SOME GLUING CHARACTERISTICS OF <u>OCOTEA</u> <u>USAMBARENSIS</u> (ENGL.)

by

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ABSTRACT

The gluability of a Kenyan tropical hardwood, camphor wood (*Ocotea usambarensis*) was investigated. Four room-temperature curing resin adhesives, phenol-resorcinol formaldehyde (PRF), urea-formaldehyde (UF), polyvinyl-acetate (PVA) and casein were used.

Machine-planed wood samples were extracted with either hot water, 10% solution of sodium hydroxide, alcohol-benzene, surface treated with 3% nitric acid, or left untreated (control) before gluing.

Using a double glue spread of 410.6 g/m^2 (85 lb per 1000 ft^2) and the manufacturers recommended assembly times of each of the 4 glues, wood blocks approximately 25.4 x 101.6 x 304 mm (1 x 4 x 12 in) were bonded together. Gluing and pressing were carried out at room temperature (21-24°C). A pressing pressure of 1,379 kPa (200 psi) was applied over a period of 24 h.

After conditioning, the joint strength and wood failure of both the untreated and treated blocks were determined by the ASTM standard glue block shear test. The blocks bonded with PVA and casein were tested dry. Block shear specimens for PRF were tested dry, after cold soaking, and boiling in water. Blocks bonded with UF were tested dry and after cold soaking in water.

Statistical analysis showed that strength of adhesion joints bonded with PVA adhesive was significantly improved by

surface treating with weak nitric acid, prior to gluing. However, none of the four wood pre-treatments significantly improved gluability of camphor wood with PVA as far as wood failure is concerned.

Surface treatment of camphor wood with sodium hydroxide solution, and extraction with alcohol-benzene, prior to gluing, enhanced its gluability with casein adhesive.

Removal of the alcohol-benzene-soluble and sodium hydroxide-soluble extractives significantly increased dry bond strength of the blocks bonded with PRF adhesive. However, only the alcohol-benzene extraction increased wood failure.

The cold soak treatment generally reduced the bond strength of blocks bonded with PRF adhesive. Wood failure percentage was increased by the cold soak treatment except in blocks made with sodium hydroxide-treated wood.

The boil treatment was observed to reduce bond strength in all the treatments except in the control. Other than in the sodium hydroxide treatment,the amount of wood failure increased as a result of the boil treatment.

Untreated camphor wood bonded well with UF adhesive. Bond strength was significantly reduced by cold soak treatment.

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1.0 INTRODUCTION

1.1 Wood Products Industry in Kenya

The demand for wood products in Kenya has been rising rapidly and will continue to rise in the future. According to FAO (10) estimates of the consumption of industrial roundwood, in Kenya, is expected to rise from 740,000 m^3 to 1.067 million m^3 by the year 2,000. These figures indicate a continuing rapid increase in the volume of domestic consumption of wood products.

The growth of wood products consumption in Kenya is attributable to a rapid population growth, estimated at an annual rate of 4%, and the increasing per capita consumption of wood and wood products, as a result of a general improvement in the standard of living. To meet this rising demand for wood products, within the constraint of a fixed forest land base, two options open to the Kenyan government are: a more intensive forest management to maximize yield from the available forest land and more rational and efficient utilization of the available wood. Over the past two decades, considerable measures of silvicultural manipulations of the growing trees stock has been implemented in the country. However, the improvement of the efficiency of timber conversion and the utilization of wood processing residues has not been adequately addressed.

Due to the high demand for wood products, the large old-growth forest in Kenya is being rapidly depleted. This has

resulted in a decrease in the availability of solid
structural timber, especially for hardwoods. Thus the future
of the wood industry, in Kenya, almost certainly lies with
the greater use of reconstituted wood products.

As the industry shifts more towards reconstituted
products there will be an increasingly greater use of
adhesives to convert second-growth timber into serviceable
products.

Gluing is one means by which the wood-products
industries fill the gap between market needs and wood supply.
If the trees are not long enough, shorter pieces are end-
jointed into longer pieces; if the wood is not wide enough,
narrow pieces are edge-glued into wider material; if the wood
is of low quality, the knots and other natural defects are
cut out and the remaining pieces are glued together (34).

The quality of wood products such as plywood, laminated
timber, and particleboard, and the performance of glued
joints in such manufactured items as wood furniture depend
upon the success of glue bond formation (15). Bonding of
wood with glue is a complex process, because the quality of a
gluing operation is dependent on a large number of factors.
The failure to properly control any one of these factors can
result in a defective or weakened glue joint. The cause of
faulty joints may be categorized as related to:

    (i)   the kind of wood and its preparation prior to
            gluing;

    (ii)   the type, quantity and quality of the adhesive;

(iii) the compatibility of the gluing process with the wood and adhesive used;

(iv) joint design, assembly time and clamping pressure; and

(v) post-treatment or exposure conditions of the finished glue joint.

A number of these factors are interrelated such that a change in one may require a change in another. The effect of these factors on a glued joint varies from adhesive to adhesive (10).

In bonding wood with adhesives one must be aware that wood is not a uniform substance, but a complex material that varies significantly in many of its properties and it would be a mere chance if the same bonding material and procedure would be suitable for the entire range of wood species. Wood factors relevant to good adhesion include; wettability, surface texture, aging prior to gluing and species (density, extractives, moisture content, and pH). Each of these factors plays an important role in the formation of strong and durable glue joints. Usually, it is therefore, difficult to pinpoint which plays a greater role than the other. Furthermore, some of these factors are closely related (41, 19)

1.2 Scope and Objective of Study

Ocotea usambarensis, commonly known as camphor wood, is abundant in the montane rain forests of Tanzania and Kenya.

Camphor wood is yellow-brown, darkening on exposure to air and has a medium texture with marked stripe or ribbon figure on radially sawn faces. The timber works easily with both power and hand tools. However, it has a moderate gluability characteristic (6).

Over the years, gluing difficulties have been observed in Kenya's wood industry when certain hardwood species, like camphor wood were used for composite products and in furniture making. There are reports of glue-joint delaminations even for joints made using kiln dried lumber. Thus, wood moisture content is not the sole determining factor of a strong durable glue bond.

Adhesion, and hence strength of the glue bonds, is affected by the surface properties of the substrate. In the case of wood the surface characteristics that may affect glue bond formation are quite complex. One of the factors that appears to affect adhesion is the amount and type of extraneous components present in the wood (15). No formal study has been carried out to investigate the specific nature of the extractives present in Kenyan hardwoods and their probable effects on adhesion.

The main objective of this study is to determine if any of the extractives removed by various solvents contributes to the adhesion problems in camphor wood.[1]

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[1] The qualitative analysis of the various extractives present in camphor wood is beyond the scope of this study.

The study proposes a hypothesis that chemical treatment of camphor wood surfaces with 10% sodium hydroxide solution, 3% nitric acid, or extraction with alcohol-benzene, and hot water, prior to gluing, improves glue bond and durability.

The following shall be investigated:

(i)   the gluing characteristics of solid camphor wood (unextracted) samples using four room temperature curing adhesives, viz; phenol-resorcinol formaldehyde (PRF), urea-formaldehyde (UF), polyvinyl-acetate (PVA), and casein;

(ii)   the amount of alcohol-benzene, hot water, cold water, and caustic soda (NaOH) soluble extractives in camphor wood and the effect of their removal on the gluing characteristics;

(iii) the effect of surface treatment (oxidation) of camphor wood with weak nitric acid solution, on the gluing characteristics; and

(iv)   the effect of cold water soak and boil treatments, before testing, on the strength of the wood-glue bond.

2.0 LITERATURE REVIEW

This section covers a selected review of existing literature on:

    (i)   effects of extractives on gluing, and

    (ii)   effects of chemical treatment of wood surfaces
           prior to gluing.

2.1 Effect of Extractives on Gluing

Woods of different species vary widely in the nature and amount of their extractives. Extractive quantities vary within tree (24). Heartwood normally has substantially higher quantities of extractives than sapwood (13, 39). Many of the tropical hardwoods are characterized by high extractive content (39).

Isenberg *et al.* (25) in their study of the extraneous components of American pulpwoods, subdivided the total substance of the tree into four groups:

    (i)   the cell walls, consisting of cellulose,
           hemicelluloses, lignin and sometimes small amounts
           of pectic substances and mineral matter;

    (ii)   the extractives which are removable by cold water
           and/or one or more of neutral solvents such as
           acetone, ether, alcohols, benzene, petroleum
           ether, chloroform or methylene chloride;

(iii) substances which though not part of the cell wall, but are not readily removable by solvents, such as starch grains and crystals of calcium oxalate or silica; and

(iv) secretions of the living tree such as resins.

In this thesis, the term extractive(s) refers to substances identified under (ii) and (iv) above, that is, extraneous material or secretions of the living tree.

Gluing difficulties have been noticed in wood industries when certain species of wood, with high extractive content, e.g. tropical hardwoods, are used for reconstituted wood products. Results from several empirical studies point out that the role of extractives may some times be very important in the gluing of wood (15, 17, 28, 39).

Troop and Wangaard (45) investigated the gluing properties of twenty-nine tropical American woods in addition to Burma teak and domestic white oak. Resorcinol and phenol-resorcinol adhesives were employed in the study. These two types of adhesives were not equally satisfactory for gluing white oak and Burma teak. The general trend was that of increasing joint strength and decreasing wood failure with increase in specific gravity. However, some anomalous results were also obtained. These suggested some interference of wood constituents with the adhesive in certain species. They postulated that such interference could be the result of defective surfacing or due to the

character of chemical components, such as gums, resins, oils, and waxes which occur in varying amounts as extractives in many woods.

Narayanamurti (35) concluded that the distribution of extractives varies both vertically and horizontally in a tree. He points out that extractives affect the hygroscopicity, swelling and shrinkage of wood, and, at high temperatures may have other effects. Their effect on the gluing of wood is of special importance.

