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Physical Properties of Hybrid Poplar Flakeboard Bonded with Alkaline Phenolic Soy Adhesives[*1]

In Yang[*2†], Monlin Kuo[*3], and Deland J. Myers[*4]

ABSTRACT

Soybean-based adhesives have recently been reconsidered as alternatives to petroleum-based adhesives due to the uncertainty of availability of petrochemical products and the increased demand for wood adhesives. This study was conducted to investigate the adhesive properties of alkaline phenolic soy (APS) resin for hybrid poplar flakeboard. The APS resin was formulated by crosslinking an alkaline soy flour hydrolyzate with lab-prepared PF resin in the soy hydrolyzate to PF resin weight ratios of 70/30, 60/40, and 50/50. The APS resins were used to fabricate homogeneous hybrid poplar flakeboards with different resin solid levels (5%, 7%, and 9%), press temperatures (175 and 200°C), and press times of 8 and 10 minutes. The IB, wet MOR, and dimensional stability properties of board improved with increasing press time, press temperature, and PF level in APS resins. Increasing press time can be used to offset poor IB strength associated with a 9% resin solid level and the excessive moisture content in the mat. The following conditions were concluded to meet the requirements of the CSA standard for exterior-grade flakeboard: a 50% PF level, a 5% resin content, a 200°C press temperature, and an 8 minute press time.

Keywords : soy flour hydrolyzate, phenol-formaldehyde (PF) resin, CSA standard, wet modulus of rupture, linear expansion, PF level.

1. INTRODUCTION

In 1998, North American oriented strand board (OSB: 3/8 inch-thick board basis) production reached 19 billion ft^2 and is expected to increase to 41 billion ft^2 by 2050 (Adams, 2000). OSB panels are mainly manufactured using phenol-formaldehyde (PF) adhesives. Wood product industries in North America consumed about 3 billion lb of phenolic resin solid in 1999 (Johnson, 2001).

While PF resin is currently in adequate supply, fluctuating crude oil prices, increased demand for wood adhesives, the uncertain future supply of petrochemicals, and demand for environmentally safe products have given an impetus to develop adhesives from renewable resources. In the search for alternative adhesives to replace conventional wood adhesives, the utilization of spent liquor lignins and tannins for wood adhe-

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[*1] Received on June 20, 2005; accepted on September 06, 2005.

[*2] Department of Forest Products, Seoul National University, San 56-1 Shillim-Dong, Seoul 151-742, Korea
[*3] Department of Natural Resource Ecology and Management, Iowa State University Ames, IA 50011-1021 U.S.A.
[*4] Department of Food Science & Human Nutrition Iowa State University Ames, IA 50011-1021 U.S.A.
[†] Corresponding author : In Yang (dahadad2000@yahoo.com)

sives has been gained increasing research interest (Kuo *et al.*, 1991; Pizzi, 1983; Sellers *et al.*, 1994). The industrial application of these substances is still a challenge to the wood industry. Because lignins have low reactivity, and thus require longer curing time or a higher curing temperature than phenolic resins (Koch *et al.*, 1987). For tannin-based adhesives, two inherent problems of the adhesive, short pot-life and poor strength of the cured glueline, limit its industrial applications (Pizzi, 1983).

Carbohydrates and proteins have been reconsidered as alternatives to petroleum-based wood adhesives, because the two substances are the most abundant renewable substances. The two substances have to be used as copolymers with synthetic resins to meet stringent performance requirements. In this aspect, proteins are more suitable than carbohydrates, because proteins possess many more functional groups than carbohydrates for the formation of cross-linkages with synthetic resins. For example, soy protein contains about 4% tyrosine, an aromatic amino acid, and the two open ortho positions of each tyrosine are available for condensation with synthetic resins (Kuo and Stokke, 2001).

