Bond durability of wood-glue joints on Kapur (*Dryobalanops spp.*) and Yellow Seraya (*Shorea spp.*) bonded with phenol-formaldehyde resin adhesive

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Bond durability of wood-glue joints on Kapur (Dryobalanops spp.) and Yellow Seraya (Shorea spp.) bonded with phenol formaldehyde resin was investigated. The tensile shear strength of wood-glue joints bonded and tested thirteen years ago were compared with samples tested after thirteen years conditioning. The effect of treating wood (boiling for two to thirtytwo hours) on the tensile shear strength of the wood-glue joint was also taken into consideration. The tensile shear strength of Kapur and Yellow Seraya treated for 20-24 h and 15-20 h, respectively, before gluing was higher than that of the untreated samples which were bonded and tested thirteen years ago. The retention ratio of tensile shear strength of Kapur after 13 years was lowest at 20-24 h boiling. Likewise, results for specimens similarly tested showed the same pattern for both dry and wet conditions. However, variation of boiling time on the solid wood of both raw materials did not affect their ratios of decrease in tensile shear strength. Bond durability of Yellow Seraya wood-glue joints was higher than that for Kapur wood-glue joints as indicated by the higher retention ratio of the tensile shear strength of the former compared with the latter.

Key words: bond durability; wood-glue joints; Kapur; Yellow Seraya; phenol-formaldehyde resin adhesive; tensile shear strength

The southeast Asian woods Kapur and Seraya have been commonly used recently as raw materials for the wood industries. However, in the case of Kapur, the effects of its extractives on its bonding properties have been found to raise problems. For this reason, several studies on its extractives, including effects on bonding have been undertaken. Some examples are the investigations by Yagishita *et al.*[1], Imamura *et al.*[2], Abe *et al.*[3] and Akaike *et al.*[4], regarding the extractives of Kapur and their effects on the curing of phenol formaldehyde resin and other properties. Together with other researchers, this author has also investigated the wettability of different tropical woods and their extractives, and also on bond strength and durability[5-8]. Moreover, regarding the bond durability of Kapur veneers and sawn lumbers, weathering tests and swelling movements have been investigated and reported[9-13]. In all cases, the bond durability was investigated using either accelerated ageing tests or environmental exposure tests. There are as yet no published studies on bond durability after a period of more than 10 years.

This paper concerns investigations on phenol formaldehyde-bonded Kapur and Yellow Seraya woods conditioned over a period of 13 years, tested for bond strength and compared with the initial bond strength. In this case, the raw material adherends were first subjected to boiling treatments, the effects of which are also included and discussed here. A report on the initial bond strength of the above wood materials was presented at the Seventh Adhesion Research Conference of the Japan Adhesion Research Society[14].

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Experimental materials and methods

Materials

The raw materials used were Kapur (*Dryobalanops spp.*) and Yellow Seraya (*Shorea spp.*) 32 × 15 cm in area and thicknesses of either 1.0 or 0.50 cm, cut on the radial section. Samples of these sawn lumbers were used as cut whilst others were boiled for varying lengths of time. After 2, 5, 10, 15, 20, 24 and 32 h boiling, the lumbers were discharged and air-dried. After two months, the 0.5 cm-thick lumbers, together with the unboiled wood, were vacuum-dried until the moisture content reached about 7%.

Gluing process and bond strength

The pre-conditioned lumber materials were then planed such that two panels were flat and phenol formaldehyde resin was then applied to the flat surfaces ('Sumitomo Durez' brand Sumilight resin 9300 T) at 300 g m^{-2}; cold-pressed at 10 kg cm^{-2} for 20 min; left for 30 min and then hot-pressed at 135°C under 10 kg cm^{-2} pressure for 10 min. Following the standard DIN 5324 (see Fig. 17 in appendix), tensile shear strength test specimens were cut and prepared from the bonded wood. Likewise, with the 1.0 cm-thick solid wood, similar test specimens were prepared. For both cases, tensile shear strength tests were conducted under both dry and wet conditions. The wet conditions involved boiling the specimens for 4 h, followed by drying at 60°C ± 3°C for 20 h then boiling again for 4 h, then finally soaking in cold water (room temperature) until specimens had cooled[15]. In this report, the dry condition test is referred to as the initial untreated test; the wet condition, as the initial wet-treated test, respectively. Correspondingly, the test results for the bonded specimens are referred to as the initial bond strength results; for solid wood, as the initial tensile shear strength results, respectively. This first stage of the experiment was completed in 1968.

Bond durability test

A portion of the test specimens were left for 13 years in a properly ventilated room. In 1981, following the same testing methods, tensile shear strength was then determined.

As illustrated in Fig. 1, both the initial untreated and the initial wet-treated bonded wood and only the initial untreated solid wood were subjected to another dry and wet condition tensile shear strength test.

In Figs 2–7, each point represents the average of five test specimens.

Analysis of fractured specimens by scanning electron microscopy

In both phases of the experiment, that is, before and after 13 years, small samples from the fractured glue line of specimens were cut and observed under the scanning electron microscope, SEM, (Hitachi X650). Specimens from the unboiled wood samples and the 20 h-boiled samples from both wood species were observed and compared.

