

## Optimization of Resinification of Liquefied Products from Trash Antiseptic Wood

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### Abstract

In this study, Anticorrosive fir was liquefied in phenol, and the liquefied wood were used to produce adhesive by resinification. The effect of resinification time, resinification temperature, molar ratio (NaOH/liquefied wood) and molar ratio (methanal/liquefied wood) on resinification was investigated by orthogonal experiments in term of the viscosity, solid content and pH of resinification product. Adhesive produced by resinification was then used for plywood production, and plywood strength was detected to assess adhesive. According to experiment results, the optimal conditions of resinification are as follow: resinification time of 150 min, resinification temperature of 90°C, molar ratio (NaOH/liquefied wood) of 0.5 and molar ratio (methanal/liquefied wood) of 1.8. Using adhesive produced under the optimal conditions to bond plywood, the bonding strength and formaldehyde emission of plywood produced accorded with related national standard (China), and the plywood produced belonged to class I.

**Key words:** Antiseptic woods; Resinification; Liquefaction; Phenol; Adhesive

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### INTRODUCTION

Researchers have become more and more interesting in Wood Liquefaction, because it is one of main ways to use biomass to provide chemicals for society. Liquefied products from biomass have a very extensive applied foreground, because they could be used to produce adhesive, molding compounds, foam materials and carbon fiber. Wood preservation could extend the service life of wood, and it is also an effective measure of using forest resources savingly. However trash antiseptic woods will lead environment pollution because of the toxicity of most wood preservatives. Therefore treatment ways of trash antiseptic woods should be developed to lower the negative effects of trash antiseptic woods on environment.

Liquefaction is a potential way of reusing trash antiseptic woods, and avoiding the negative effects of trash antiseptic woods on environment. Besides liquefied product could also be converted to valuable products by resinification, such as adhesive. In the present study, Anticorrosive fir was liquefied in phenol, and then liquefied product was used to produce adhesive by resinification. Optimization of four resinification process parameters (resinification time, resinification temperature, NaOH/liquefied wood molar ratio and methanal/liquefied wood molar ratio) was carried out by using orthogonal experiment design. Adhesive produced under the optimal conditions was then used to bond plywood, and the bonding strength and formaldehyde emission of plywood produced were detected to decide the quality of the adhesive.

### MATERIAL AND METHODS

#### Raw Material

Trash CCA antiseptic fir was smashed and sieved, and the particle between 40-80 mesh was dried at 103°C for 12 h before being used. Phenol, vitriol (99%), acetone

and formaldehyde (37%) used were of analytical grade. Besides 36% vitriol and 40% NaOH (w/v) were also used.

**Liquefaction and Resinification**

Liquefaction was carried out in 4% vitriol with a solid-to liquid ratio (wood flour/phenol) of 1:5. Liquefaction liquid was cooled and filtrated at the end of liquefaction, and then filter liquor was kept in glass container for resinification.

A certain volume NaOH solutions (40%) and water were added into certain volume liquefaction liquid in a reactor, respectively. Reactor was then kept at 40-45°C in water bath, and formaldehyde (37%) was added into reactor slowly. The temperature was increased to 60±2°C. After maintained temperature at 60±2 °C for 20 min, the temperature was increased to set point until the end of reaction. Reaction liquid was cooled to 30°C for further analysis.

**Orthogonal Design Methodology Experiment**

As showed in table 1, four resinification factors, including resinification time, resinification temperature, molar ratio (NaOH/liquefied wood) and molar ratio (methanal/liquefied wood), were selected to be optimized by using the L16 (45) orthogonal table in term of the viscosity, solid content and pH of resinification product. Extra experiments were also carried out to test the result of orthogonal design methodology experiment.

**RESULTS AND DISCUSSION**

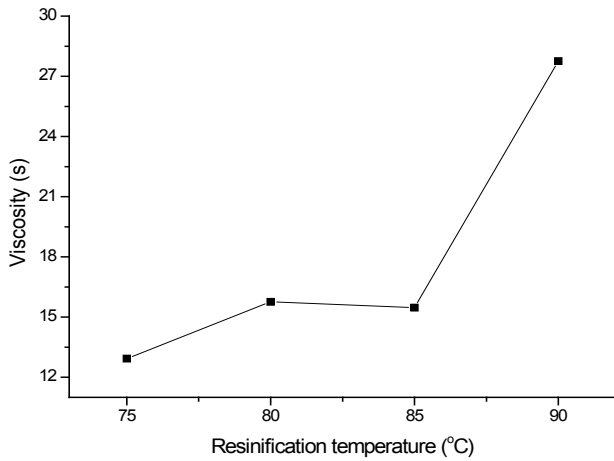
**Results of Orthogonal Design Methodology Experiment**