Recently, Kuo *et al.* (2001) formulated neutral phenolic soy resins having a high viscosity and a short pot life. Thus, the viscous adhesive resin is more suitable for plywood than for OSB. Hydrolyzation of soy proteins is a way to reduce the viscosity of the soy-based adhesive resins resulting in a resin that is easily sprayable to wood furnishes. Kuo and Stokke (2001) developed a PF-crosslinked soy resin comprised of 70% soy hydrolyzate and 30% PF, and prepared flakeboard panels with the resin. The boards were as strong in bending strength, but lower in internal bonding strength and in water resistance than boards bonded with a commercial PF resin. A similar soy resin has been developed by Hse *et al.* (2001). These results suggested that the soybean-based phenolic adhesive resin could be improved by increasing the amount of PF ingredient, resin content, or altering pressing conditions. Therefore, the objective of this study was to investigate the effects of PF level in the soy resins, board resin content, press temperature, and press time on the physical properties of flakeboard bonded with the soybean-based phenolic adhesives.

2. MATERIALS and METHODS

2.1. Flake Preparation

Four- to six-year-old hybrid poplar (*Populus deltoides)* logs with diameters ranging from 5 to 10 inches were harvested from the Hinds Farm located near Ames, Iowa. Logs were debarked, and then reduced into flakes by means of a disk flaker with flake thickness set at 0.02 inch. The flakes were screened using a lab-made rotary concentric screener to eliminate fines, and then dried to 3 ± 1 percent moisture content in a drier. Flake target dimension was 2.8 inch length × 0.4 inch width × 0.02 inch thickness.

2.2. Adhesive Preparation

Alkaline soy hydrolyzates were prepared by reacting defatted soy flour in a sodium hydroxide solution as described Kuo and Stokke (2001). A highly methylolated phenol-formaldehyde (PF) resin was prepared in the laboratory with a formaldehyde to phenol molar ratio of 2.4 and a sodium hydroxide to phenol molar ratio of 0.1. Synthesis of the PF resin was done in an 1-L resin kettle. The phenol, 37% formaldehyde, and 50% sodium hydroxide were charged to the reactor and held at 75°C for 60 minutes. The reaction mixture was then heated to 90~95°C, and cooked for 65 minutes.

Synthesis of APS resins was done at the soy hydrolyzate to PF resin weight ratios of 70/30, 60/40, and 50/50. Before spraying, 1% lignosulfonate-based wax emulsion (ovendry weight basis of flakes), having a 50% solid content, was added into the resin formulations at the final stage to improve the water repellency properties. A control PF resin (45 557H) with a resin solid level of 50 percent and viscosity level of 200 cps was obtained from Borden Chemical Company.

2.3. Characteristics of Soy-based Resins

Viscosity, solid content, and pH tests of the APS resins were performed to provide a comparison with a commercial PF resin. The solids content of the APS resin was determined by a pan solids technique (ASTM, 1993). Viscosity of each resin was measured at room temperature with a Brookfield digital Viscometer, DV-II (Stoughton, MA) spindle Number 21 at 10-rpm rotation, after wax emulsion was mixed with APS resins. The pH of each resin was measured with a Fisher Scientific (Pittsburgh, PA) ACCUMET®, Model 1600 pH meter.

2.4. Flakeboard Fabrication and Testing

The APS resins at a rate of 5%, 7%, and 9% resin levels, based on the ovendry flake weight, were sprayed onto flakes in a rotating drum blender. The target density of the single-layer flakeboards was 40.6 lb/cubic feet (0.65 g/cm^3). The board design was 15 inch by 15 inch and 0.5 inch in thickness, using steel stops for thickness control. The required amount of resin was applied to the flakes with an air-atomization nozzle (30 psi air pressure). The blended flakes were hand-felted into a randomly oriented, homogeneous mat with a forming box on a caul plate. The mat was transferred to a hot-press and pressed with the specified conditions. The hot-press platen temperatures were 175°C or 200°C. Total press times were 8 and 10 minutes. A sixty-ton force was applied to reach a panel thickness of 0.5 inch in less than 30 seconds, and decompressed the last half period of the cycle. After conditioning at ambient conditions overnight, the density of each board was measured.