Results

Initial bond strength

Concerning Kapur, the initial bond strength and the initial tensile shear strength in relation to boiling time are shown in Fig. 2. The data for both dry and wet conditions show similar trends, in which the 10 h-boiled wood exhibited strength values lower than that of the unboiled wood. However, boiling within 15 to

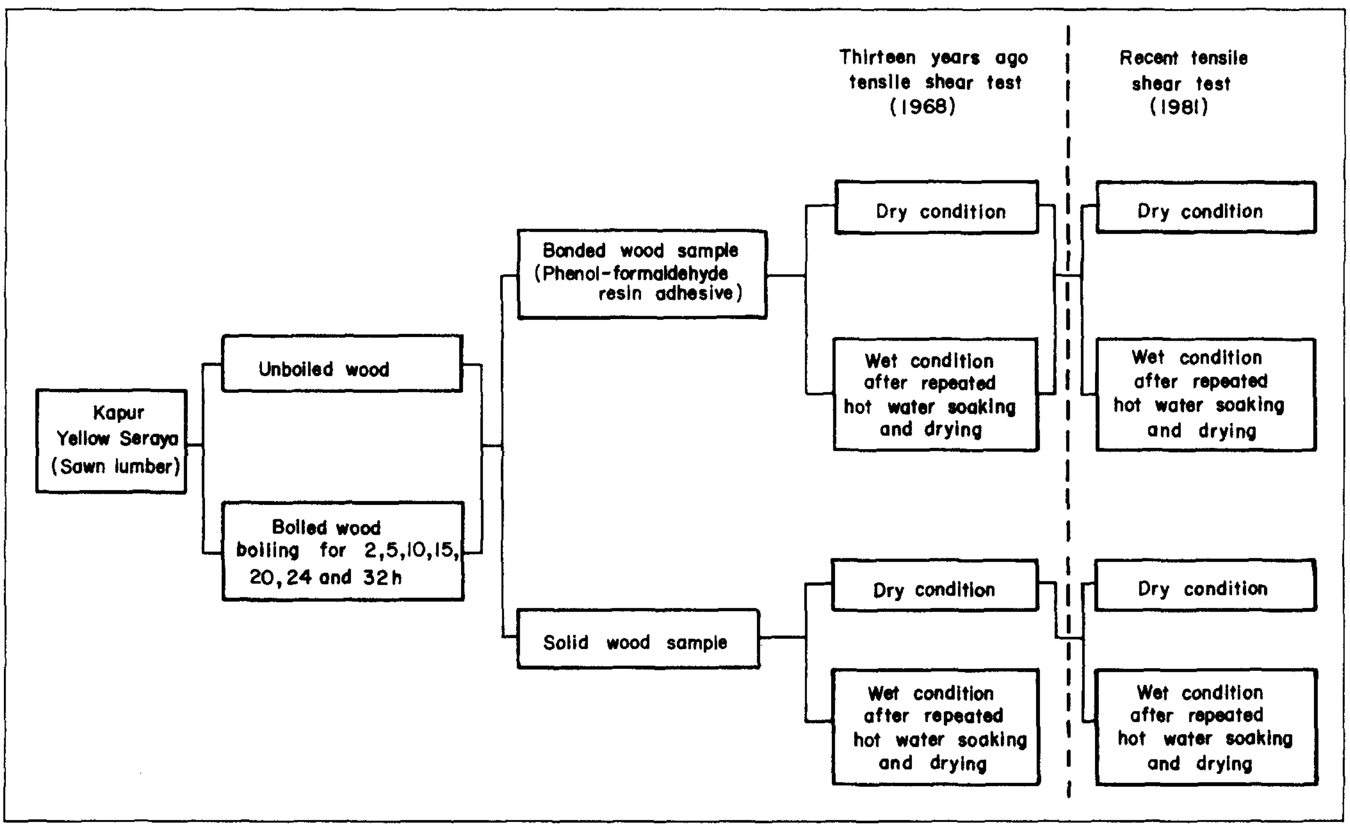

Fig. 1 Design for the preparation of samples and test conditions

24 h revealed increasing strength with longer boiling time; all the strength values of which were higher than for the unboiled wood. Boiling for 32 h proved destructive as very low values were obtained.

In relation to this, the tensile shear strength data for the solid wood shows a reverse trend in which the 10 h-boiled wood had strength values lower than that of the unboiled wood. Longer boiling times revealed lower values which did not differ from the unboiled wood.

From these results, boiling treatment apparently had an effect on bonding properties, that is, short boiling times effect a lower bond strength, while up to a certain limit, longer boiling times effect a higher bond strength.

The initial bond strength and tensile shear strength data for Yellow Seraya are shown in Fig. 3. The dry condition data show a trend similar to that for the Kapur wood. However, in the wet condition data, the bond strength of materials boiled for 20 h was similar to that of the unboiled material, while that for material boiled for 32 h decreased. With solid wood, the dry condition data show that the tensile shear strength decreased as the raw material was boiled. All strength values from any of the boiled wood samples were lower than the unboiled wood values. In the wet conditions, the effect of boiling was negligible; to wit, the unboiled and boiled wood had comparable values. The effect of boiling treatments on Yellow Seraya may be somewhat similar to Kapur, although not totally, as is clearly indicated for 15-24 h boiling, where an increase in bond strength was clearly observed.

Bond strength after 13 years

The relationship between boiling time and bond strength of Kapur is shown in Fig. 4. Under the dry conditions, the initial wet-treated specimens from the 10 h- and 32 h-boiled wood samples showed considerably lower values. All the other strength values were generally the same as that for the initial untreated specimens, and in all cases, the boiled wood had values lower than that for the unboiled wood. Under the wet conditions both the initial wet-treated and initial untreated specimens exhibited similar trends. Specifically, the 10 h-boiled wood samples showed values higher than that for the unboiled samples, whereas, at all other boiling times, the values were lower.

The effect of boiling treatment on the tensile shear strength of solid wood was negligible. After 15 h boiling, the values were slightly higher than the unboiled but all the others were generally the same.

In the case of Yellow Seraya, as shown in Fig. 5, in both dry and wet conditions, the trends of strength values were generally the same. Initial wet-treatment showed no apparent influence. Boiling for 5 h and beyond resulted in an increase in bond strength, with a 20 h boiling time showing the highest values. In this case, the trend in tensile shear strength of both the solid wood and the bonded wood were generally the same.