Table 1 showed the results of orthogonal design methodology experiment. All of resinification factors investigated have effect on the viscosity and solid content. Only molar ratio (formaldehyde/liquefied wood) and molar ratio (NaOH/liquefied wood) have effect on the pH of resinification product. For the viscosity, the sequence of factors is resinification temperature, molar ratio (NaOH/liquefied wood), molar ratio (formaldehyde/liquefied wood) and resinification time. For the solid content, the sequence of factors is molar ratio (NaOH/liquefied wood), molar ratio (formaldehyde/liquefied wood), resinification temperature and resinification time. For pH, the sequence of factors is molar ratio (formaldehyde/liquefied wood) and molar ratio (NaOH/liquefied wood). Though different factors affected the solid content and pH to different extent, the solid content and pH of resinification product accorded with the national standard (china) related whatever conditions were used. Therefore the effect of resinification factors on the viscosity of resinification product will be emphatically discussed.

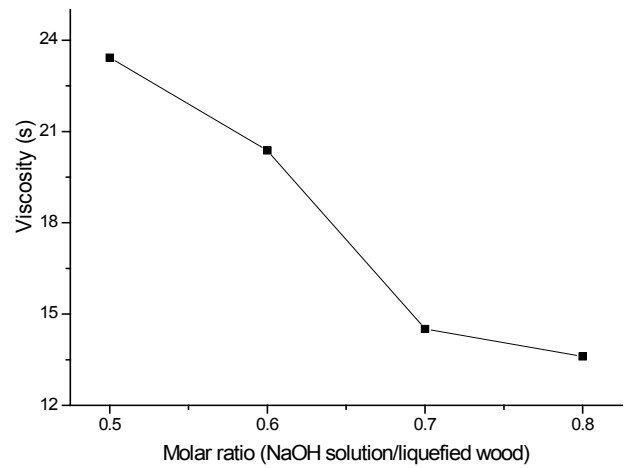
**Effect of Resinification Factors on the Viscosity of Resinification Product**

**Table 1**  
**The Results of Orthogonal Design Methodology Experiment**

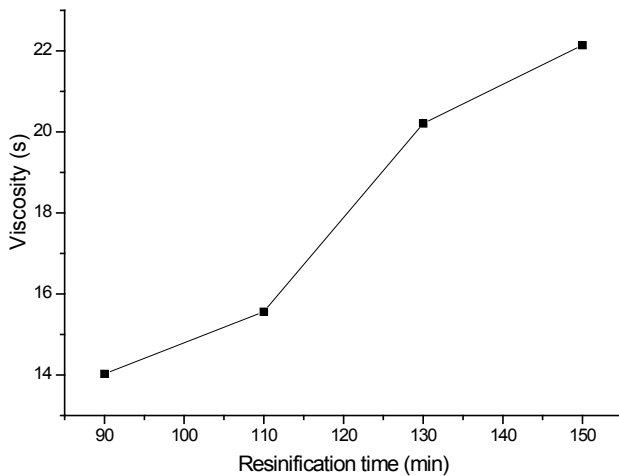
factors	level	The viscosity (s)		solid content (%)		pH	
		Average	Range	Average	Range	Average	Range
temperature (°C)	75	12.93	14.83	46.85	6.75	12.13	0.25
	80	15.76		44.70		12.13	
	85	15.47		39.88		11.88	
	90	27.76		40.10		12.13	
time (min)	90	14.03	8.11	43.48	5.55	12.00	0.13
	110	15.56		40.38		12.13	
	130	20.21		45.93		12.00	
	150	22.14		41.75		12.13	
molar ratio (NaOH/liquefied wood)	0.50	23.43	9.82	23.43	21.05	12.00	0.38
	0.60	20.38		44.48		11.88	
	0.70	14.51		43.05		12.25	
	0.80	13.61		38.65		12.13	
molar ratio (formaldehyde/liquefied wood)	1.20	13.27	9.19	37.68	11.18	12.13	0.50
	1.50	13.97		41.55		12.25	
	1.80	22.22		43.45		12.13	
	2.10	22.46		48.85		11.75	
empty case	1	20.58	7.29	42.18	5.16	12.00	0.38
	2	21.64		45.98		11.88	
	3	15.37		42.25		12.25	
	4	14.35		40.83		12.13	