Physical properties, dry and wet modulus of rupture (MOR), modulus of elasticity (MOE), internal bond (IB), water absorption (WA) and thickness swell (TS) after 24-hour soaking and 2-hour boiling, and linear expansion (LE) after 2-hour boiling, of the flakeboard were tested in accordance with ASTM 1037-78 (1998). Two MOR and MOE specimens in dry state, six IB specimens, four TS-24 and WA-24 specimens, one wet MOR, TS-2, WA-2, and LE specimen were obtained from each panel. When wet MOR was tested, the specimen tested was still wet but cooled.

2.5. Experimental Design and Statistical Analyses

This study was designed to examine the effects of PF level in APS resins, resin content, press temperature, and press time on physical properties of flakeboard bonded with APS resins. A full-factorial experimental design (3×3×2×2) was employed. The four factors were resin content (5%, 7%, and 9%) and PF level in APS resins (30%, 40%, and 50%), press temperature (175°C and 200°C), and press time (8 and 10 minutes). A total of 222 boards were made with 6 boards for each adhesive resin (216 boards), and six control boards bonded with a commercial PF resin also were prepared.

The data were analyzed using the Statistical Analysis System programming package. The

Table 1. Properties of three alkaline phenolic soy resins and a control PF resin used to bond flakeboard

Soy hydrolyzate (%)	PF (%)	pH	Non-volatile solids (%)	Viscosity (cps)[a]
70	30	9.6	45.93	1670
60	40	10.2	46.72	1620
50	50	10.9	47.11	1570
Control		11.5	56.70	200

[a] cps: centipoise (mPa · s).

The viscosities of each resin were measured after wax was mixed with alkaline phenolic soy resins.

Table 2. Summary of statistical analyses for the effects of PF level in APS resins, resin content, press temperature, and press time on physical properties of flakeboards bonded with alkaline phenolic soy (APS) resins

	Dry MOR[a]	Wet MOR[b]	MOE[a]	**IB**[c]	WA-24[d]	TS-24[d]	WA-2[b]	TS-2[b]	LE[b]
	-------- (psi)	---------	-- (kpsi)--	-- (psi) --	------------------------		(%)		------------------------
PF level in APS resin									
30%	5434 B	492 C	759 B	77 C	29 C	97 C	67 C	125 C	0.44 C
40%	5799 A	1014 B	771 AB	97 B	16 B	57 B	50 B	95 B	0.35 B
50%	5946 A	1568 A	787 A	109 A	13 A	39 A	45 A	75 A	0.28 A
Resin content									
5%	5592 B	832 C	742 B	88 C	25 C	79 C	62 C	116 C	0.40 C
7%	5847 A	1016 B	792 A	100 A	21 B	64 B	57 B	98 B	0.34 B
9%	5739 AB	1226 A	783 A	94 B	12 A	49 A	44 A	82 A	0.33 A
Press temperature									
175°C	5762	630	754	86	25	88	63	118	0.42
200°C	5690	1419	790	103	14	40	46	79	0.30
	(p=0.44)[e]	(p=0.01)	(p=0.01)	(p=0.01)	(p=0.01)	(p=0.01)	(p=0.01)	(p=0.01)	(p=0.01)
Press time									
8 minutes	5780	843	772	83	22	72	59	106	0.35
10 minutes	5672	1207	772	106	16	56	50	91	0.36
	(p=0.25)[e]	(p=0.01)	(p=0.96)	(p=0.01)	(p=0.01)	(p=0.01)	(p=0.01)	(p=0.01)	(p=0.35)
CSA O437[f]	4200	900	800	50	15				0.35

[a, b, c, d] Each value represents the average of 12, 6, 36, and 24 specimens, respectively.

[e] The p-value indicates the probability for the effects of press temperature and press time on each property.

[f] Canadian Standard association minimum standard for exterior-grade flakeboard

Means within a column not followed by a common capital letter differ significantly at p = 0.05 (Least Significance Difference test).

