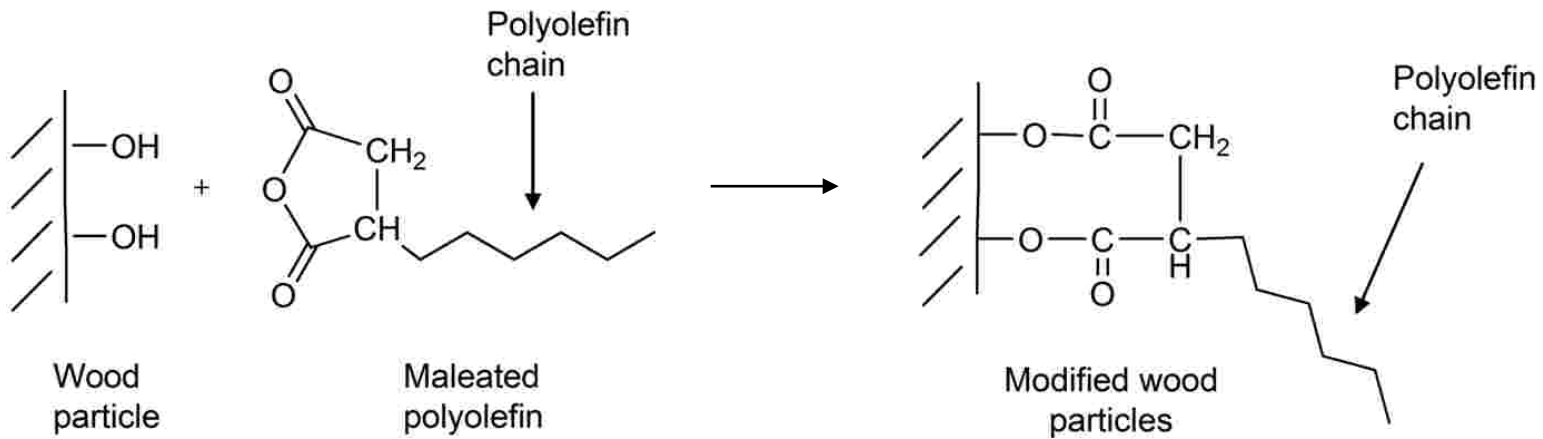


# XPS and Hydroxyl Index (HI)

Materials	O/C atomic ratios	Analysis of C <sub>1s</sub> peaks (%)				OH index (HI)
		C1	C2	C3	C4	
Unmodified wood particles	0.47	39.75	51.20	8.46	0.58	1.00
Pure MAPE	0.11	87.13	7.71	5.16	0.00	0.15
Wood modified with 15% MAPE	<b>0.07</b>	92.80	<b>5.29</b>	1.91	0.00	<b>0.10</b>
Wood modified with 20% MAPE	<b>0.03</b>	95.08	<b>4.34</b>	0.58	0.00	<b>0.08</b>