Western larch (*Larix occidentalis*) usually exhibits, on the surface of machined wood, a sugary exudate. The quality of glue bonds obtained with this species, using an exterior type phenolic resin adhesive, is influenced to a markable extent by the amount of this exudate. Plywood panels made from veneers exhibiting heavy deposits develop glue bonds which do not meet the industry standard for exterior glueline quality. The bond quality increased as the amount of surface deposit decreased (5).

Narayanamurti *et al.* (36) report on the influence of extractives on the setting of adhesives. The effect of various extractives from *Acacia catechu* and *Tectona grandis* (teak) on the gelation time and rigidity modulus of animal glue and Aerolite, an urea-formaldehyde resin, were investigated. They concluded that extractives affect the viscosity and rigidity of glue. The effect may vary from glue to glue and from species to species. The effects of teak extractives were more pronounced than those of the *Acacia* species.

Hancock (21) studied the influence of native fatty acids on the formation of glue bonds with heat treated Douglas-fir veneers. He noted that heat inactivated the veneer, thus forming a weak glue bond. He postulated that, the removal of water from wood, and the application of heat, permits the migration of fatty acids onto the wood surface. The fatty acids reduce the wettability of veneer and also affect rate and depth of penetration of glue.

Chugg and Gray (17) in a study, using *Afrormosia elata* wood, report that extractives inhibit the setting of most adhesives, particularly animal glue, catalysed phenol-formaldehyde, and polyvinyl acetate. Extractives also lower the surface tension of the wood and reduce wettability, which is essential for a strong glue bond.

Goto *et al.* (20) investigated the gluing of tropical woods. They concluded that the relationship between the glue joint strength and percentage of either cold or hot water-soluble extractives was not significant. However, glue joint strength increased with the decrease of percentage of ether soluble extractives, if the effect of specific gravity is excluded. They also noted the value of pH and percentage extract had less important effects on the glue joint strength than wettability and specific gravity.

Kawamura (30) reports on the influence of extracts from kapur wood (*Dryobalonops lanceolata*) in the curing of unsaturated polyester resin varnish. Certain portions of the extractives were shown to impede the polymerization of the

polyester resin varnish. On the other hand, other portions of the kapur wood extractives had no inhibitory effect.

Sakuno and Goto (40) in their study of thirty six species report that specific glue joint strength was significantly correlated with percent ether extract, for urea-formaldehyde, for woods of specific gravity 0.8 and less. There was no significant correlation (at 5 percent level of significance) between glue joint strength and ether extracts percent for wood species with specific gravity higher than 0.8.

Imamura *et al.* (26) studied the effect of wood extractives on gluing and coating of kapur wood (*Dryobalanops* spp.). They also noted that, n-hexane and ether-soluble extractives inhibited the gluing ability of phenolic resin. Further, the extractives inhibited the curing of unsaturated polyester resin varnish, as well. As already noted by Kawamura (30), the inhibiting effect was shown by only certain portions of the extractives.

Onishi and Goto (37) investigated the influence of water (hot and cold) and alcohol-benzene soluble wood extractives on the gelation time of urea-formaldehyde and the compressive strength of setting material. The gelation time of urea-formaldehyde, to which wood extractives were added, increased with the increased amount of hot and cold water-soluble extractives. For cold water extractives the gelation time decreases with decrease in pH. Alcohol-benzene soluble

extractives also affected the gelation time of urea-
formaldehyde, though to a lesser extent than water extract.

Wellons and Krahmer (47) analyzed a random assortment of
delaminated exterior hardwood plywoods with the aim of deter-
mining the causes for delamination. They state that there
are several factors that could cause such delamination. If
the adhesive failed to wet and penetrate the wood, a weak and
less durable glue bond would be formed. Also, the adhesive
may have bonded to a thin layer of extractives that later
could be leached from the glueline, hence the delamination.
Weak gluelines might also occur if the extractives at the
wood surface dissolve into the adhesive and either accelerate
its cure or make it too viscous to penetrate the wood
structure.

Anaike *et al.* (3) report on the inhibitory effect of
kapur wood extracts on the gelation of the urea resin
adhesives. Water extracts were more efficient in retarding
the gelation of urea and urea-melamine resins followed by
methanol extracts. They delayed the gelation of the urea
resin more effectively than that of the urea-melamine resin.
Ether and n-hexane extracts had little or no inhibitory
effect.

Jain *et al.* (28) observed that extractives play a very
important role in gluing of *Shorea robusta* (sal) with urea-
formaldehyde and phenol-formaldehyde resins.

The inhibitory effect of *Taxus mairei* heartwood
extractives on the curing of the unsaturated polyester resin

has been reported by Lee *et al.* (27). The methanol extract itself and the ether-soluble, the water-soluble and the water-insoluble fractions derived from the methanol extract showed an inhibitory effect on the curing of unsaturated polyester resins, but the n-hexane soluble fraction did not.

Lee *et al.* (33) studied the inhibitory effect of *Diospyros* sp heartwood extractives on the curing of the unsaturated polyester resin. The methanol extract showed an inhibitory effect on the curing of unsaturated polyester resins. The methanol extract was fractionated into several fractions. Among these fractions, n-hexane soluble acid, ether-soluble acid and ether-soluble phenolic fractions showed an inhibitory effect but n-hexane-soluble neutral, ether-soluble neutral, water-soluble and water-insoluble fractions did not.

Abe and Akimoto (1) investigated the insufficient gluability of kapur wood by studying the effect of the curing reaction of wood extractives on the resol type phenolic resin. The strong acid and weak acid fractions of the ether soluble part and the ether-insoluble part of the ethanol-benzene extractives showed strong inhibitory effects on curing of the resol resin. However, the ether-soluble neutral fraction showed no or lesser effect. Raising the temperature during curing tended to reduce the inhibition.

Kanazawa *et al.* (29) reported on the poor gluability of teak wood with urea resin adhesives. They investigated the effects of hot-water extractives on the curing reaction of

the adhesive. The hot water extract, especially its methanol insoluble part, was found to retard the gelation of the urea resin adhesive.

Chow and Chunsi (16) examined adhesion strength and wood failure relationships in wood-glue bonds of six Burmeses hardwoods. Phenol-resorcinol formaldehyde, urea formaldehyde and casein adhesives were used. They concluded that glue-joint shear strength had no significant relationship with the extractive content of substrate woods. This finding is essentially the same as that of Goto et al. (20).

Abe and Ono (2) reported on the effect of acidity of some tropical wood extractives on the curing of resol. Extractives from kapur and apitong wood were used in the study. The results demonstrated that the acidity of the extractives interferes with the curing rate of resol resins.

Yatagai and Takahashi (48) examined the effect of wood extractives, from 70 tropical wood species, on curing time of unsaturated polyester resin varnish. Ethyl ether, n-hexane, acetone and methanol were used as solvents for removal of the extractives. The acetone extractives had no influence on the varnish curing time. The curing time of the varnish was prolonged by n-hexane extractives.

Slay et al. (45) investigated the catalytic effects of extractives from pressure-refined fibers on the gel time of urea-formaldehyde resin. Six species of wood, black Tupelo, hickory, white oak, red oak, sweet-gum and loblolly pine were used in the study. Their findings indicate that the gel time

of the UF resin was affected by addition of small amounts of extractives. The gel time of the resin-extractive mixture increased exponentially as its pH increased.

2.2 Effects of Chemical Treatment of Wood Surfaces Prior to Gluing

Rapp (45)[2] investigated the possibility of using various surface treatments to improve the gluability of lignum vitae (*Guaiacum officinale*) by removal of at least part of the resinous extractives contained in lignum vitae wood. Among the treatments applied to normal machine-planed lumber were solvents such as carbon tetrachloride, benzene, acetone and alcohol. The most successful treatment was an application of 10% caustic soda solution, wiped on the surface, allowed to remain for 10 minutes, and removed by washing with water. A resorcinol resin was employed. The average results are shown below:

	Shear strength (psi)	Wood failure (%)
Control (untreated)	1185 (8,170 kPa)	22
Washed with NaOH	1770 (12,204 kPa)	30
Sanded; washed with NaOH	2000 (13,790 kPa)	37

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[2] Original not seen. Cited from Troop and Wangaard (45).

In one series of tests the results obtained from the combination of sanding and caustic soda treatment averaged 2760 psi (19,030 kPa) with 56% wood failure.

In a study carried out by Gamble Bros., Inc. (45)[3] it was shown that washing of teak (*Tectona grandis*) surface (contains an oily extractive) with acetone prior to gluing with a resorcinol adhesive, improved the joint shear strength and increased wood failure. By means of this technique joint strength was increased from 1025 to 1262 psi (7,067-8,701 kPa) and wood failure from 63 to 83% in comparison with untreated control specimens.

Troop and Wangaard (45) concluded that Burma teak does not necessarily require preliminary treatment before gluing with resorcinol adhesive. The joints had an average shear-strength of 2040 psi (14,065 kPa) and 92% wood failure.

In a study done at the Forest Products Laboratory (Madison (4)) it was found that surface treatment with 10% caustic soda improved the gluability of certain American hardwoods. The entire group of treated joints (13 species) when glued with animal glue showed 51% greater average strength and 97% more wood failure than the untreated joints of the same species glued under starved-joint conditions. A similar trend was observed with casein glue. However, one species (osage-orange) gave striking results. Osage-orange contains a large amount of extractives and is very difficult

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[3] Original not seen. Cited from Troop and Wangaard (45).

to join with casein glue. Untreated osage-orange wood showed practically no adhesion; the joints showed an average shear strength of only 294 psi (2027 kPa) and no wood failure. After treatment with caustic soda, the average joint strength was over 3000 psi (20,684 kPa) and the wood failure was 35%.

Knight, reported by Hancock (22), indicates that it was at one time common practice to wipe the surfaces of many hardwoods with a solution of 10% sodium hydroxide, when using animal glue, in order to improve bond strength.