General Linear Model (GLM) procedure and the analysis of variance (ANOVA) were used to determine differences among different resins and to characterize the individual APS resins. Significant effects with a $p < 0.05$ were further characterized by the Least Significance Difference (LSD) between means.

3. RESULTS and DISCUSSION

3.1. Resin Characterization

The pH and solid content of soy resins were increased by increasing the amount of PF. Viscosity was decreased with increasing PF level in APS resins. The properties of three APS resins and a commercial PF resin used to bond flakeboard are presented in Table 1. The APS resins are suitable to produce OSB panels for two days, in which the resins have been prepared, because they displayed sufficiently low viscosities (1570～1670 cps) for spraying, with high solid levels ranging from 45% to 47% and pH values from 9.6 to 10.9. After then, however, the viscosity of APS resins was gradually increased, and consequently the increased viscosity of the resin made the APS resins difficult for spraying the resins. Therefore, further studies for decreasing viscosity and increasing pot life of this APS resins are required. The solution of the two problems should be approached with various hydrolyzing conditions, such as hydrolyzing reagents, time and temperature, of soy flour. It might make the resin possible for its industrial application.

3.2. Panel Properties

Table 2 summarizes the results of statistical analyses of the effects of PF level in APS resins, resin content, press temperature, and press time on the physical properties of the flakeboards.

3.2.1. Effect of Resin Composition

PF level in APS resins had a highly significant influence on the physical properties of flakeboard. Increasing PF level in APS resins increased dry MOR and IB (Table 2). In particular, dry MOR increased 7% when PF level increased from 30% to 40%, but increased only 2% with further increasing PF level. Similarly, IB increased 26% when the PF level increased from 30% to 40%, but increased only a 12% when the PF level increased from 40% to 50%. MOE and wet MOR significantly increased with increasing PF level (Table 2).

Mechanical properties of wood-based composites, especially IB, are highly dependent upon adhesive strength of a binder. Most mechanical strength properties were increased more when the PF level increased from 30% to 40% than from 40% to 50%. These results are probably due to the different degree of crosslinking in cured gluelines. In other words, when the PF level increased from 30% to 40%, the degree of crosslinking in cured gluelines might have been significantly increased, resulting in improved adhesive strength. With 40% PF in soy resins, most soy protein segments might have been fully crosslinked, and thus further increases in PF level do not proportionally increase the mechanical properties of flakeboard.

Dimensional stability properties of boards also improved with increasing PF level in APS resins (Table 2). TS-24 and TS-2 reduced with increasing PF level. Within the same range of PF levels tested, WA-24 and WA-2 also reduced with increasing PF level. LE improved with increasing amounts of PF in APS resins. The LE reduced approximately 20% when PF level increased with a 10% increment. These results are probably caused by more crosslinking in cured resin systems with increasing PF level in APS resins, resulting in improving the dimensional stability properties.

3.2.2. Effect of Resin Usage

As shown in Table 2, dry MOR and MOE increased when amount of the resin applied onto wood furnishes increased from 5 to 7%, but the properties did not change significantly or decreased slightly with increasing resin content. IB value of boards with 7% resin content had the highest (100 psi), followed by boards with 9% resin content (94 psi) and boards with 5% resin content (88 psi).

Generally, greater resin coverage leads to more interparticle bonds, and thus the increased interparticle bonding should provide higher adhesive strength and durability. In addition to the resin coverage, board properties may also be influenced by mat moisture content. Excessive moisture in a mat requires a long press cycle to allow removal of moisture and to prevent delamination of the final board upon pressure release. In this study, mats with 5%, 7%, and 9% resin content have approximately 11%, 14%, and 17% total mat moisture contents, respectively. When mats with 9% resin content were pressed with a short press cycle, the IB value of the final board was lower than that with 7% resin content. This result is probably from low-density core or delamination in the final board.