$$HI = \frac{C2_{(modified)}}{C2_{(unmodified)}}$$

# Modification Scheme



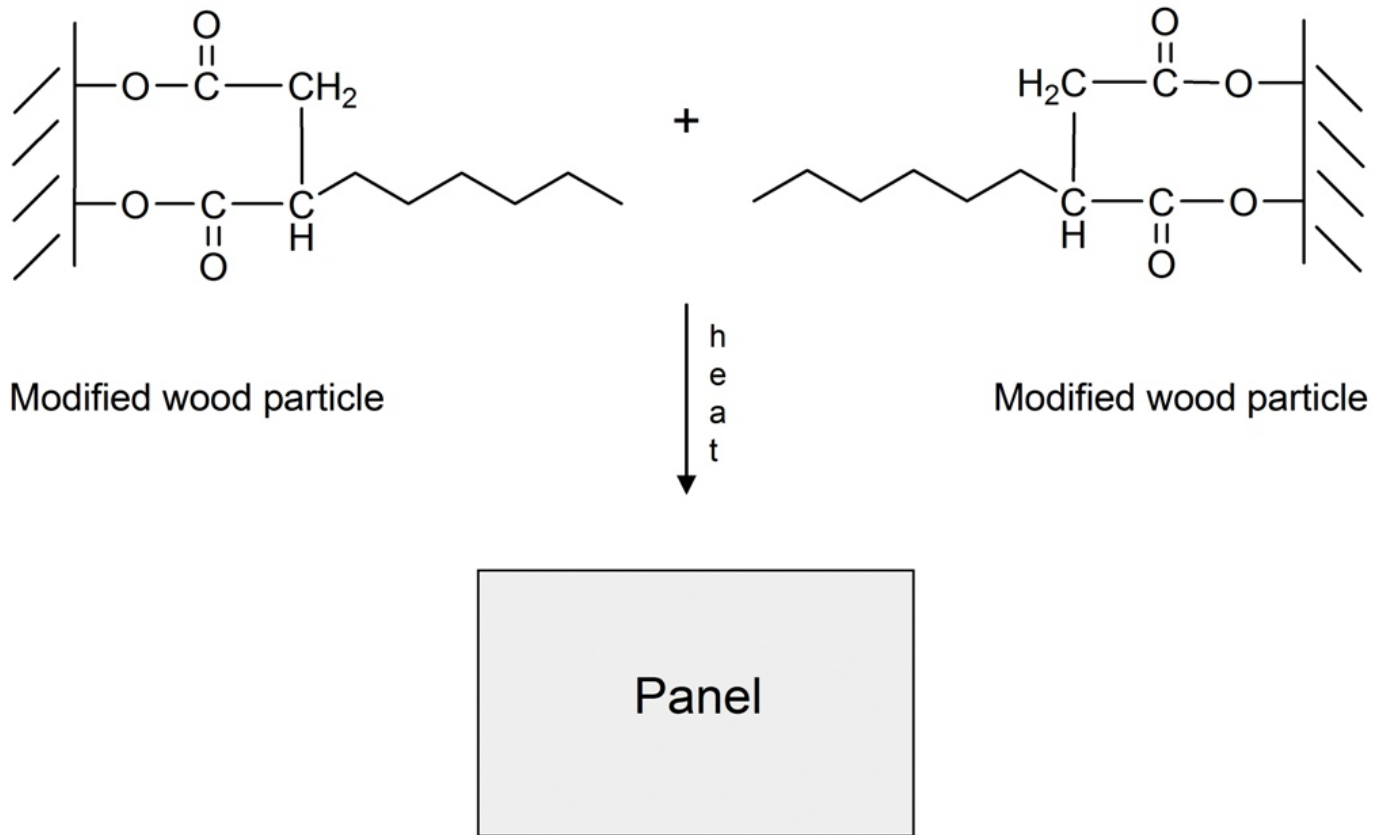
# Part 1 - Conclusions

- ❑ Maleated polyolefins were grafted to maple particles in a reactive extrusion process;
- ❑ Surface characterization data verified the reaction between the wood particles and maleated polyolefins.