In gauging bond durability, the ratio between the bond strength after 13 years and the initial bond strength is presented. Up to 15 h boiling, Kapur, as shown in Fig. 6, revealed more than 80% strength

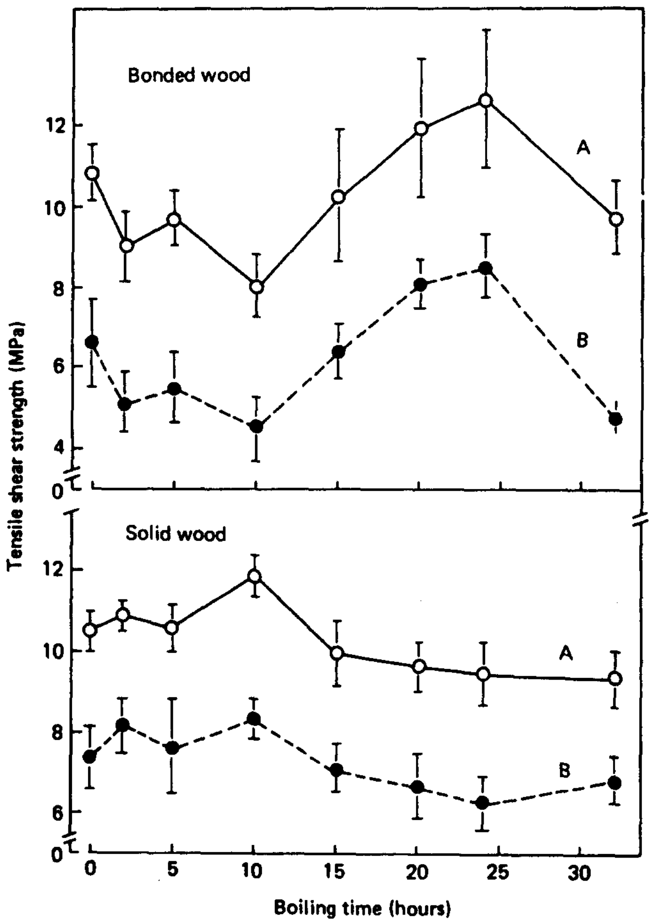

Fig. 2 Effect of boiling time on tensile shear strength of bonded wood and solid wood of Kapur tested thirteen years ago: (A): dry condition; (B): wet condition after repeated hot water soaking and drying

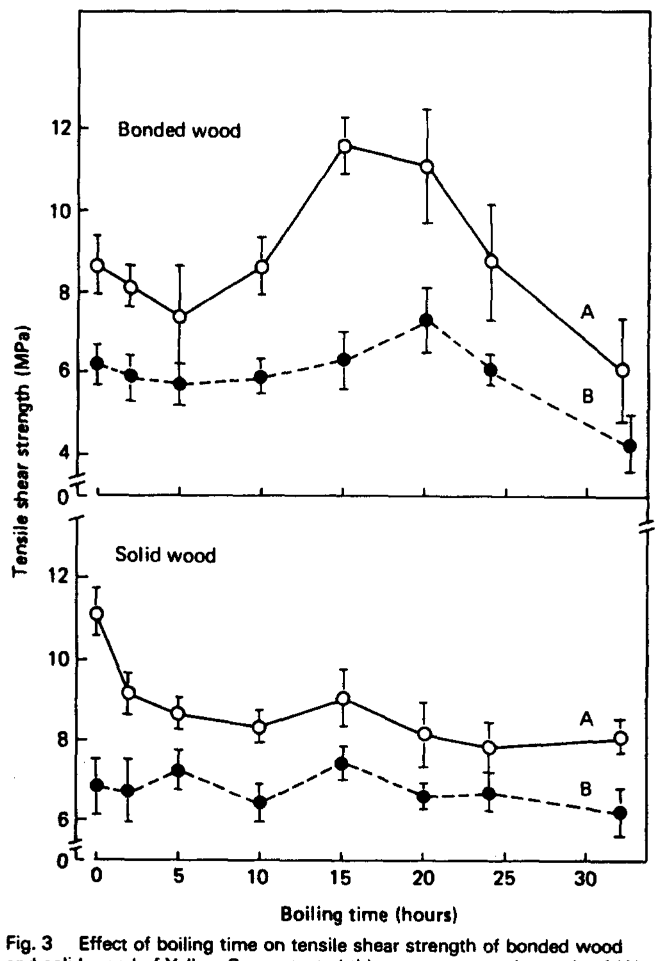

Fig. 3 Effect of boiling time on tensile shear strength of bonded wood and solid wood of Yellow Seraya tested thirteen years ago. Legends of (A) and (B) are as for Fig. 2

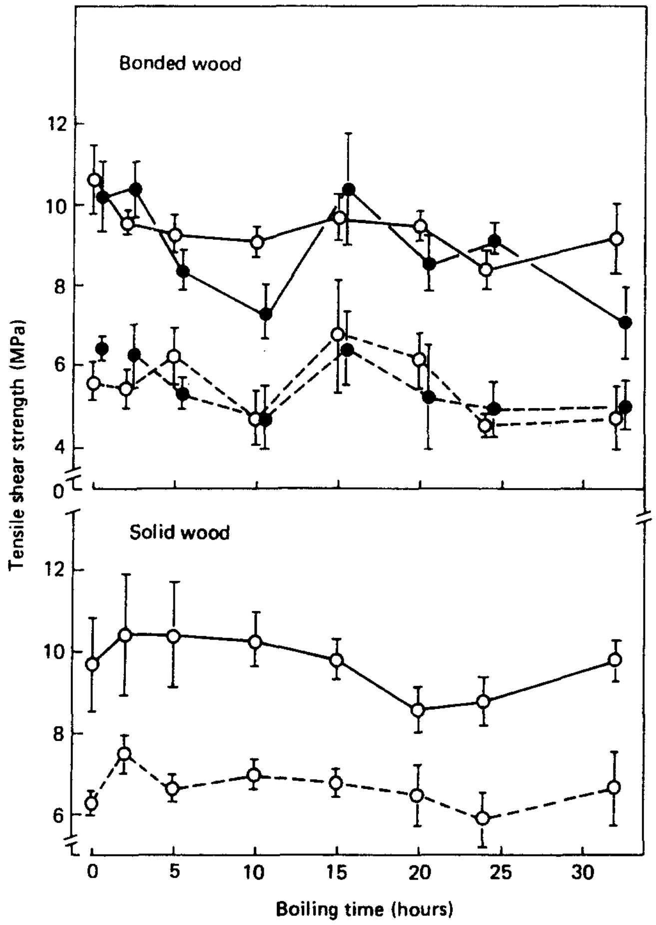

Fig. 4 Effect of boiling time on tensile shear strength of bonded wood and solid wood of Kapur tested after thirteen years conditioning: (O) conditioned for 13 years after bonding; (●) conditioned for 13 years after bonding and hot water soaking and drying repeatedly; (———) dry condition; (— — —) wet condition after repeated hot water soaking and drying