There were significant differences in wet MOR and all dimensional stability properties among three resin contents (Table 2). The results of increasing resin contents yielding improvement of wet MOR and dimensional stability properties can be explained by a higher resin content that leads to more resin coverage onto wood furnishes. Consequently, boards made with a higher resin content have improved interparticle bonding, which improves the properties. LE also significantly reduced with increasing resin content (Table 2). LE reduced 15% as resin content was increased from 5% to 7%, but further resin addition had little impact, a 3% reduction. The reason for the different response of LE depending on resin content is probably that boards made with higher resin contents might already have enough interparticle bonds, resulting in little benefit of further increasing resin content.

3.3. Effects of Press Conditions on Board Properties

3.3.1. Press Temperature

Increasing press temperature positively affected physical properties except dry MOR (Table 2). Wet MOR and IB increased by 125% and 20% as press temperature increased from 175°C to 200°C. The wet MOR and IB properties are closely related to degree of resin cure and crosslinking. As results indicate, a press temperature of 200°C is needed to more completely cure or crosslink APS resins. Therefore, it can be concluded that APS resin system needs to be pressed at a 200°C press temperature to obtain a high adhesive strength.

Press temperature strongly influenced dimensional stability properties (Table 2). WA-24 and WA-2 showed 27% and 34% reductions with increasing press temperature. The reductions of WA values are probably due to improved resin curing in final boards. In other words, water entry into the well-cured board occurred at a slow rate due to the increased interparticle bonds, resulting in decreased porosity in the board. TS-24 and TS-2 also decreased by 45% and 54% with increasing press temperature. The reductions of TS showed a similar trend to Hse (1975). He reported that as press temperature was increased, a softening and plastic flow of resin and wood elements occurred, and this relieves internal stress build up during hot-pressing. Consequently, the high press temperature may contribute to thickness stability. With increasing press temperature, LE dropped from 0.42% to 0.30%, a 29% reduction. The reason for the differences in dimensional stability properties between the two press temperatures is probably due to increased degree of resin curing at high press temperature. In addition, the reduced hygroscopic property of wood flakes at a higher press temperature is likely to stabilize dimensional changes.

3.3.2. Press Time

Neither dry MOR nor MOE were affected significantly by press time (Table 2). However, wet MOR and IB significantly increased, when press time was increased from 8 minutes to 10 minutes. Generally, the curing of the resin does not occur uniformly, because an entire board is not uniformly heated within the thickness of a given board (Kelly, 1977). To allow the maximum resin curing in the core, lengthening of the press time at a constant temperature or increasing of the press temperature at a constant press time is needed. Press temperature, however, must be limited to prevent board surfaces in contact with the platens from being thermally degraded. Therefore, increased core temperature is accomplished by lengthening the press time (Kelly, 1977). Since the reactivity of APS resins is not as high as the PF or UF adhesives, it is necessary to use a high temperature to allow resin curing in the core, and this can be accomplished by a longer press time.

Lengthening of press time also significantly affects WA and TS of boards bonded with APS resins, but there was no significant effect on LE between 8 and 10 minutes (Table 2). When press time was increased, TS-24, TS-2, WA-24, and WA-2 reduced by 28%, 22%, 15%, and 14%, respectively. Longer press time might help the core part reach a sufficiently high temperature to allow the resin to cure more, and consequently, the uniformly well-cured resin network might improve the dimensional stability properties.

3.3.3. Press Temperature Press Time Interaction

When the effect of press temperature within a certain press time was compared, dry MOR ($p=0.01$), MOE ($p=0.02$), IB ($p=0.01$), TS-24 (0.01), and WA-24 ($p=0.01$) were found to have significant differences. Increasing press temperature did not affect dry MOR at a press time of 8 minutes, but the property decreased with increasing press time at 10 minutes. For MOE and IB, large differences were found between two press temperatures at 8 minutes of press time. Increasing press temperature at a press time of 8 minutes yielded much greater reductions of TS-24 and WA-24 than at 10 minutes. These results indicate that press temperature is a more significant factor than press time for improving physical properties of flakeboard bonded with APS resins.