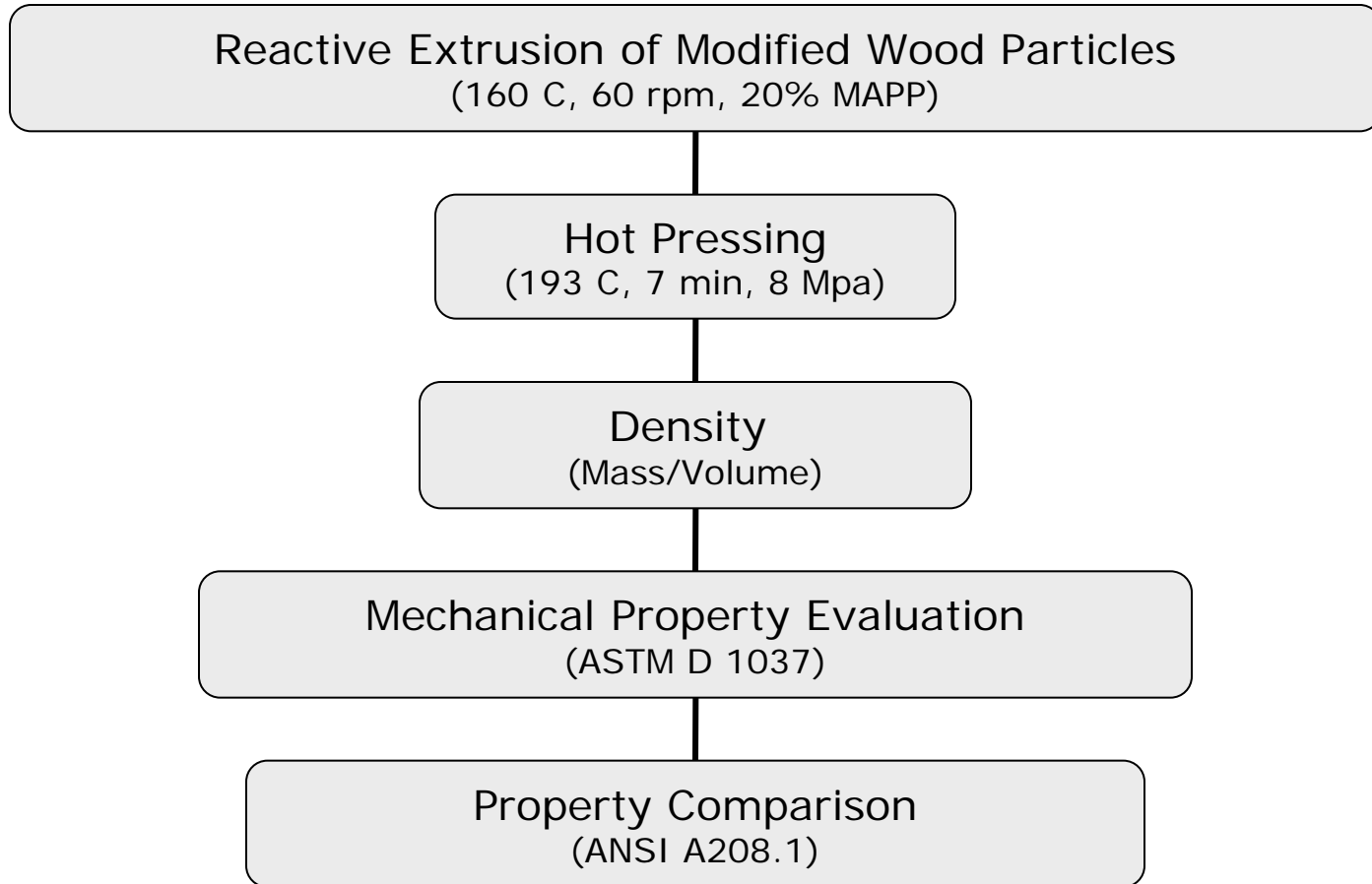
## Part 2

Panel manufacture and property  
evaluation

# Particle-Particle Bonding Scheme



# Research Scheme - Part 2



# Materials

- ❑ Wood (American Wood Fibers)
  - Hardwood maple, 425 microns (40 mesh size)
  
- ❑ Maleated polyolefins (Eastman Chemical Co.)
  - PP-based (MAPP or G-3003 and G-3015)
  
- ❑ Zinc acetate catalyst (Baker Analytical Reagents)
  
  
- ❑ Batch makeup
  - 79:20:1 weight ratio wood:binding agent:catalyst

# Panel Manufacture



Dry blending at room temperature for 10 min.