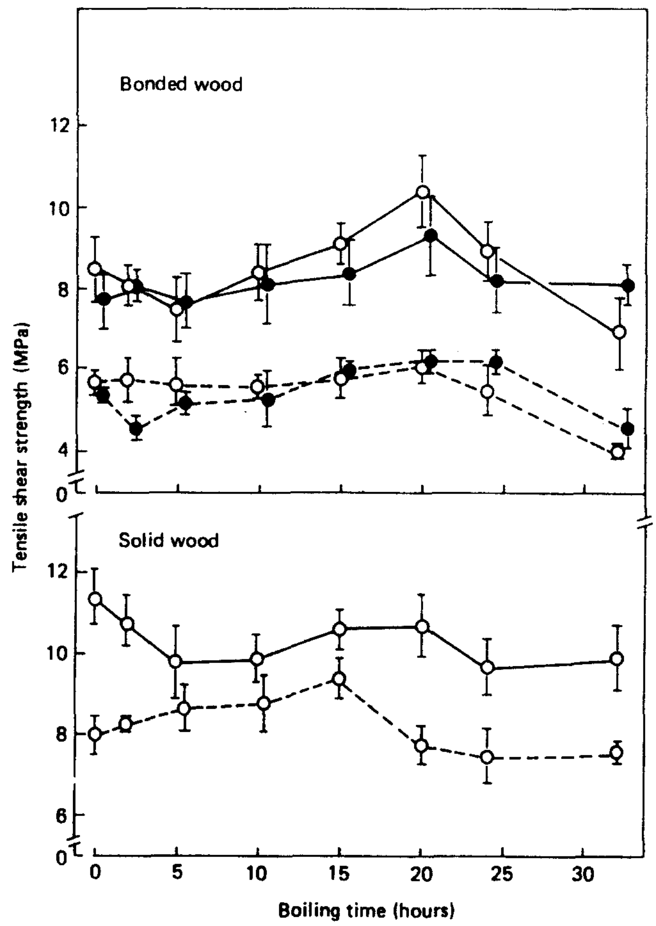

Fig. 5 Effect of boiling time on tensile shear strength of bonded wood and solid of Yellow Seraya tested after thirteen years conditioning. Legends are as for Fig. 4

retention ratios in all cases. Also several values indicate full or greater strength retention, especially in the wet condition wherein at the 2 h boil, the bond strength of the initial wet-treated specimen was greater than the initial strength by 20%.

On the other hand, in the case of the wood boiled for 20 h or more, the strength values were lower than the initial bond strength values with strength retention ratios lower than 80%. Within 20 to 24 h boiling, the initial bond strength values were exceedingly high (specifically at 24 h-boiling time). Wet condition data for both the initial wet-treated and the initial untreated specimens revealed strength retention ratios lower than 60%.

With solid wood, all the tensile shear strength values, after 13 years, were lower than the initial values. Strength ratios higher than 80% were observed in all samples, indicating that the boiling treatments had no influence.

With Yellow Seraya, as shown in Fig. 7, except for the bond strength of the 32 h-boiled wood, all values were lower than the initial bond strength. However, the decrease in strength ratios was very small and, except in only two cases, did not even go below 80%. Whereas, except for the wet condition data, the initial untreated bonded specimens from the 32 h-boiled wood showed strength values higher than the initial bond strength. The increase is quite marked with the initial wet-treated specimens tested in dry conditions.

In the case of solid wood, strength retention ratios were more than 80% in almost all cases, implying that the boiling treatments had no significant effect.

Analysis of fractured glue line with scanning electron microscope

The initial strength of the 20 h-boiled wood was found to be higher compared with that of the unboiled wood. However, the bond strength after 13 years compared with the initial bond strength showed a remarkably low

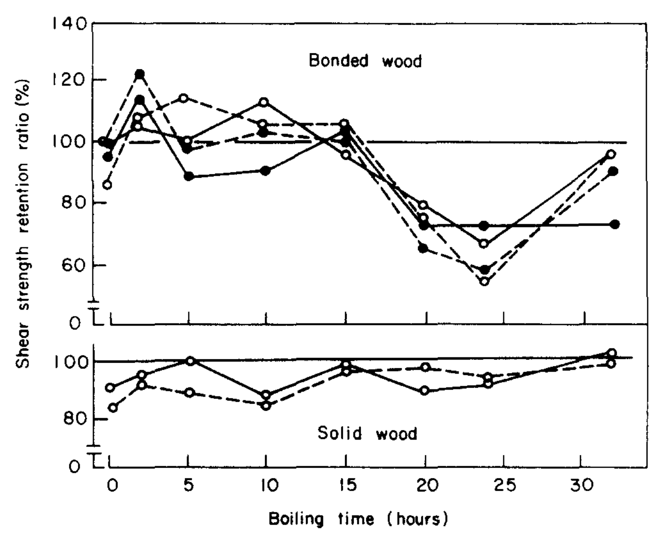

Fig. 6 Effect of boiling time on the shear strength retention ratio of bonded wood and solid wood of Kapur after 13 years conditioning. Legends are as for Fig.4