3.4. Optimization of Processing Variables

A longer press cycle and a high press temperature are the best conditions for complete resin curing throughout the thickness of a board. However, hot pressing is one of the most significant and expensive operations in wood composite panel manufacture. The pressing conditions, therefore, should be carefully controlled to ensure that the core reaches a sufficiently high temperature to cure an adhesive without subjecting the board surface to a degradative temperature. In particular, the reduction of press time is the most effective way to improve the productivity for industrial applications of APS resins. From the results obtained, it can be concluded that an 8 minutes press time and a 200°C press temperature are the optimum conditions within the range of pressing conditions tested. Therefore, the conditions were used to optimize PF level in APS resins and resin

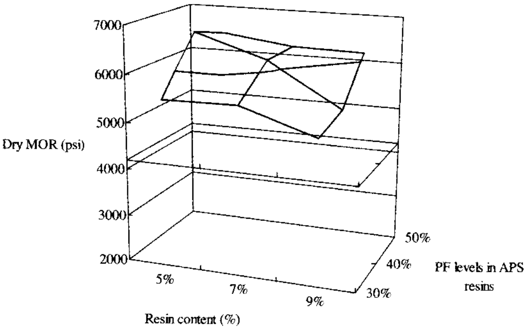

Fig. 1. Dry MOR of flakeboards prepared by APS resins with a press temperature of 200°C and press time of 8 minutes (The minimum requirement of CSA=4200 psi).

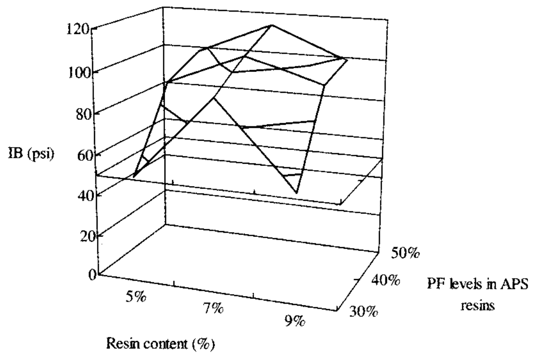

Fig. 2. IB of flakeboards prepared by APS resins with a press temperature of 200°C and press time of 8 minutes (The minimum requirement of CSA=50 psi).

content for exterior-grade flakeboards bonded with APS resins.

In comparison to CSA standards (SBA, 2001), dry MOR and IB values were well above the minimum requirement (dry MOR: 4200 psi; IB: 50 psi), regardless of PF level in APS resins, resin content, press temperature, and press time (Fig. 1 and 2). As shown in Fig. 3, most MOE values did not meet the minimum requirement (800 kpsi), but the property can be easily improved by flake alignment (Geimer and Crist, 1980; Liu, 1988; Shupe *et al.*, 2001). In order to meet the requirement of wet MOR (900 psi), APS resin should be formulated with 40% PF

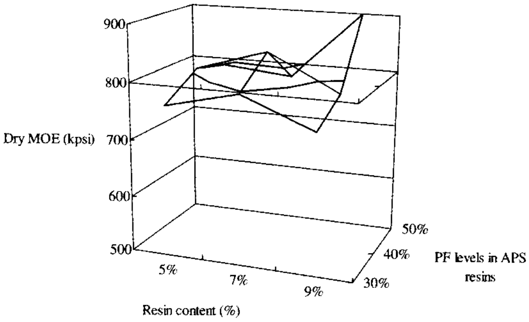

Fig. 3. Dry MOE of flakeboards prepared by APS resins with a press temperature of 200°C and press time of 8 minutes (The minimum requirement of CSA=800 kpsi).