Narayanamurti (35) noted that the effect of extractives on wood gluing is of special importance. He further noted that improvement of gluability of heartwood after extraction was evident.

Thomas (44) has shown that removal of a large part of the ether and benzene-soluble portion of the wax present in the tropical wood of determa (*Ocotea rubra* Mez), caused a considerable increase in the gluebond quality obtained with a phenolic resin. The removal of the extractives caused in change in the bond quality with melamine-formaldehyde as the adhesive. He concluded that a chemical incompatibility exists between phenol-formaldehyde and the wax-like substance in determa and that with the wax removed, a satisfactory joint can be obtained with phenol-formaldehyde.

Hancock (22) showed that the reduced adhesion of oven-dried Douglas-fir veneer is primarily the result of extractive migration to the surface. He also determined that the

acetone or a combination of acetone and methanol/benzene extractable fractions were responsible for the inhibition.

Wangaard and Granados (46) investigated the effect of extractives on water-vapor sorption by wood. Nine tropical hardwoods ranging from 3 to 17% in extractive content were used. They found out that there was no significant relationship between total sorption and accessibility of bonding sites for either high or low extractive content material.

Bryant (14) studied the effect of various chromium complexes (pH 1.95 - 4.05). Douglas-fir veneer surfaces were modified for the purpose of improving the bond quality of the plywood. The treatment of veneers with chromium complex solutions impaired the glue-bond quality rather than improving it. Few of the plywood panels made with treated veneers compared favorably with the controls. He postulated that the high pH of the typical phenolic adhesive mix used may have rendered the chromium complexes ineffective as bonding agents, and may have had a deleterious effect on the adhesive itself as judged by the increase in viscosity. When a urea resin was used the results were more encouraging due to better pH compatibility.

Chen (15) studied the effect of extractive removal on adhesion and wettability of some tropical hardwoods. Machined wood surfaces of eight tropical species were treated with a 10% solution of sodium hydroxide, acetone, and alcohol-benzene. After reconditioning, wood blocks were glued with urea-formaldehyde and resorcinol-formaldehyde

resin adhesives. Untreated wood blocks were used as controls. The results indicated that the adhesive joint strength was improved by all the treatments in all the species, except one. Extractive removal treatment improved wettability and increased pH of wood in all the species tested.

Jain *et al.* (27) studied the effect of extractives on gluing of *Pinus roxburghii* (Chir) from India. Veneers were subjected to extraction with hot water, ether, alcohol-benzene, 1% NaOH solution individually and also successively. Plywood panels were made using the extracted and unextracted (control) veneers. Urea-formaldehyde (cold-set and hot-set) and phenol-formaldehyde (water-soluble and alcohol-soluble) adhesives were used. The results showed that, in case of cold-set UF glue, an improvement in dry-strength occurred in case of ether, 1% NaOH and extractive free samples. However, it is only in the case of ether-extracted samples that an improvement in boil test strength was noticed. With hot-set UF, definite improvements in case of hot water and ether extracted samples were observed in dry and boil-test values but the results were better with hot water extracted samples. Results of gluing with PF (water-soluble) indicated that alcohol-benzene extracted samples and ether extracted samples glued better than the controls. In the case of PF (alcohol-soluble) the removal of extractives seemed to abate gluability.

Roffael and Rauch (38) investigated the influence of extractives on the gluing of oak particles with alkaline phenol-formaldehyde resin. The results showed that extractives from old oak trees are more acidic and exhibit a higher buffering capacity than those from younger trees. In general, extraction of oak particles with boiling water favourably affected the gluability of the particles. Extraction with 1-n sodium carbonate solution instead of water brought about a further improvement in the gluability of oak using alkaline PF resin. Addition of sodium hydroxide (3% related to dry wood) considerably improved the gluability of oak particles.

Dougal *et al.* (18) examined the glueline characteristics and bond durability of southeast Asian species after solvent extraction and planing of veneers. Veneers of five wood species were either planed, extracted with a one percent caustic solution, or left untreated before gluing. Two types of phenolic adhesives were used. One of their conclusions was that, one percent caustic extraction for 60 seconds, followed by a 60-second rinse before gluing, darkened the veneer surfaces and did not consistently increase the percentage of wood failure.

In 1983, Kelly *et al.* (31) investigated the effects of five surface activating reagents on the shear strength of bonded solid wood panels: nitric acid, sulfuric acid, hydrogen peroxide, potassium persulfate and potassium periodate. Five different wood species were used in this

study: three hardwoods and two softwoods. No wood adhesive was used, the reagents acted as catalysts for activating bridging materials such as lignin and hemicelluloses. The results demonstrated that solid wood panels can be bonded by such activation of the wood surface. However, the shear strength of the products was highly dependent on the surface activating reagent and wood species used. Prolonged nitric acid activation was shown to degrade the wood substrate.

In summary, wood extractives, and particularly the acidic extractives, were shown in the past to seriously interfere with the setting of adhesives. The inhibitory effect was most pronounced on casein, phenol (resorcinol) - formaldehyde and least on urea-formaldehyde.

Neutralization with alkali solutions (1-3%) or removal with suitable solvents was necessary to obtain acceptably strong glue bonds. The extent of the inhibitory effect was shown to be proportional to the concentration of acidic extractives with even relatively low concentrations being also effective.

3.0 MATERIALS AND METHODS

3.1 Wood Samples

Air-dry samples of camphor wood, *Ocotea usambarensis,*
in form of sawn lumber with dimensions of 25.4 mm x 101.6 mm
x 609.6 mm (1 in x 4 in x 24 in) were obtained from three
separate timber yards in Nairobi, Kenya. Thus, the samples
should represent the camphor wood population used in the
furniture and joinery industry in Kenya.

Some of the boards were quarter sawn while others were
flat sawn. The samples, on arrival in Vancouver, were
stacked with 12.7 mm (0.5 in) spacers between, in a 50 ± 2%
relative humidity room [dry bulb 23 ± 1°C (73.4 ± 2°F) having
an air speed approximately 70 m/min (200 ft/min)] to permit
controlled conditioning.

3.2 Wood Adhesives

Four room-temperature curing glues were used in this
study. The casein, phenol-resorcinol-formaldehyde (PRF) and
urea- formaldehyde (UF) resins were supplied by Borden
Chemical Company (Canada) Limited. The polyvinyl-acetate
(PVA) was supplied by Crown Paints and Building Products
(Kenya) Limited.

3.2.1 Casein

Casco-casein 42-28G is a premium grade glue that is
readily mixed with water. It is room curing and is

recommended for interior use. It complies with the type III glue bond requirements of Canadian Standard Association (CSA) 0132.2 M 1977 (8).

3.2.2 Phenol-resorcinol-formaldehyde

Cascophen LT-75 is a liquid resin which when mixed with a dry powdered hardener, catalyst FM-282C, produces a phenol-resorcinol-formaldehyde adhesive that is room temperature setting. Tests in Borden laboratories indicate that representative lots of this adhesive meet CSA standard 0112.7-1960 (Type 1) specification for wet use service. It produces a waterproof and weatherproof bond with wood (8).

3.2.3 Urea-formaldehyde

Casco UF 109 with 221 hardener is a cold-setting urea-formaldehyde glue that cures to a highly water-resistant bond. It meets the requirements of CSA 0112.5-M1977 for type 1 urea resin adhesives (7).

3.2.4 Polyvinyl-acetate

Polyvinyl resin emulsions are marketed as milky-white fluids to be used at room temperature in the form supplied by the manufacturer, normally without addition of separate hardeners. The adhesive sets when the water of the emulsion partially diffuses into the wood and the emulsified resin coagulates. There is no apparent chemical curing reaction associated with the hardening of this adhesive (41).

The resin emulsions tend to creep when joints are subjected to loads at temperatures above 49°C (120°F) for sustained periods, and possess only fair water and moisture resistance (19).

3.3 Experimental Design

The experiment was carried out using a completely randomized design. The design of the experiment included two variables, glue types and wood. The design used was as indicated below:

Variables	Levels
Glues	Four – PVA – Casein – UF – PRF
Wood	Five – unextracted wood (control) – extracted with hot water – surface treated with dilute nitric acid – surface treated with 10% sodium hydroxide solution – extracted with alcohol-benzene.
Replicates (assemblies)	Three – (for PVA) Three – (for casein) Six – (for UF) Nine – (for PRF).
Duplicates (shear blocks/assembly)	Five
Total combinations (shear blocks)	405[4]

The tests were carried out as indicated in Table 1.

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[4] Only one wood level (unextracted wood) was used with UF glue.

3.4 Experimental Procedure

3.4.1 Specific gravity determination

Specific gravity was determined by obtaining green and oven-dry volume of four specimens, each 25 mm x 25 mm x 77 mm (1 in x 1 in x 3 in) in size.

The specimens were soaked in a steel cylinder filled with water for 4 days, at 827 kPa (120 psi) to make sure they were water saturated. The length, width and thickness dimensions, of the water saturated specimens, were taken at 3 different points, the ends and the center of the specimen. Green weight and volume of each specimen was determined.

The specimens were then oven-dried at 103 ± 2°C to constant weight. The basic (green) specific gravity of each of the specimen was calculated as follows:

$$\text{Green specific gravity} = \left(\frac{\text{O.D weight}}{\text{Green volume}}\right) / \text{Density of water}$$

Where: O.D weight = weight of oven-dry specimen.

Green volume = volume of water saturated specimen.

The average value of the four specimens was adopted as the specific gravity of camphor wood.

3.4.2 Wood pH determination

The sawdust generated during the initial cutting of the boards, before gluing, was used for pH determination.

Five grams of sawdust was mixed with 50 cm^3 of distilled water. The mixture was allowed to stand for 2 h with occasional stirring. Then the mixture was filtered and the filtrate used for pH determination using a standard pH meter.

Average pH value of five samples (wood sawdust and distilled water) was taken as the pH of camphor wood at room temperature.