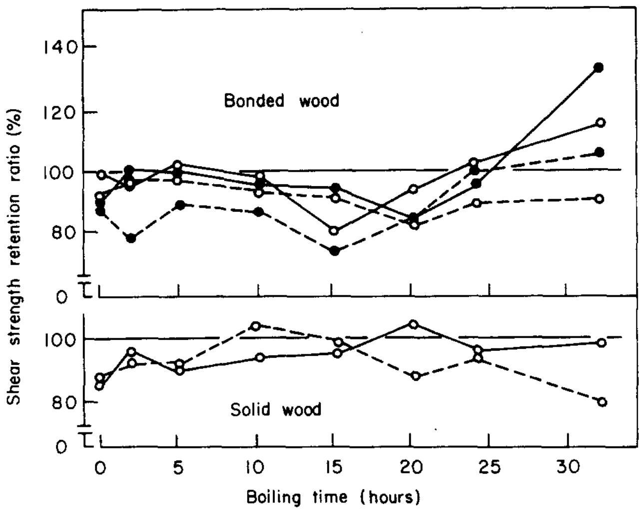

Fig. 7 Effect of boiling time on the shear strength retention ratio of bonded wood and solid wood of Yellow Seraya after 13 years conditioning. Legends are as for Fig. 4

ratio. This observation was especially marked with Kapur. For this reason, from the dry condition test samples, the glue line of the 20 h-boiled material was compared with that of the unboiled.

Firstly, regarding the initial experiment, the fractured glue line from the unboiled material tested under dry conditions is shown in Fig. 8. The fracture occurred near the wood portion of the glue line. However, the delaminated portions (arrow a) and a broken part of vessel glue cast (arrow b) can also be seen.

In Fig. 9 the fractured surface of the 20 h-boiled material tested in wet conditions is shown. Similarly to Fig. 8, it shows fracture on the wood part and also some unbonded sections of the glue line (arrow).

Fig. 10 shows the fractured surface of a bonded specimen from unboiled Yellow Seraya tested in dry conditions. Generally, the fracture was on the wood part but in a certain section a broken portion of a vessel glue cast can be seen (arrow).

For the 20 h-boiled material tested in wet conditions, the fracture was likewise greatest at the wood section (Fig. 11). However, at the vessel glue cast, separation at the interface between the glue and the wood adherend can also be clearly seen. This is different from Kapur

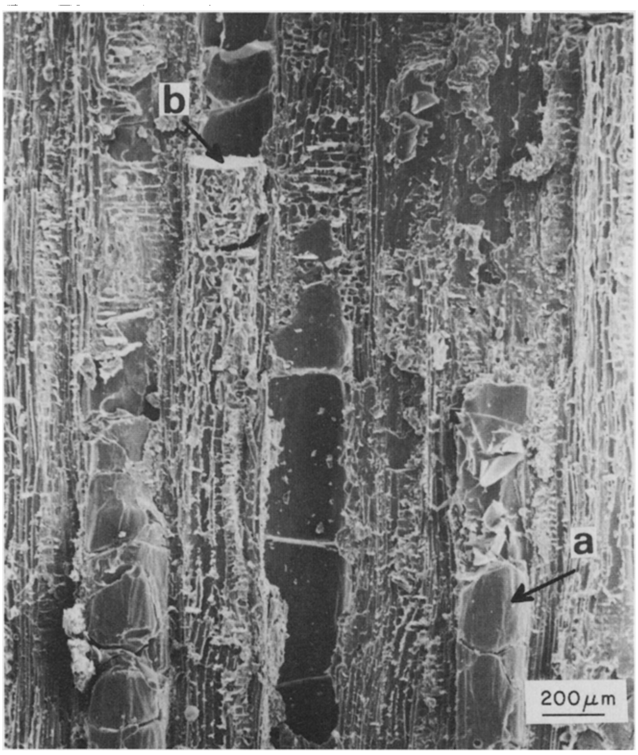

Fig. 8 Fractured surface on the wood-glue joint of Kapur bonded and tested by tensile shear under dry condition thirteen years ago. Adherend: unboiled wood; arrows: (a) delamination part of glue cast, (b) broken part of glue cast

Fig. 9 Fractured surface on the wood-glue joint of Kapur bonded and tested by tensile shear under wet condition thirteen years ago. Adherend: 20 h boiled wood; arrow: a part of unbonded cured glue

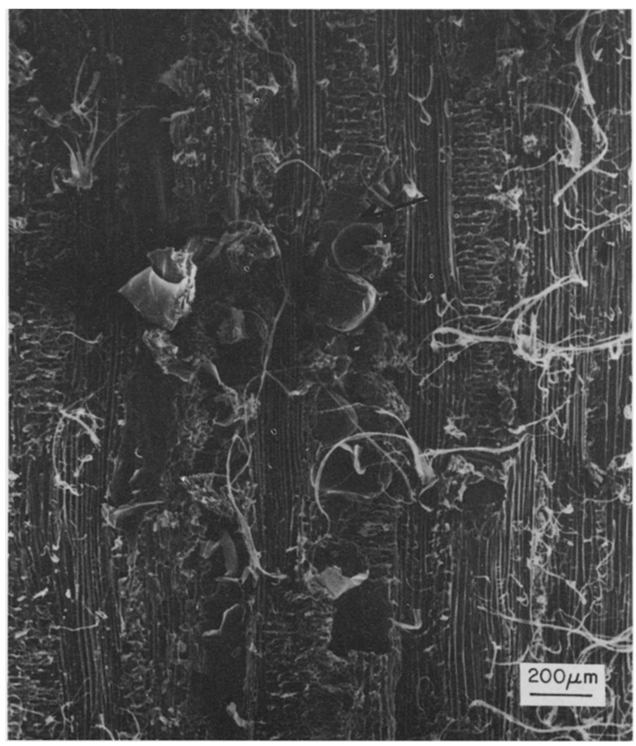

Fig. 10 Fractured surface on the wood-glue joint of Yellow Seraya bonded and tested by tensile shear under dry condition thirteen years ago. Adherend: unboiled wood; arrow: fractured part of glue cast