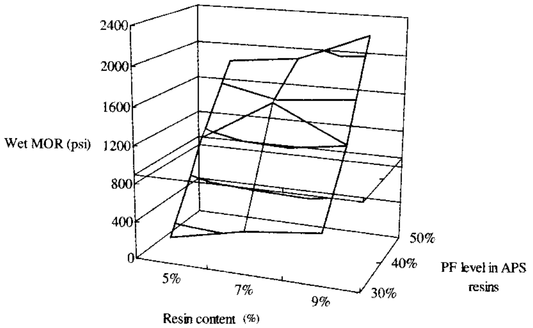

Fig. 4. Wet MOE of flakeboards prepared by APS resins with a press temperature of 200°C and press time of 8 minutes (The minimum requirement of CSA=900 psi).

in the resins regardless of resin content (Fig. 4). As shown in Fig. 5 and 6, the optimum PF levels for TS-24 and LE were determined to be 50% to meet the requirement (TS-24: 15%; LE: 0.35%). The LE, similar to MOE, can be improved by flake alignment.

Based on the results obtained, the following conditions with APS resins produced good board properties, which are above all requirements specified in the CSA standard: a 50% PF level, a 200°C press temperature, and an 8 minute press time at 5% resin content. These results indicate that APS resin for the manufacture of wood composite panels has potential, but a longer press time might limit its industrial

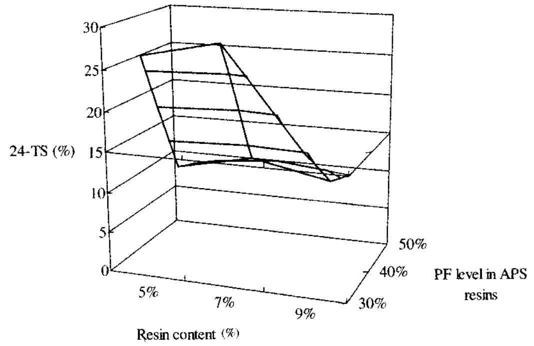

Fig. 5. Thickness swelling after 24-hour swelling of flakeboards prepared by APS resins with a press temperature of 200°C and press time of 8 minutes (The minimum requirement of CSA =15%).

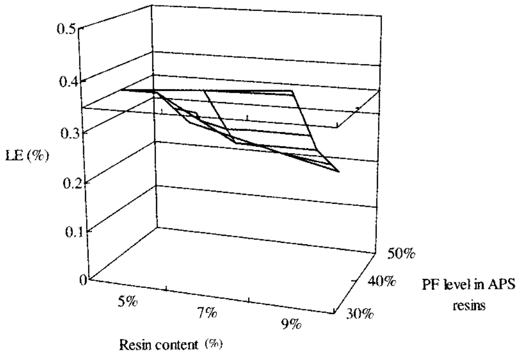

Fig. 6. Linear expansion after 2-hour boiling of flakeboards prepared by APS resins with a press temperature of 200°C and press time of 8 minutes (The minimum requirement of CSA =0.35%).

applications. The utilization of steam injection pressing and the construction of a layered-mat with different resins or resin contents may be used to reduce press time for practical uses of APS resin. Future work, therefore, should include these aspects. Other areas of research should include the study of the resistance of the boards bonded with APS resins to decay and insect degradation.

4. CONCLUSIONS

Physical properties increased with increasing PF level in APS resins. Dry MOR, MOE, and IB were higher at 7% resin level than at 9% resin level due to the low-density core or delamination of boards prepared with 9% resin level. However, the properties increased steadily with increasing resin level at a higher press temperature and a longer press time. Regardless of press conditions, wet MOR and dimensional stability properties improved steadily with increasing resin level. Increasing press temperature increased significantly physical properties except dry MOR. Press time showed no significant effects on dry MOR, MOE, and LE, but IB, wet MOR, TS, and WA improved with a longer press cycle. Results of this study suggest that APS resin can be used to manufacture hybrid poplar flakeboards. Within all the variables tested in this study, the following conditions were concluded to meet the requirements of the CSA standards for exterior-grade flakeboard at a 200°C press temperature and an 8 minute press time: a 50% PF level and a 5% resin content (total 2.5% PF), which was a 50% reduction of PF level for commercial flakeboard panels (5% pure PF resin).

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