3.4.3 Total extractives content determination

Again, sawdust generated during the cutting of the boards before gluing provided the wood powder that was used in this part of the study. Three solvents: alcohol-benzene, water, and 1% sodium hydroxide were used. The procedure adopted by ASTM (see Appendix III) was followed when conducting this test.

3.5 Preliminary Experiments

To determine the best conditions for gluing camphor wood, some preliminary experiments were conducted. Camphor wood blocks approximately 25.4 mm x 101.6 mm x 304.8 mm (1 in x 4 in x 12 in) were used in this part of the study. The blocks were planed down to a thickness of 19 mm (3/4 in) just prior to gluing. Using a double glue spread of 410.6 g/m^2 (85 lb per 1000 ft^2) and the recommended assembly times of each of the 4 glues, the following gluing conditions were tested:

(i)   Gluing and pressing at room temperature, 21-23°C,

for 24 h using a pressure of 1,379 kPa (200 psi).

(ii)   Gluing and pressing at room temperature, 21-23°C,

for 24 h using under 1,724 kPa (250 psi).

For each glue, three glued stocks were made. Five

modified (see Figure 11) test specimens were cut from each of

the glued blocks. The specimens were tested in accordance

with Sections 8 and 9 of the ASTM standard, D905-81 (Appendix

II).

The first set of conditions gave satisfactory results

and was adopted for the main experiment.

3.6   Treating of Wood Samples Prior to Gluing

Five different groups of camphor wood samples were used

in this study. Each group of samples was randomly assigned

one of the following treatments:

(i)   extraction with alcohol-benzene;

(ii)   extraction with hot water;

(iii) surface treatment with 3% nitric acid;

(iv)   surface treatment with 10% sodium hydroxide

solution;

(v)   no treatment (control).

3.6.1 Extraction with alcohol-benzene

For this treatment a solvent consisting of one volume of

benzene and two volumes of 95% ethanol was used. The wood

samples consisted of machine-planed pieces of 25.4 mm x 101.6

mm x 304.8 mm (1 in x 4 in x 12 in) in size. The samples
were placed in a glass tank after which the tank was filled
with alcohol-benzene solution. The tank was covered with a
glass pane and silicon grease was applied all around to
ensure that the set up was airtight. The samples were
allowed to stand for 6 days, at room temperature (21-24°C),
with two renewals of the alcohol-benzene solvent. After six
days the alcohol-benzene solution was replaced with methanol
and the samples were allowed to stand for 24 h. The methanol
was used to extract the alcohol-benzene solvent from the wood
samples.

Then the samples were conditioned until most or all of
the two solvents mentioned above, had evaporated. One face
of each sample was lightly planed [1.6 mm (1/16th in)
removed] to remove any extraneous materials that might have
migrated from the core to the surface of the sample during
the conditioning period. After this, the samples were planed
down to a thickness of 19 mm (3/4 in). The gluing, cutting
of test specimens and testing was carried out in accordance
with the method outlined earlier.

3.6.2 Extraction with hot water

Machine-planed camphor wood samples, 25.4 mm x 101.6 mm
x 609.6 mm (1 in x 4 in x 24 in) were used. The samples were
placed in a water bath containing cold water and allowed to
soak for 24 h. Extraction was carried out at 97 ± 2°C for
48 h, with two changes of water. At the end of this

treatment the samples were conditioned for one week in a conditioning chamber set at 80% relative humidity [dry bulb 49 ± 1°C (120 ± 2°F)]. This step was aimed at eliminating/reducing any migration of extractives from the core to the wood surface, which usually occurs due to fast drying of green lumber.

The wood samples were conditioned at 50% [dry bulb 23 ± 1°C (73.4 ± 2°F)] relative humidity until the desirable equilibrium moisture content was attained. Conditioning was followed by planing the samples to a thickness of 19 mm (3/4 in) and cross-cutting into 304.8 mm (12 in) pieces. The gluing, cutting of test specimens and testing was done following the procedure outlined in the preceding section(s).

3.6.3 Surface treatment with nitric acid

Camphor wood samples were machine-planed and cut into suitable size, approximately 19 mm x 101.6 mm x 304.8 mm (3/4 in x 4 in x 12 in). A three percent nitric acid solution was brushed onto the surfaces that were to be joined. These surfaces were then heat-treated under an infra-red source for 30 min. After the treatment the board surfaces were neutralized by wetting with distilled water followed by exposing the wet surface to ammonia vapour for five minutes.

The samples were then conditioned again to the desirable equilibrium moisture content in a controlled temperature and humidity (C.T.H.) room. Gluing, cutting of test specimens,

and testing was carried out in accordance with the method outlined earlier on.

3.6.4 Surface treatment with sodium hydroxide solution

The wood samples were machine-planed and cut to size, approximately 19 mm x 101.6 mm x 304.8 mm (3/4 in x 4 in x 12 in). The wood surfaces, to be joined, were wiped with a piece of cloth saturated with sodium hydroxide solution consisting of 10 parts by weight of sodium hydroxide (caustic soda) and 90 parts of water. After 10 minutes the surfaces were rinsed with distilled water and then wiped dry with a clean white cloth until they showed no discoloration. Following this step, the treated samples were conditioned to the desired moisture content. Gluing, cutting of test specimens, and testing was carried out in accordance with the method described above.

3.7 Main Experiment

From the results obtained in the preliminary experiment the following conditions were used for all the four glues:

(i)   A glue spread of 410.6 g/m^2 (85 lbs per 1000 ft^2).

(ii) Gluing and pressing at room temperature (21-24°C).

(iii) Press pressure of 1,379 kPa (200 psi).

(iv) Pressing time of 24 h.

Camphor wood samples which were of straight grain and free from defects such as knots, birds eyes, short grain, decay and unusual discolorations within the shearing area

were used. The grain direction was parallel to the longest dimension of the block.

The blocks were planed just prior to gluing and assembled in pairs in such a way that blocks of approximately the same specific gravity (air dry) were glued together. The surfaces of the blocks remained unsanded and free from dirt.

The moisture content of the wood at the time of gluing was 9-12% based on oven-dry weight as determined by use of representative samples.

3.7.1 Adhesive application, pressing and conditioning of test joints

Each glue was mixed according to the manufacturer's instructions and used within the recommended time (pot life). A glue spread of 410.6 g/m^2 (85 pounds per 1,000 sq. ft.) of joint area double spread was used. The weighed amount of adhesive was spread manually with a rubber roller. The formulation of the glues and gluing conditions used are shown in Table 2.

The glue-coated blocks were then assembled and pressed at room temperature (21-23°C) with a pressure of 1,379 kPa (200 psi) for 24 h. The blocks, upon removal from the press, were conditioned at a relative humidity 50 ± 2% and at a temperature of 23 ± 1°C (73.4 ± 2°F) for a period of 7 days, after which they were cut into test specimens.

3.7.2 Preparation of block shear specimens

Due to the limited amount and width of camphor wood material which was available to conduct this study, a slight modification of the test specimen size was required. The camphor lumber had a nominal width of 101.6 mm (4in).

Instead of the ASTM standard block of 44.4 mm x 50.8 mm x 12 mm (1 3/4 in x 2 in x 3/4 in) a modified specimen, 44.4 mm x 44.4 mm x 12 mm (1 3/4 in x 1 3/4 in x 3/4 in) was adopted for this study. The 6.4 mm (1/4 in) reduction in width was checked to be of little significance to the validity of the test results.

Stickler (43), in a study on block shear specimen geometry, concluded that specimen width has no significant effect on unit shear strength. On the other hand, specimen length had a significant effect on unit shear strength. A length of shear plane parallel to grain of 1 inch, he observed, is due to optimum dimension of the block shear specimen.

The block shear specimens were cut in such a way that the grain direction was parallel to the direction of loading during test. Surfaces were smooth, parallel to each other and perpendicular to the height. The width and length of each specimen at the glueline was measured to the nearest 0.25 mm (0.01 in) and the shear area calculated.

The specimens were left in the conditioning atmosphere described in Section 3.7.1 until tested, except during the

cutting operations. The cold soak and boil test specimens were treated before they were tested.

3.7.3 Testing procedure

The block shear specimens used in this study had shear area varying between 1677.4 and 1806.4 mm^2 (2.6 and 2.8 sq. in.) instead of the 1935.5 mm^2 (3 sq. in.) of the ASTM (D905-81) standard block shear specimen.

A Tinus Olsen hydraulic testing machine, fitted with a shearing tool containing a self-aligning seat to ensure uniform distribution of load, was used (Fig. 12). The load was applied at a uniform rate, with a continuous motion of the movable head at a rate of 6.35×10^{-3} mm/s (0.015 in/min) to failure.

3.7.3.1 Dry test

The dry blocks were tested following the procedure outlined above. Shear stress at failure for each test block was recorded and the shear strength calculated. The shear strength at failure in kilopascals (kPa) was based on the glueline area between the two laminations measured to the nearest 0.25 mm^2 (0.01 in^2), and rounded. Estimated percentage of wood failure was recorded for each block.

3.7.3.2 Cold soak test

A portion (see Table 2) of the test specimens glued with phenol-resorcinol-formaldehyde and urea-formaldehyde were

soaked in cold water, at 21°C (70°F) to 24°C (75°F) for 48 h. While still wet, they were tested to failure in a similar method to that used for the dry specimens. Shear strength and percent wood failure of each specimen was recorded.

3.7.3.3 Boil test

The boil test was used only for the test specimens glued with phenol-resorcinol-formaldehyde. The test specimens were boiled in water for 4 h and then dried at a temperature of 60 ± 3°C (140 ± 5°F) for 20 h. Then the test specimens were boiled again in water for another 4 h, cooled in water at 21 ± 3°C (70 ± 5°F) and tested by loading to failure while they were still wet. Eventually, the percentage wood failure was estimated and the shear strength calculated.

3.8 Statistical Analysis

The average values of both the shear strength and wood failure were calculated. Shear strength and wood failure were analysed separately.