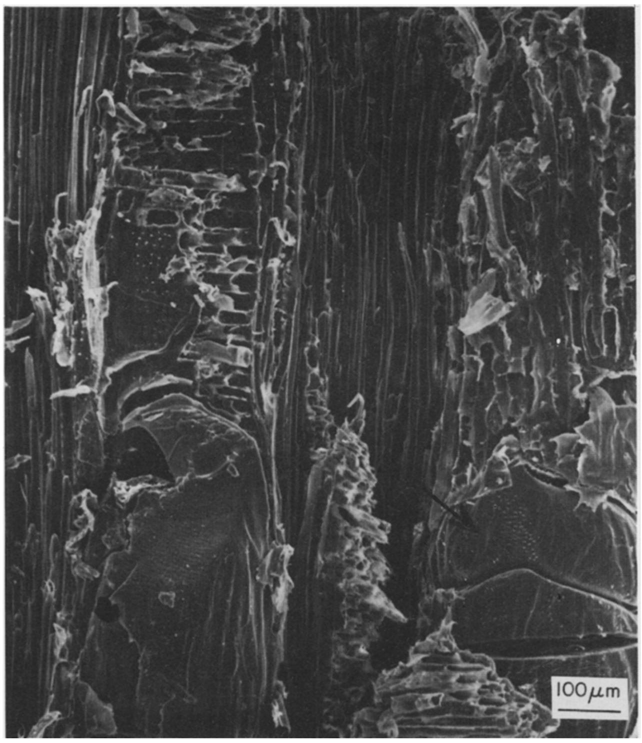

Fig. 11 Fractured surface on the wood–glue joint of Yellow Seraya bonded and tested by tensile shear under wet condition thirteen years ago. Adherend: 20 h boiled wood; arrow: a part of delaminated surface of glue cast

in that the replica of the wood adherend is clearly printed on the glue surface (arrow) thereby indicating the wood adherend was well-bonded.

The following are the results of observations on fractured surfaces of 13-year conditioned specimens.

In Fig. 12 the sample from the unboiled, initial untreated specimen tested in dry conditions is shown. A large area of the fracture was also on the wood part. Where large vessels were filled with glue, either delamination at the surface of the wood adherend (arrow a) or crosswise breaking of the vessel glue cast (arrow b) occurred. In this case the vessels were fully filled and well-bonded by the glue such that the fracture damage was greater on the wood part.

The sample from the 20 h-boiled material, initial wet-treated and tested under wet conditions also revealed delamination at the vessel glue cast as shown in Fig. 13. The bottom section shows a heavily damaged portion. Also, on the delaminated surface the glue-penetrated pits can be seen which, on separation, remained as a protruding glue cast (arrow). This particular portion was enlarged as shown in Fig. 14. The delaminated surface shows the porous or sponge-like cured glue, while the glue-penetrated pits were protected from damage.

With the Yellow Seraya bonded specimen from the unboiled wood, initial untreated and tested under dry conditions, showed similar results to the initial specimens. A large part of the fracture was also on the wood part. Likewise, penetration of glue into the vessels seemed standard.

A specimen from the 20 h-boiled material, initial wet-treated and tested under wet conditions is shown in Fig. 15. This shows separation at the vessel glue cast at the interface between the glue and the wood adherend. This again indicates good bonding between the glue and the adherend. A magnification along the perforation rim of the vessel glue cast is shown in Fig. 16. The glue spread and penetrated well such that at the delaminated surface of the glue line the replica of the well-bonded adherend can be observed. Penetration and drawing out of glue through the pits and vessels can also be clearly seen.

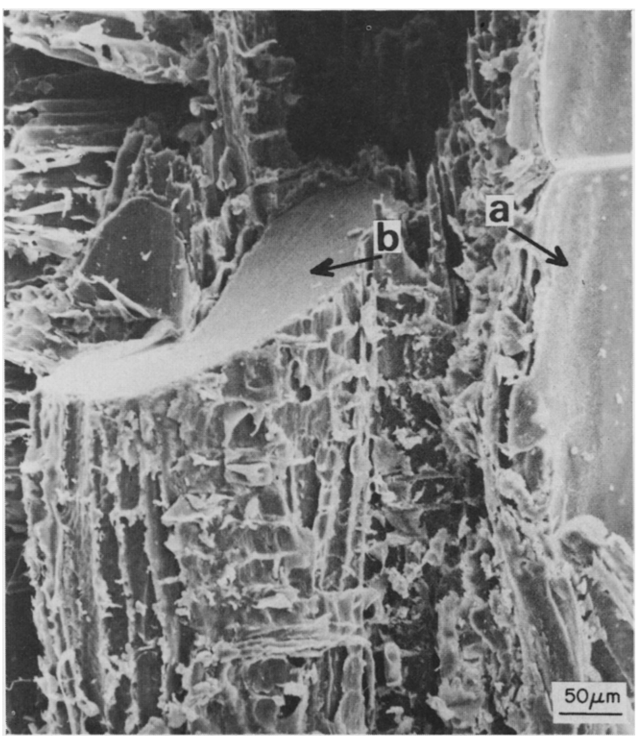

Fig. 12 Fractured surface on the wood–glue joint of Kapur bonded and conditioned for thirteen years and tested by tensile shear under dry condition. Adherend: unboiled wood; arrow: (a) delamination part of vessel glue cast, (b) broken part of glue cast