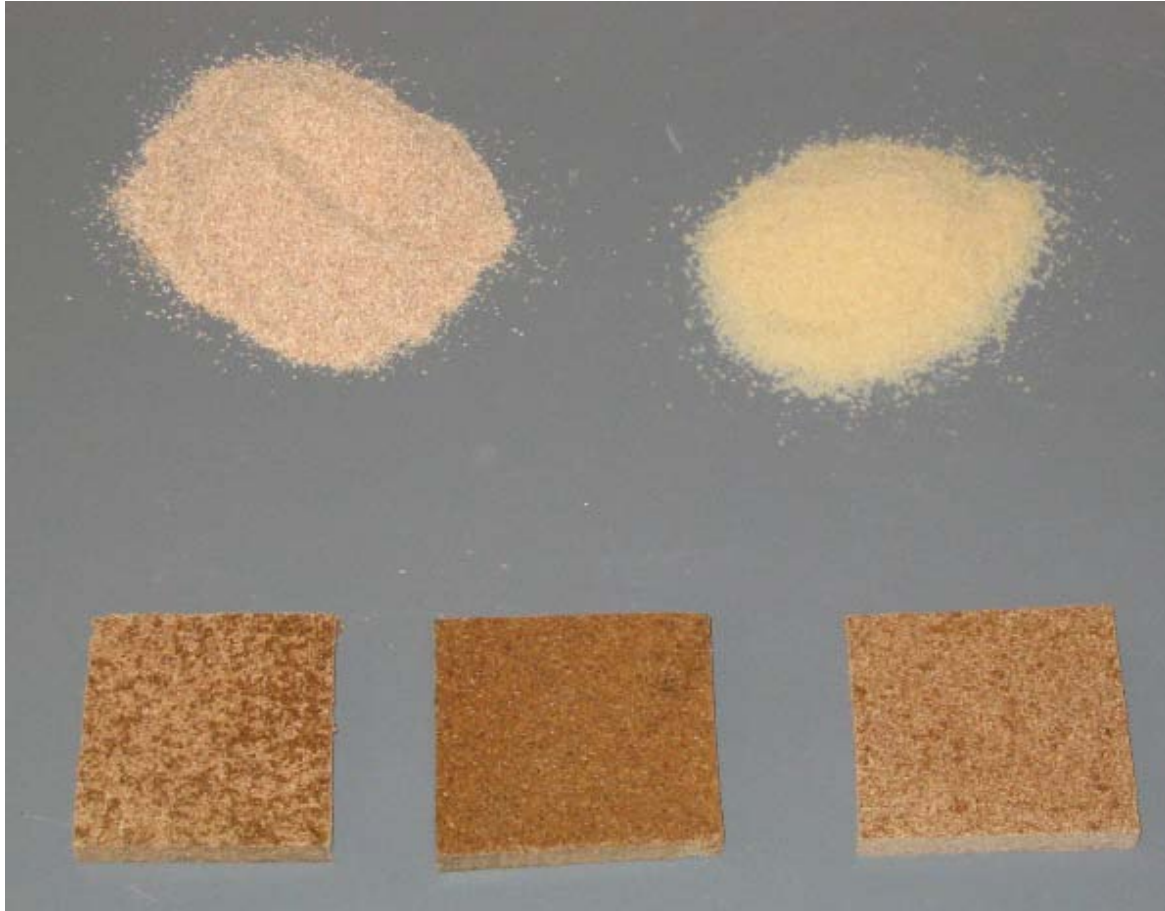
Reactive extrusion  
(160°C, 60 rpm)



Hot press  
(193°C, 7 min, 8 Mpa)



# Raw Materials and Panel Samples



# Property Evaluation

- Density
- Mechanical property tests ASTM D1037.
  - MOR, MOE
  - Internal bond (IB) strength
  - Screw holding capacity
- Property values compared with standard ANSI A208.1 (Particleboard).
- A two-sample t-test with  $\alpha = 0.05$  level of significance.

# Bending Properties

Particleboard of medium density (640-800 kg/m <sup>3</sup> )	ANSI Grades				Experimental Values	Average Density (kg/m <sup>3</sup> )
	M-1	M-S	M-2	M-3		
MOR (MPa)	11.0	12.5	14.5	16.5	23.00 ± 4.7	775 ± 8
MOE (MPa)	1725	1900	2250	2750	2875 ± 347	

From Standard ANSI A208.1-1999 Particleboard

# IB Strength and Screw Holding Capacity

Panel Types	Properties	
	IB Strength(MPa)	Screw holding (N)
Medium density grades (Standard ANSI A208.1)	0.40 – 0.55	900 - 1100
MAPP-panel	1.50	1580

# Conclusions

- Panels were formed from modified wood particles produced through reactive extrusion;
- Composites contained no formaldehyde-based adhesives;
- Mechanical properties meet or exceed standard requirements.

# Acknowledgements

- USDA-CSREES Grant – Advanced Technology Applications to Eastern Hardwood Utilization;
- McIntire-Stennis Cooperative Forestry Research Program;
- Dr. Karana Carlborn.

# References

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2. K. Carlborn and L.M. Matuana, "Modeling and Optimization of Formaldehyde-Free Wood-Based Composites Using a Box-Behnken Design," *Polymer Composites*, **27** (5): 497-503 (2006).
3. K. Carlborn and L.M. Matuana, "Influence of Processing Conditions and Material Compositions on the Performance of Formaldehyde-Free Wood-Based Composites," *Polymer Composites*, **27** (6): 599-607 (2006).
4. K. Carlborn and L.M. Matuana, "Functionalization of Wood Particles Through a Reactive Extrusion Process," *Journal of Applied Polymer Science*, **101** (5): 3131-3142 (2006).
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