One way analysis of variance was performed for both the shear strength and wood failure according to type of test.

Where significant differences were noticed, Duncan's new multiple range test was used to separate the set of means into subsets of homogeneous means.

4.0 RESULTS AND DISCUSSION

4.1 Wood Characterization

The specific gravity (G) (at 12% m.c.) values, based on oven-dry weight and green volume, are in the range of 0.52 to 0.53, with a sample mean of 0.52 and a standard deviation of 0.005.

This mean G of 0.52 ± 0.005 obtained for camphor wood samples used in this study, compares well to a value of 0.59 reported by Bengough (11) (Appendix I).

pH values, of five samples, are in the range of 2.89 to 2.96. The average pH value is 2.93 and with a standard deviation of 0.026. This indicates that camphor wood is strongly acidic.

The results of total extractive content determination are summarized in the Table 3.

Weight percentage of camphor wood matter, soluble in hot one percent caustic soda solution (NaOH) is 24.1, on the moisture-free basis. This value is an average of three determinations.

These results indicate that a high proportion of camphor wood extractives are soluble in alcohol-benzene, hot water and cold water. It should be pointed out that these values are not from successive extractions. Also these results were obtained from a small set of samples and do not therefore represent the quantity of extractives in every sample of camphor wood species, since the distribution of extractives

is expected to vary considerably within a single tree and among trees.

4.2   Gluing with Polyvinyl-acetate (PVA) Adhesive

  4.2.1   Dry Test: Bond shear strength and wood failure
                      percent

The average shear strength and percentage wood failure are shown in Tables 4a and 4b. The standard deviation, minimum, maximum and range for both shear strength and percentage wood failure are also presented. These values are the average of 15 measurements per treatment.

Figure 1 represents a histogram of the average shear strength and percentage wood failure. As is evident from Tables 4a,b and Figure 1, the blocks made with wood that had been surface treated with dilute nitric acid, and neutralized with ammonia gas produced the highest average shear strength (14,692 kPa); while those made from wood, surface treated with 10% sodium hydroxide solution showed the highest percentage wood failure (70%).

Alcohol-benzene extracted wood showed the lowest values for both the average shear strength (7,452 kPa) and percentage wood failure (6%).

One way analysis of variance for shear strength and wood failure are shown in Table 5.

The statistical ranking of the means, by Duncan's multiple range test, for both the shear strength and wood failure are presented in Table 6.

The analysis of variance results for shear strength (Table 5a) indicate that the treatment means are significantly different (i.e., at 0.01 level of significance). Duncan's multiple range test (Table 6) ranks unextracted, hot water extracted, and sodium hydroxide treated wood as having similar average bond shear strength. Samples surface treated with nitric acid had the highest bond shear strength while alcohol-benzene extracted had the lowest. The average bond shear strength of nitric acid treated wood is significantly higher than that of the unextracted wood (control). On the basis of average bond shear strength it is evident that surface treating the wood with nitric acid, prior to gluing, improved its gluability with PVA Treating the wood with sodium hydroxide or extracting it with hot water, prior to gluing, does not improve its gluability with PVA. Extracting the wood with alcohol-benzene, prior to gluing, resulted in an inferior joint when compared to the unextracted wood (control).

The analysis of variance results (wood failure) shown in Table 5b indicate that the treatment means are not all equal. The wood failure ranking in Table 6 shows that the highest percentage wood failure (70%) obtained with sodium hydroxide treated wood is not significantly different at 0.05 level from the 56% value of the control. The percentage wood failure given by the nitric acid treatment (49%) is not significantly, at 0.05 level, different from the 56% exhibited by the untreated wood. However, it (nitric acid

treatment) is significantly different from the 70% of the sodium hydroxide treated wood. The percentage wood failure showed by the hot water extracted wood (18%) and alcohol-benzene extracted wood (6%) are not significantly different (at 0.05 level) but both of them are significantly lower than those of the other three treatments. On the basis of average percentage wood failure none of the four wood pre-treatments significantly improved the gluability of camphor wood when glued with PVA adhesive.

These results are not very much different from those obtained by Goto *et al.* (20). They observed that the relationship between glue-joint strength and percentage of either cold or hot water soluble extractives is not significant. The results obtained in this study suggest that extractives removal, using hot water, sodium hydroxide solution or alcohol-benzene does not improve glue-joint strength.

At the same time the results of this study are in contradiction with those reported by Chugg *et al.* (17), Wellons *et al.* (47), Abe *et al.* (1). Generally results from their studies suggest that extractives affect the formation of a glue bond either physically, chemically or both physically and chemically.

4.3 Gluing with Casein Adhesive

   4.3.1 Dry Test: Bond shear strength and wood failure
                      percent

Tables 7a and 7b summarize the average shear strength

and percentage wood failure for the wood blocks bonded with

casein adhesive. The standard deviation, minimum, maximum

and range values are also presented in the tables. Figure 2

also depicts average bond shear strength and percentage wood

failure for the five treatment combinations of wood and

casein adhesive.

Blocks made from wood that had been treated with sodium

hydroxide solution, produced the highest average bond shear

strength of 14,297 kPa; while those made from nitric acid

treated wood gave the lowest, 5,911 kPa. The blocks made

from untreated wood (control) had the second lowest average

bond shear strength.

As shown in Table 7b, blocks made from hot water

extracted wood gave the highest percentage wood failure

(78%). No wood failure was noticed in the blocks made from

wood that had been surface treated with nitric acid prior to

gluing. The blocks made from alcohol-benzene extracted wood

and sodium hydroxide treated wood exhibited percentage wood

failures of 51% and 45%, respectively; these values are

higher than that of the control (15%).

The analysis of variance tables for average shear

strength and wood failure developed are presented in Tables

8a and b. From the analysis of variance results it is

evident that the shear strength treatment means are significantly different at 0.01 level.

Duncan's Multiple Range test (Table 9) ranks the sodium hydroxide, and alcohol-benzene treatment means for shear strength development in the same group. This means that although the treatment mean of sodium hydroxide treated wood (14,297 kPa) is higher than that of alcohol-benzene treated wood (12,955 kPa) the two means are not significantly different, at 0.05 level. The range test further indicates that the average shear strengths for blocks made with alcohol-benzene extracted wood, and hot water extracted wood are not significantly different, at 0.05 level. However, the hot water treatment mean is significantly different (at 0.05 level) from that of sodium hydroxide treatment. The control and hot water treatment means are not significantly different, at 0.05 level. The nitric acid treatment mean (5,911 kPa) is the lowest and is also significantly different from the other four treatment means. From the shear strength results it is evident that surface treating of camphor wood with sodium hydroxide, and extracting with alcohol-benzene prior to gluing improves its gluability with casein adhesive. Adhesion in camphor wood is not significantly improved by extracting the wood with hot water i.e., prior to the gluing operation. It seems that surface treating of camphor wood with nitric acid, prior to gluing, lowers its gluability with casein adhesive.

As indicated by analysis of variance results (Table 8a), for percentage wood failure, the treatment means are not significantly equal. As shown in Table 8, hot water treatment mean (78%) is the highest and also significantly higher than the others. Percentage wood failures of 51% and 45% produced by sodium hydroxide, and alcohol-benzene treatments, respectively, are not significantly different at the 0.05 level. The 15% wood failure exhibited by the control is not significantly different from the lowest percentage wood failure (0%) produced by the nitric acid treatment.

In view of the percentage wood failure results, it can be stated that gluability of camphor wood with casein adhesive was improved by surface treating it with sodium hydroxide, extracting it with hot water and alcohol-benzene prior to gluing. On the other hand, surface treating with nitric acid had a negative effect on adhesion.

These results are quite similar to those obtained by other workers. In tests done at Forest Products Laboratory, Madison (4), it was shown that when wood surfaces were treated with chemical solutions before gluing, the quality of the joints was improved on several species with animal and casein glues. Treatment with a solution of caustic soda (sodium hydroxide) strengthened casein glue joints in woods that ordinarily are joined with this glue with some difficulty. This type of treatment increased the joint strength as well as the wood failure. A similar trend was observed in this study.

According to Knight (22)[5], it was at one time common practice to wipe the surfaces of many hardwoods with 10% sodium hydroxide, before gluing with animal glue, in order to improve bond strength.

Narayanamurti *et al.* (36) found that hot water extractives of *Tectona grandis*, in low concentrations (2%), slightly increase the modulus of rigidity and reduce the gelation time of animal glue nearly to half. They also noted that higher concentrations reduce the rigidity modulus by about 11% but increase the gelation time by over 200%; these two aspects give rise to a starved joint which exhibits low strength and low percentage of wood failure. This probably explains the results, of the hot water treatment, obtained in this study i.e., an increase in both bond strength and wood failure with removal of hot water extractives.

Chugg and Gray (17) have indicated that extractives from *Afrormosia elata* inhibit the setting of animal glue. The same authors report that extractives lower the surface tension of the wood surface and reduce wettability, which is essential for a good glue bond.

On the other hand, some of the results obtained in this study are in contradiction with some results obtained by other researchers while, investigating gluing of tropical hardwoods. Goto *et al.* (20) point out that the relationship between glue-joint strength and percentage of either cold or

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[5] Original not seen. Cited from Hancock (22).

hot water soluble extractives is not significant. Chow and
Chunsi (16), in their study of adhesion strength of six
Burmese hardwoods, concluded that glue-joint shear strength
had no significant relationship with the extractive content.

4.4 Gluing with Phenol-Resorcinol-Formaldehyde Adhesive

4.4.1 Dry test: Bond shear strength and wood failure percent

The results of bond strength and percentage wood failure
are shown in Tables 10a and 10b. The relationship between
bond strength and percentage wood failure is shown in Figure
3. Both, in the case of alcohol-benzene extracted (14,543
kPa) as well as that of sodium hydroxide treated wood (14,629
kPa), the bond strength is higher than that of the control.
On the other hand, that of hot water extracted (10,411 kPa),
and nitric acid treated wood (9,709 kPa) is lower than that
of the control (12,924 kPa). Again, nitric acid treatment
produced the lowest average bond strength.