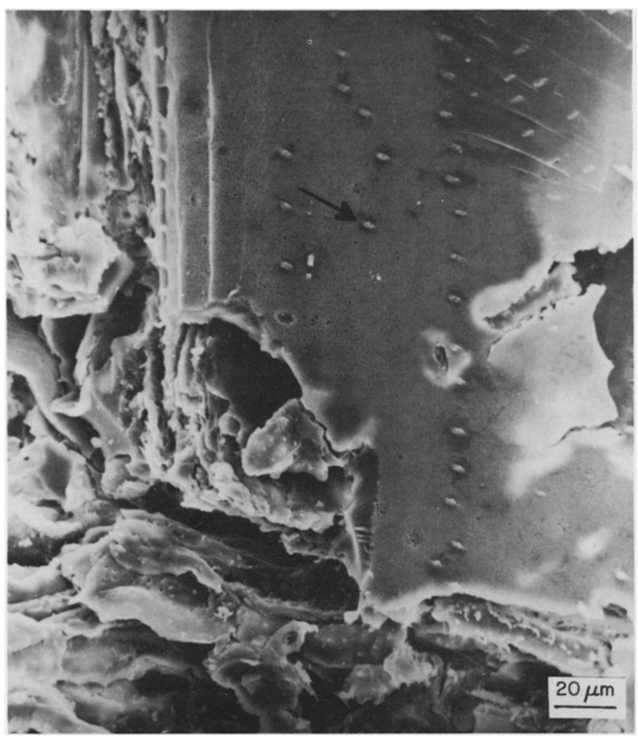

Fig. 13 Fractured surface on the wood–glue joint of Kapur bonded and conditioned for thirteen years after repeated hot water soaking and drying and tested by tensile shear under wet condition. Adherend: 20 h boiled wood; arrow: delamination part of vessel glue cast and pit glue cast

Fig. 14 Magnification from Fig. 13 pointed by arrow. The surface of the cured glue is porous

Discussion

Initial bond strength

That Kapur is not a good adherend because, as reported, of its extractives has been previously mentioned. Such extractives hinder the curing of the glue[2-4], and the wettability of the wood with respect to the glue is also badly affected[5-8]. Depending on the extraction method, the type of extractives can differ. Known to render particular effects on bonding are the ethers, in which the hot water extracts are plentiful in amount[1,2]. These hot water-soluble extracts, together with other ethers, on the contrary shorten the curing time of adhesives such that the penetration of the adhesive into the wood material is hindered thereby effecting poor bonding[16].

In that connection, the higher the initial strength of the wood boiled for 15 to 24 h compared with the unboiled wood must have been due to the removal of the hot water extracts. In relation to this, within 10 h of boiling the wood exhibited strength lower than that for the unboiled wood. This is probably because the hot water extracts had moved to the wood surface thereby badly affecting the wettability and penetrability of the wood with respect to the glue.

On the other hand, the tensile shear strength of the solid wood decreased with longer boiling time, a trend opposite to that of bond strength. This clearly indicates that boiling treatments affect glue bonding.

Regarding the hot water soaking-drying cycle test, the wet condition data showed strength values lower than the dry conditions with almost consistent effects on the unboiled and all the boiled wood. Almost the same pattern was observed with the solid wood although the ratios were lower than those with bond strength. Damage to wood-glue joints was greater than on solid test specimens such that the difference with respect to boiling time was not found.

Observations on the nature of damage near the glue line of the 20 h-boiled wood, tested in wet conditions, indicated severe wood fracture. However, delamination of the glue line was also found at various points which indicates areas of poor bonding of the adherend. These portions may have possibly accelerated the extent of damage causing the decrease in bond strength.

The same thing can be said with Yellow Seraya.

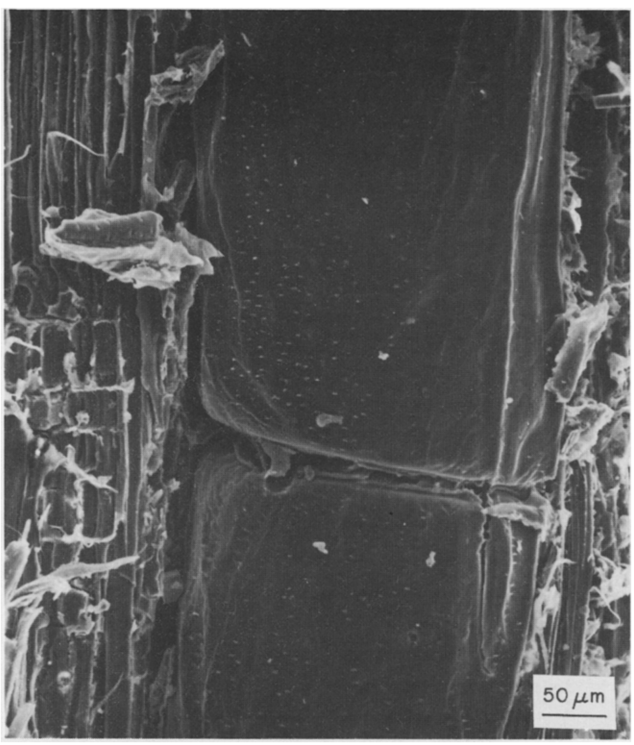

Fig. 15 Fractured surface on the wood-glue joint of Yellow Seraya bonded and conditioned for thirteen years after repeated hot water soaking and drying and tested by tensile shear under wet condition. Adherend: 20 h boiled wood. Delamination surface of the vessel glue cast appeared on the replica of adherend surface

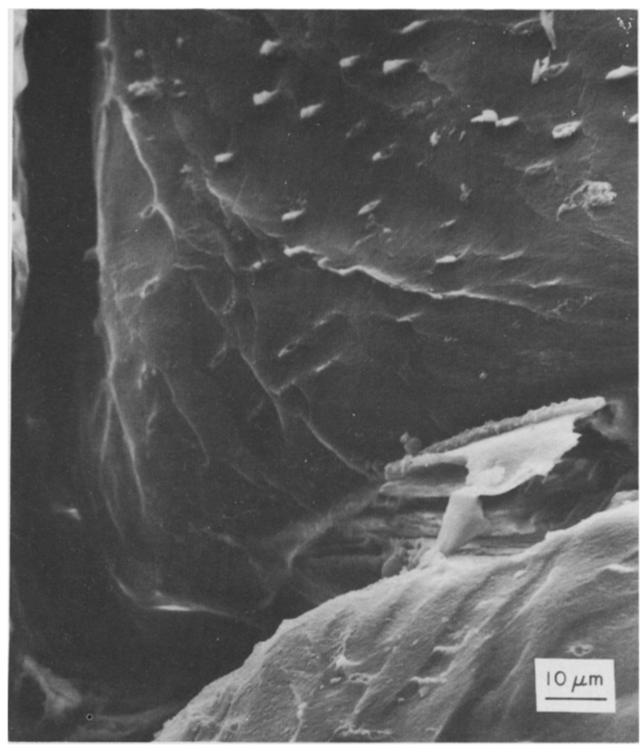

Fig. 16 Magnification from Fig. 15 showing the perforation rim of the vessel glue cast. The replica of adherend surface can be seen more clearly

This, therefore, affirms that hot water extracts influence bonding performance. Until this time, the effect of extractives on bonding performance with regard to Yellow Seraya has not been previously reported.