**Figure 1**  
**The Effects of Resinification Temperature on The Viscosity of Resinification Product**

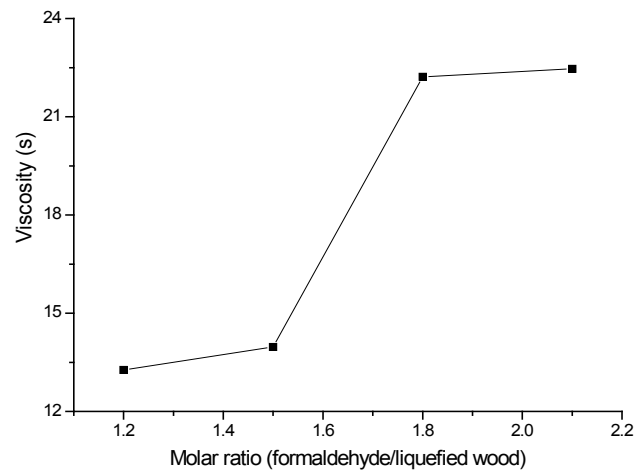


**Figure 3**  
**The Effects of Molar Ratio (NaOH/liquefied wood) on the Viscosity of Resinification Product**



**Figure 2**  
**The Effects of Resinification Time on the Viscosity of Resinification Product**

Figure 1 showed the effect of resinification temperature on the viscosity. The viscosity increased slightly when resinification temperature was increased from 75°C to 80°C, and then increasing temperature from 80°C to 85°C made the viscosity a slight decrease of 0.29s. When temperature was higher than 85°C, increasing temperature obviously improved the viscosity. The probable reason for the results is that the reactivity of phenolic compounds with formaldehyde increased with resinification temperature, which increased the viscosity of resinification liquid. Therefore the optimized temperature is 90 °C. As shown in Figure 2, the viscosity increased slightly with increasing resinification time. The probable reason for this is that increasing reaction time leads a much complete reaction. When using long time, the viscosity of resinification product was apt to accord with related national standard (China). Therefore the optimized time is 150 min.



**Figure 4**  
**The Effects of Molar Ratio (Formaldehyde/Liquefied Wood) on the Viscosity of Resinification Product**

Figure 3 showed the effect of molar ratio (NaOH/liquefied wood) on the viscosity. The viscosity decreased with increasing molar ratio (NaOH/liquefied wood). Increasing the amount of NaOH solution increased the dissolubility of resin, which could decrease the viscosity of resinification product. The viscosity decreased by 3.05s when the molar ratio increased from 0.5 to 0.6. A decrease of 5.87s was obtained when the molar ratio increased from 0.6 to 0.7. When the molar ratio was higher than 0.7, the viscosity decreased slowly with increasing molar ratio. Taking both of process cost and product quality into account, the optimized molar ratio is 0.5.

The effect of molar ratio (formaldehyde/liquefied wood) on viscosity was illustrated in Figure 4., The viscosity increased with increasing molar ratio (formaldehyde/liquefied wood). The viscosity increased obviously when molar ratio was increased from 1.5 to 1.8. When molar ratio (formaldehyde/liquefied wood) was

higher than 1.8, increasing molar ratio (formaldehyde/liquefied wood) improved the viscosity slightly, but increased residual formaldehyde obviously. Therefore a molar ratio of 1.8 was chose as optimized molar ratio (formaldehyde/liquefied wood).

### Result of Verification Experiment

According to the result of Orthogonal design methodology experiment, the optimal conditions for resinification are as follow: a resinification time of 150 min, a resinification temperature of 90 °C, a molar ratio (NaOH/liquefied wood) of 0.5 and molar ratio (methanal/liquefied wood) of 1.8. An experiment under the optimal conditions were carried out to assess the optimal conditions. Using the optimal resinification conditions, adhesive produced by resinification obtained a viscosity of 74.7 s, solid content of 45.0% and a pH of 11, which accorded with related national standard (china) for adhesive. Adhesive produced under the optimal conditions was also used to produce three layer poplar plywood under the conditions: hot pressing temperature (150°C), hot pressing time (6 min) and glue spread (260g/m<sup>2</sup>). The bonding strength and formaldehyde emission of plywood produced were 1.275 MP and 0.1 mg/L, respectively, which indicated that the plywood produced belonged to calss I.

### CONCLUSIONS

According to results of orthogonal design methodology experiment, the primary and secondary relation of the four variables for the viscosity is: resinification temperature > molar ratio (NaOH/liquefied wood) > molar ratio (formaldehyde/liquefied wood) > resinification time. The primary and secondary relation of the four variables for the solid content is: molar ratio (NaOH/liquefied wood) > molar ratio (formaldehyde/liquefied wood) > resinification temperature > resinification time. The sequence of curing factors for pH is molar ratio (formaldehyde/liquefied wood) and molar ratio (NaOH/liquefied wood). Resinification temperature and time have no effect on the pH of resinification product.

According to verification experiment, the optimal conditions for resinification are as follow: resinification time of 150 min, resinification temperature of 90°C, molar ratio (NaOH/liquefied wood) of 0.5 and molar ratio (methanal/liquefied wood) of 1.8. Using adhesive produced under the optimal conditions to bond plywood, the bonding strength and formaldehyde emission of plywood produced accorded with the national standard (china) for calss I, and formaldehyde emission was much lower than that required in the national standard GB/T 9486-2044 for E1 plywood.

### ACKNOWLEDGEMENTS

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