As can be seen in Table 10b, the percentage wood failure
for alcohol-benzene extracted wood is the highest (98%). No
wood failure was observed in the blocks made with nitric acid
treated wood. Blocks made with sodium hydroxide treated
wood, and hot water extracted wood showed percentage wood
failures of 75% and 60%, respectively. These two values, as
well as that for alcohol-benzene extracted wood are higher
than that shown by the control (30%).

In Table 16, the statistical ranking of the shear strength, treatment means, by Duncan's Multiple Range test puts alcohol-benzene extracted wood, and sodium hydroxide treated wood in the same group. These two treatment means are significantly higher than that of the control. The average shear strengths of blocks made with hot water extracted wood, and nitric acid treated wood are not significantly different, at 0.05 level. However, both of them are significantly lower than that of the control.

On average bond shear strength basis, it can be stated that, extracting camphor wood using alcohol-benzene or surface treating it with sodium hydroxide, prior to gluing, improves its gluability with phenol-resorcinol formaldehyde adhesive. Surface treating the wood with nitric acid or extracting it with hot water, prior to gluing, does not improve its gluability with phenol-resorcinol formaldehyde adhesive.

As indicated by analysis of variance results (Table 11a) the percentage wood failure means are significantly different, at 0.05 level. Only the sodium hydroxide, and hot water treatment means are not significantly different, at 0.05 level (Table 11a). Also from the ranking of the means shown in Table 16, it is evident that the treatment means for alcohol-benzene extraction, hot water extraction, and sodium hydroxide treatment are significantly higher than that of the control. On the other hand, the nitric acid treatment mean is significantly lower than that of the control. Thus, on

wood failure basis, it can be stated that benzene, hot water, and sodium hydroxide treatments enhanced adhesion in camphor wood. On the contrary, treating the wood with nitric acid, prior to gluing, lowers its gluability with phenol-resorcinol formaldehyde adhesive.

It is also important to note that the highest shear strength and wood failure were obtained for the alcohol-benzene extracted wood.

4.4.2 Cold soak test: Bond shear strength and wood failure percent

Results of the cold soak test, average shear strength and percentage wood failure, are shown in Tables 12a and 12b, respectively. The average shear strength, for the different treatments, in order of increasing magnitude is as follows: Nitric acid-treated wood (1321 kPa), hot water extracted wood (10,421 kPa), control (11,509 kPa), sodium hydroxide treated wood (12,708 kPa), and alcohol benzene extracted wood (14,609 kPa). Percentage wood failure for the cold soak test in the same order is nitric acid treated wood (0%), sodium hydroxide treated wood (42%), control (60%), hot water extracted wood (91%), and alcohol-benzene extracted wood (100%).

Analysis of variance results shown in Table 13a indicate that the bond shear strength means are not significantly equal at 0.05 level. Similarly this observation is also true for the wood failure percent (Table 13b).

Statistical ranking by average shear strength values (Table 16) indicates that none of the means is statistically

similar to any other. Alcohol-benzene and sodium hydroxide treatment means are significantly higher than the mean of the control. This is an indication that these two types of treatments improved gluability of camphor wood with phenol-resorcinol formaldehyde, as far as cold soak test is concerned. The hot water treatment appears to have lowered adhesion, although not to a very great extent. Blocks made with nitric acid treated wood developed an extremely low bond strength, about 9 times lower than that of the control.

Duncan's Multiple Range test (Table 16) ranks alcohol-benzene, and hot water treatments as having similar wood failure percent, the control is ranked third, sodium hydroxide treatment fourth and nitric acid treatment last.

These results clearly indicate that on the basis of wood failure percent, alcohol-benzene, and hot water treatments improved adhesion; while sodium hydroxide and nitric acid treatments had a negative effect on adhesion.

4.4.3 Boil test: Bond shear strength and wood failure
                  percent

The glueline shear strength for the six treatments in a decreasing order is alcohol-benzene (13,759 kPa), control (12,926 kPa), sodium hydroxide (10,103 kPa), hot water (9,920 kPa), and nitric acid (9,523 kPa) (Table 14a). Hot water, sodium hydroxide, and nitric acid treatments have shear strength values lower than that for the control. The alcohol-benzene treatment mean is higher (see Figure 5).

Table 14b presents the percentage wood failure results for the boil test. As can be seen from the results, blocks made with alcohol-benzene treated wood showed the greatest (100%) amount of wood failure, followed by those made from hot water extracted wood (87%). The control (56%) is placed third, sodium hydroxide treated wood fourth (45%), and nitric acid treated wood last with a 13 percent wood failure.

Analysis of variance results for both bond shear strength and percentage wood failure are shown in Tables 15a and b. These results indicate that the bond shear strength treatment means are significantly different at 0.05 level of significance; this is also the case for percentage wood failure treatment means.

The results for Duncan's Multiple Range test, average shear strength, and wood failure percent are shown in Table 16. The alcohol-benzene treatment and the control are ranked as having average shear strength values which are not significantly different. Sodium hydroxide, hot water, and nitric acid treatments are also ranked as being similar in average bond shear strength. These results seem to indicate that none of the four different treatments improved adhesion, as far as the boil test is concerned.

For wood failure percent, the range test puts alcohol-benzene and hot water treatments in the same group i.e., they are not significantly different at 0.05 level. Both of these treatments showed an average wood failure percent, which according to the range test, are significantly higher than

that for the control. The control and the sodium hydroxide
treatment means are classified as not significantly
different. The nitric acid treatment mean is significantly
lower than the other four means. For this particular test,
the results suggest that alcohol-benzene, and hot water
treatments improved bond quality (in terms of percentage wood
failure).

4.6 Comparison of Bond Strength Tests for Wood Blocks Bonded with Phenol-Resorcinol-Formaldehyde Adhesive

As is evident from Table 17, the bond shear strength
values of the dry test are, generally higher than those of
the cold soak or boil test. Blocks made from unextracted
wood lost 11% of their shear strength due to soaking in cold
water. When subjected to the boil test, blocks made from
unextracted wood gained about 0.01% shear strength i.e.,
taking the dry shear strength as the standard. The reduction
in strength observed in the cold test can be assumed to be a
result of the decrease in strength of the wood, due to high
moisture content at the time of testing. Most of the
strength properties and elastic characteristics of wood vary
inversely with the moisture content of the wood (13, 23).
The wood failure results support the above-mentioned
assumptions; it increased from 30% (dry test) to 60% for the
cold soak test. Thus, a reduction in shear strength of the
wood itself rather than a stronger glue joint.

However, the boil test gave some unexpected results in
that instead of the bond strength decreasing it actually

increased. Probably the increase in bond strength is due to a post-cure phenomenon of the adhesive as a result of raising the temperature (boiling). Although, the wood failure percent for the boil test is lower than that of the dry test, it is slightly higher than that for the cold soak test. Again this is contrary to what one would have expected. Due to thermal plasticization of wood by the hot water, the shear strength would be expected to decrease.

For the boil test the loss in shear strength is 5% for blocks made from hot water extracted wood. However, blocks made from hot water extracted wood showed slightly higher strength in the cold soak test than in the dry test. The wood failure values for the cold and boil tests are higher than that of the dry test.

Blocks made from wood that had been treated with nitric acid prior to gluing showed a major loss (86%) in strength when subjected to the cold soak test. For the boil test, blocks made with nitric acid treated wood showed a very slight loss (2%) in shear strength. The wood failure values were 0%, 0% and 13%, for the dry, cold soak test and boil test, respectively. Considering the shear strength and wood failure results, it can be stated that soaking of the test specimens in cold water prior to testing resulted in a degrade of the glue itself rather than the wood. Boiling the test specimens seems to have resulted in some weakening of the wood itself; hence the increase in the amount of wood failure.

Cold soaking and boiling of blocks made with wood that had been treated with sodium hydroxide solution resulted in a 13% and 31% drop in shear strength, respectively. Surprisingly, the wood failure values of the cold soak test and boil test were lower than that of the dry test. It seems that the drop in bond strength is probably the result of degradation that might have taken place in the glue itself rather than in the wood.

Blocks made with alcohol-benzene extracted wood had slightly higher (0.4%) strength in the cold soak test than found for the dry test specimen. The strength values for the boil test were lower than that obtained in the dry test. Alcohol-benzene treatment showed the highest amount of wood failure (dry test 98%, cold soak 100% and boil test 100%), when compared to the hot water, nitric acid, sodium hydroxide and the control treatments. The high amount of wood failure suggests that the wood-glue joint, in alcohol-benzene extracted wood, failed mainly as a result of the wood stress and that the adhesion inhibitory factors evident with other treatments and adhesive were removed by this solvent system most effectively.

4.6.1 General discussion on gluing camphor wood with phenol-resorcinol-formaldehyde adhesive

Over the years, attempts have been made by many researchers to determine the influence of extractives on gluability of wood with phenolic resins (15, 18, 22, 47). Some of the results from this study compare favourably with

their findings reported in the existing literature on adhesion in wood. Rapp (45)[6] showed that various surface treatments can be employed to improve adhesion in a tropical hardwood. Among the surface treatments used was wiping the wood surface, prior to gluing, with 10% caustic soda solution. The results indicated that both the shear strength and wood failure values were higher than those of the untreated wood. Gamble *et al.* (45)[7] have shown that removal of acetone-soluble portion of the wax present in a tropical wood (*Tectona grandis*), resulted in a considerable increase in the glue-bond quality obtained with a resorcinol adhesive. Similar results are reported by Thomas (44), using ether and benzene as extracting solvents. Hancock (22) has shown that a reduction in adhesion of oven-dried veneer is primarily because of extractives migration to the surface. He also determined that the inhibition was caused by acetone or a combination of acetone and methanol/benzene extractable fractions.