From observations of fractured specimens by SEM, in both the dry and wet conditions, failure on the wood part was greater with Seraya than Kapur. Also, from the samples observed after wet testing, there was a replica of the wood adherend on the glue surface where glue and wood separation occurred. Due to the damage promoted by the wet test, these well-bonded portions of wood may have become swollen bringing about this failure.

Bond durability after 13 years

After 13 years, the bond strength of Kapur, whether initial untreated or initial wet-treated, was practically the same. This indicates very strong bonding durability, that, after 13 years, no amount of destructive treatment could affect. Compared with the unboiled wood, the boiled wood exhibited almost the same or lower bond strength values. Concerning long-term bond durability, boiling treatments indicated no significant effect. Furthermore, with regard to strength ratios at each boiling treatment in relation to initial strength, the values were very low at 20–24 h boiling where the initial strength values were very high. There was, however, not much change with respect to short-term boiling, such that the bond strength values were also higher than the initial values.

On the other hand, the tensile shear strength of the solid wood decreased slightly. However, there was actually not much damage and boiling had no significant effect. Consequently, as the bond durability is manifested by the glue line, there is not much need to consider the change in strength in solid wood. Finally, it becomes clear that with more than 20 h extended boiling treatment, the initial bond strength increases but the long-term bond durability is meager.

Why the long-term bond durabilty is so small cannot, however, be clearly explained. From the SEM observations the following can be deduced: with the 20 h-boiled wood, destruction at the interface between the glue and the wood adherend was marked. Because bubbles generated during the gluing process, the surface became porous (Fig. 13) probably causing the decrease in bond strength. Before this, SEM analysis of phenol formaldehyde-bonded plywood specimens eliminated of wood, revealed glue penetrating inside cell lumens and pits[10]. However, analysis of the fractured surface showing the pits penetrated with glue and, upon delamination, protruding, has never previously been reported. This indicates that long boiling times cause specimens, left for extended periods, to yield easily to delamination.

With Yellow Seraya, the solid wood showed a similar trend to Kapur with virtually the same behaviour with regard to changes in bond strength. Accordingly, the damage at the glue line was nil and the effect of initial treatment was also insignificant. Examining further, at 20 h-boiling treatment, where the initial strength was extremely high, after 13 years the strength values were still high at more than 80% retention ratio. This is quite different from the Kapur, where the boiling treatments had definite effects on bond durability after 13 years.

In addition, the 32 h-boiling treatment caused considerably lower initial bond strength but in no way influencing the bond strength values after 13 years. Several values higher than the initial ones indicate improved bond durability. Unfortunately, no reason for this phenomenon was clearly identified.

From SEM observations of the fractured specimens, delamination was marked at the surface of the large vessel glue cast. On either side of the adherend where large vessels are, glue penetrated and cured during the gluing process. In such portions, the glue line became thick with the adherend shrinking after repeated drying. Such movements, which differed from that of the adhesive, probably caused these portions to delaminate easily. However, with Yellow Seraya, as can be seen in Fig. 16, there was good bonding between the adhesive and wood adherend. The wood's cell structure is considerably well-defined and this might be one reason for its superior durability.

Conclusion

The bond strengths of phenol formaldehyde-bonded Kapur and Yellow Seraya before and after a lapse of 13 years were investigated and compared. In this case, to increase bond performance, the wood adherends were boiled first before bonding, the influence of which was analysed. The following conclusions were arrived at:

1) As to the initial strength, boiling of Kapur for 20 to 24 h and of Yellow Seraya for 15 to 20 h gave high strength values. The wet condition results, although lower than the dry condition results, also exhibited a similar trend. With solid wood, the effect of boiling being insignificant clearly indicates that certain boiling treatments render some improving effects on bonding performance.

2) With Kapur, after 13 years all treatments resulted in strength values which were either equal to or lower than that of the unboiled wood. The 20 to 24 h-boiled wood showed meager bond durability, that is, strength retention ratios were very low compared with other treatments.

With Yellow Seraya, there was a lesser degree of decrease in strength. As to the effect of boiling treatments, the trend was generally similar to that with initial bond strengths with satisfactory bond durability. Moreover, none of the boiling treatments caused any significant decrease in the tensile shear strength of the solid wood.

3) As to the SEM observations on the fractured specimens, delamination at the surface of the vessel glue cast were clearly evident. With Yellow Seraya, the delaminated portion showed an exact replica of the wood adherend which indicates better bonding compared with Kapur.

Acknowledgement

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Appendix

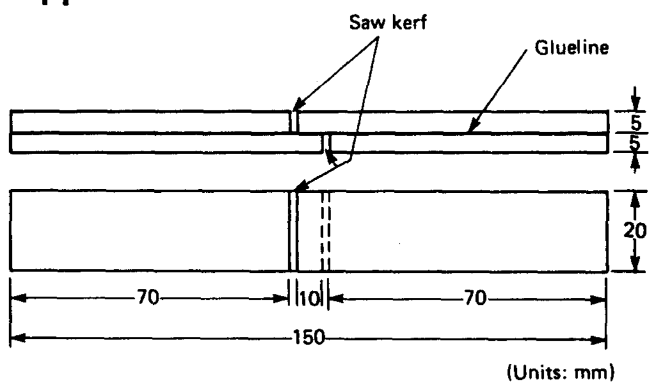

Fig. 17 Form and dimensions of test specimens