Extractives may have an unfavorable effect on the polarity and wettability of wood (24). Chen (15) found that the removal of extractives from wood surfaces with various solvents improved the wettability of wood. However, he also observed that there was no direct relationship between wettability and gluability using resorcinol-formaldehyde

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[6] Cited from Troop and Wangaard (45).

[7] Cited from Troop and Wangaard (45).

resin. Jain *et al.* (27) indicate that alcohol-benzene extracted samples of *Pinus roxburghii* glued better than the unextracted samples. They further noted that hot water extracted samples did not show a remarkable improvement in bond quality, in comparison with the unextracted samples. The results of the present study show a similar trend.

Roffael *et al.* (38) observed that extraction of oak particles with boiling water favourably affected the gluability of the particles. This contradicts the findings of the present study. However, they also report an improvement in gluability with addition of sodium hydroxide solution; this bit of their findings is in close agreement with the present results.

It is evident from the present results that surface treatment with sodium hydroxide solution significantly increased the wood-failure values of camphor wood blocks, glued with phenol-resorcinol-formaldehyde adhesive. However, these results, indicating that the sodium hydroxide treatment was effective, contradict the findings of Dougal *et al.* (18). They reported that extraction of some S.E. Asian hardwoods, with sodium hydroxide solution, did not consistently increase the wood failure values.

4.7 Gluing with Urea Formaldehyde

For this part of the study, only one wood level was used i.e., unextracted wood. The results obtained, especially the wood failure, lend to the conclusion that camphor wood glues

well with urea formaldehyde. Hence, the author did not find
it essential to investigate the adhesion with UF resin i.e
using pre-treated camphor wood.

4.7.1 Dry test: Shear strength and wood failure
percent

Bond shear strength and percentage wood failure results
are shown in the Tables 18a and 18b.

The untreated camphor wood showed a reasonably high
average shear strength when bonded with urea-formaldehyde
adhesive. It also showed a very high percentage of wood
failure which suggests that its the wood itself that failed,
during testing, rather than the adhesive. From this
observation, it can be stated that camphor wood glues well
with urea formaldehyde adhesive.

4.7.2 Cold soak test: Shear strength and wood failure
percent

Tables 19a and 19b present the shear strength and wood
failure results for the cold soak test.

The results show a reduction in shear strength from
13,654 kPa for the dry test to 6,898 kPa for the cold soak
test. There is also a reduction in wood failure percentage
from 97% to 66%.

Most assemblies bonded with urea-formaldehyde are
intended for interior use because the gluelines are not fully
waterproof, only highly water-resistant (19). A combination
of high relative humidity and high temperature deteriorates

urea resin glue bonds in a relatively short time. Resistance to cyclic soaking and drying exposures is reasonably good if the test pieces are plywood or thin members, but only moderate if the pieces are thick (like the one used in this study) (41).

5.0 SUMMARY AND CONCLUSION

A summary of the results obtained in this study is given in Table 20. Figures 6, 7, 8, 9, 10 further illustrate these results. Under the light of this study is concluded that:

(1) On bond strength basis, surface treatment of camphor wood with nitric acid, prior to gluing, improved its gluability with PVA. However, on percentage wood failure basis, none of the four wood pre-treatments significantly improved adhesion between PVA adhesive and camphor wood.

(2) From the standpoint of bond shear strength, surface treatment of camphor wood with sodium hydroxide, and extraction with alcohol-benzene, prior to gluing, enhanced its gluability with casein adhesive. Adhesion was not improved by either extracting the wood with hot water or by surface treating with nitric acid prior to gluing it with casein adhesive. In view of percentage wood failure, gluability of camphor wood with casein was improved by surface treating it with sodium hydroxide solution, and extracting with alcohol-benzene or hot water prior to gluing operation. Surface treating with nitric acid had a negative effect on adhesion.

(3) Extracting camphor wood using alcohol-benzene or surface treating it with sodium hydroxide, prior to gluing with PRF, improved adhesion. On the

contrary, treating it with nitric acid or extracting it with hot water lowered adhesion. Alcohol-benzene, and hot water extraction improved adhesion; while sodium hydroxide and nitric acid treatments had a negative effect.

(4) Cold water soaking of specimens bonded with PRF specimens, prior to testing, resulted in a reduction in bond strength of blocks made from unextracted wood, nitric acid treated and sodium hydroxide treated wood. Blocks made with nitric acid treated wood showed a remarkable drop in bond strength. But a slight increase in bond strength was observed with alcohol-benzene and hot water treatments. There was an increase in percent wood failure for the hot water, and alcohol-benzene treatments.

(5) A drop in bond strength was observed when test specimens made from nitric acid treated, alcohol benzene extracted, and sodium hydroxide treated wood were boiled in water before testing. However, the blocks made from unextracted wood gained in bond strength after boiling. Probably, a post-cure of the adhesive took place as a result of temperature increase. The boil treatment was observed to increase amount of wood failure in all the treatments except the sodium hydroxide treatment.

(6)   Using dry bond strength and percent wood failure as
      a criteria, it was found that camphor wood bonds
      well with urea-formaldehyde adhesive without any
      surface treatment as suggested herein.

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Table 1. Type of tests

| Wood | Adhesive/Test | | | |
	PRF	Casein	UF	PVA
Unextracted wood	Dry Cold soak Boil	Dry	Dry Cold soak	Dry
Extracted wood:				
(i) with alcohol-benzene	Dry Cold soak Boil	Dry	-* -	Dry
(ii) with hot water	Dry Cold soak Boil	Dry	-	Dry
Surface treated:				
(i) with 10% NaOH	Dry Cold soak Boil	Dry	- -	Dry
(ii) with dilute nitric acid	Dry Cold soak Boil	Dry	-	Dry

*No joints were made using treated camphor wood and UF adhesive.

Table 2: Formulation of glues and gluing conditions

Item	Glue	Phenol resorcinol-formaldehyde	Urea-formaldehyde	Polyvinyl-acetate	Casein
Mixing ratio of glues	Resin	100	100	100	100
	Water	-	-	-	200
	Hardner	15	10	-	-
Gluing conditions	Glue spread (g/m^2)	410.6	410.6	410.6	410.6
	Open assembly time (min)	10	5	3	5
	Closed assembly	30	20	10	20
	Temperature (^{o}C)	21	21	21	21
	Pressure (kg/cm^2)	14	14	14	14
	Time (h)	24	24	24	24

Table 3: Total Extractives Content in Camphor Wood

Species	Hot Water Extractives (%)	Alcohol–Benzene Extractives (%)	Cold Water Extractives (%)
Camphor wood	9.0	10.2	8.8

** Each value is an average of 3 determinations and is on a moisture–free basis.

Table 4a: Average Bond Strengtn of Blocks Bonded with PVA (DRY TEST).

Treatments (Wood)	Mean Bond Strength (kPa)	Standard Deviation	Minimum Bond Strength (kPa)	Maximum Bond Strength (kPa)	Range (kPa)	Standard Error of Mean	Coeff. of Variation (%)
Unextracted (Control)	13209	1992	9735	16251	6516	514	15
Hot Water Extracted	12164	1315	9839	14789	4950	340	11
Nitric acid Treated	14692	1889	11514	16968	5454	488	13
10% NaOH Treated	12745	2247	9742	16072	6330	580	18
A.-Benzene Extracted	7452	1690	3889	9818	5929	436	23

Table 4b: Average Wood Failure of Blocks Bonded with PVA (DRY TEST) .

Treatments (Wood)	Mean Wood Failure (%)	Standard Deviation	Minimum Wood Failure (%)	Maximum Wood Failure (%)	Range (%)	Standard Error of Mean	Coeff. of Variation (%)
Unextracted (Control)	56	30	20	100	80	8	54
Hot Water Extracted	18	24	5	80	75	6	129
Nitric acid Treated	49	28	0	100	100	7	58
10% NaOH Treated	70	25	20	100	80	6	35
A−Benzene Extracted	6	9	0	25	25	2	156

Table 5a: Analysis of Variance for Bond Srength of Blocks Bonded with PVA (DRY TEST) .

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	Computed f
Wood	4	449392385.253	112348096.313	32.71
Error	70	240426625.067	3434666.072	
Total	74	689819010.320		

Table 5b: Analysis of Variance for Wood Failure of Blocks Bonded with PVA (DRY TEST) .

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	Computed f
Wood	4	42721.333	10680.333	18.09
Error	70	41316.666	590.238	
Total	74	84038.000		

Table 6: Statistical Ranking by Duncan's Multiple Range Test for Bond
Strength and Wood Failure of Blocks Bonded with PVA .

STRENGTH (kPa)

RANKING OF TREATMENT MEANS

Dry Test	NIT.	UNX.	NAO.	WAT.	BEN.
	14692	13209	12745	12164	7452

WOOD FAILURE (%)

RANKING OF TREATMENT MEANS

Dry Test	NAO.	UNX.	NIT.	WAT.	BEN.
	70	56	49	18	6

N.B. 1. Means underscored by the same line are not significantly
different at the 5% level of significance.

2. BEN.=A.–Benzene extracted ; NIT.=Nitric acid treated ; NAO.=NaOH treated;
WAT.=Hot water extracted ; UNX.=Unextracted(Control).

Table 7a: Average Bond Strength of Blocks Bonded with Casein (DRY TEST).

Treatments (Wood)	Mean Bond Strength (kPa)	Standard Deviation	Minimum Bond Strength (kPa)	Maximum Bond Strength (kPa)	Range (kPa)	Standard Error of Mean	Coeff. of Variation (%)
Unextracted (Control)	9933	4207	2723	16175	13452	1086	42
Hot Water Extracted	11520	1398	9522	14493	4971	361	12
Nitric acid Treated	5911	1727	2744	8336	5592	446	29
10% NaOH Treated	14297	2523	9632	17961	8329	651	18
A–Benzene Extracted	12955	2168	8963	17078	8115	560	17

Table 7b: Average Wood Failure of Blocks Bonded with Casein (DRY TEST).

Treatments (Wood)	Mean Wood Failure (%)	Standard Deviation	Minimum Wood Failure (%)	Maximum Wood Failure (%)	Range (%)	Standard Error of Mean	Coeff. of Variation (%)
Unextracted (Control)	15	19	0	60	60	5	129
Hot Water Extracted	78	30	25	100	75	8	39
Nitric acid Treated	0	0	0	0	0	0	0
10% NaOH Treated	51	34	10	100	90	9	67
A.—Benzene Extracted	45	23	10	90	80	50	6

Table 8a: Analysis of Variance for Bond Srength of Blocks Bonded with Casein (DRY TEST)

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	Computed f
Wood	4	629570759.013	157392689.753	23.35
Error	70	471846902.533	6740670.036	
Total	74	1101417661.547		

Table 8b: Analysis of Variance for Wood Failure of Blocks Bonded with Casein (DRY TEST)

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	Computed f
Wood	4	57137.520	14284.380	24.13
Error	70	41437.600	591.966	
Total	74	98575.120		

Table 9: Statistical Ranking by Duncan's Multiple Range Test for Bond
Strength and Wood Failure of Blocks Bonded with Casein.

STRENGTH (kPa)

RANKING OF TREATMENT MEANS

Dry Test	NAO.	BEN.	WAT.	UNX.	NIT.
	14297	12955	11520	9933	5911

WOOD FAILURE (%)

RANKING OF TREATMENT MEANS

Dry Test	WAT.	NAO.	BEN.	UNX.	NIT.
	78	51	45	15	0

N.B. 1. Means underscored by the same line are not significantly
different at the 5% level of significance.

2. BEN.=A.—Benzene extracted ; NIT.=Nitric acid treated ; NAO.=NaOH treated;
WAT.=Hot water extracted ; UNX.=Unextracted(Control).

Table 10a: Average Bond Strength of Blocks bonded with PRF (DRY TEST).

Treatments (Wood)	Mean Bond Strength (kPa)	Standard Deviation	Minimum Bond Strength (kPa)	Maximum Bond Strength (kPa)	Range (kPa)	Standard Error of Mean	Coeff. of Variation (%)
Unextracted (Control)	12924	1597	9687	15258	5571	412	12
Hot Water Extracted	10411	1866	7750	15113	7363	482	18
Nitric acid Treated	9709	2150	6385	14300	7915	555	22
10% NaOH Treated	14629	2752	10977	18719	7742	711	19
A.–Benzene Extracted	14543	1534	12038	17037	4999	396	11

Table 10b: Average Wood Failure of Blocks Bonded with PRF (DRY TEST) .

Treatments (Wood)	Mean Wood Failure (%)	Standard Deviation	Minimum Wood Failure (%)	Maximum Wood Failure (%)	Range (%)	Standard Error of Mean	Coeff. of Variation (%)
Unextracted (Control)	30	24	0	70	70	6	79
Hot Water Extracted	60	35	0	100	100	9	58
Nitric acid Treated	0	0	0	0	0	–	–
10% NaOH Treated	75	21	20	100	80	5	28
A.–Benzene Extracted	98	4	90	100	10	1	4

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Table 11a: Analysis of Variance for Bond Srength of Blocks Bonded with PRF (DRY TEST) .

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	Computed f
Wood	4	315354452.618	78838613.153	19.15
Error	70	288109729.733	4115853.282	
Total	74	603464182.347		

Table 11b: Analysis of Variance for Wood Failure of Blocks Bonded with PRF (DRY TEST) .

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	Computed f
Wood	4	88655.333	22163.833	49.66
Error	70	31243.333	446.333	
Total	74	119898.667		

Table 12a: Average Bond Strength of Blocks Bonded with PRF (COLD SOAK TEST).

Treatments (Wood)	Mean Bond Strength (kPa)	Standard Deviation	Minimum Bond Strength (kPa)	Maximum Bond Strength (kPa)	Range (kPa)	Standard Error of Mean	Coeff. of Variation (%)
Unextracted (Control)	11509	1164	9556	13638	4082	301	10
Hot Water Extracted	10421	856	8832	12245	3413	221	8
Nitric acid Treated	1321	469	772	2144	1372	121	36
10% NaOH Treated	12708	2158	7398	15893	8495	557	17
A.–Benzene Extracted	14609	1570	10666	16217	5551	405	11

Table 12b: Average Wood Failure of Blocks Bonded with PRF (COLD SOAK TEST) .

Treatments (Wood)	Mean Wood Failure (%)	Standard Deviation	Minimum Wood Failure (%)	Maximum Wood Failure (%)	Range (%)	Standard Error of Mean	Coeff. of Variation (%)
Unextracted (Control)	60	33	10	100	90	9	56
Hot Water Extracted	91	15	60	100	40	4	16
Nitric acid Treated	0	0	0	0	0	—	—
10% NaOH Treated	42	24	10	70	60	6	58
A.—Benzene Extracted	100	0	100	100	0	0	0

Table 13a: Analysis of Variance for Bond Srength of Blocks Bonded with PRF (COLD SOAK TEST) .

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	Computed f
Wood	4	1565248081.556	391312020.389	26.05
Error	70	131038117.957	1899103.159	
Total	74	1696286199.514		

Table 13b: Analysis of Variance for Wood Failure of Blocks Bonded with PRF (COLD SOAK TEST) .

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	Computed f
Wood	4	97282.000	24320.500	63.60
Error	70	26766.666	382.381	
Total	74	124048.667		

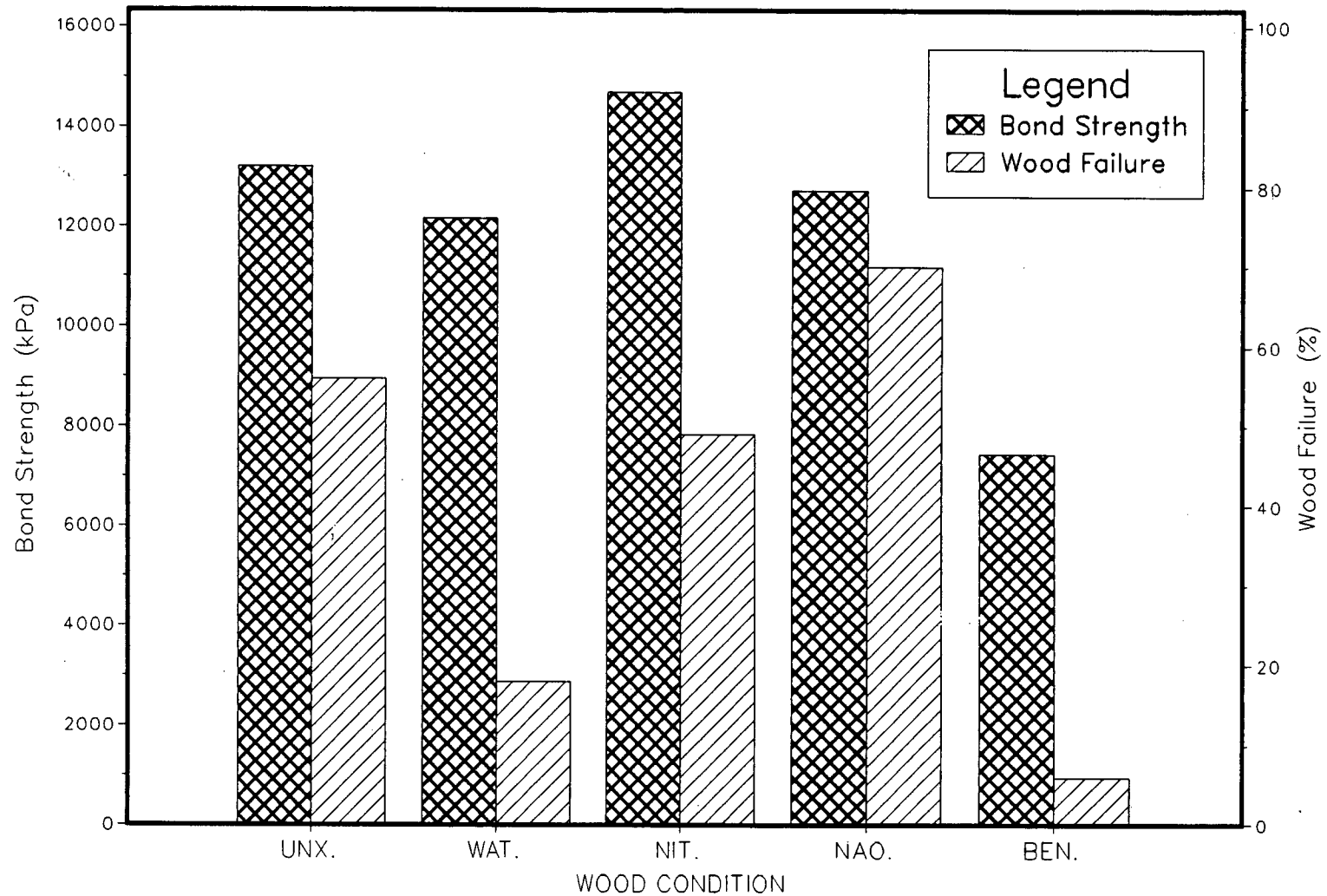Table 14a: Average Bond Strength of Blocks Bonded with PRF (BOIL TEST).

Treatments (Wood)	Mean Bond Strength (kPa)	Standard Deviation	Minimum Bond Strength (kPa)	Maximum Bond Strength (kPa)	Range (kPa)	Standard Error of Mean	Coeff. of Variation (%)
Unextracted (Control)	12926	1840	10590	15210	4620	555	14
Hot Water Extracted	9920	1212	8157	12707	4550	366	12
Nitric acid Treated	9523	2478	5130	12411	7281	747	26
10% NaOH Treated	10103	1271	8143	12997	4854	383	13
A.-Benzene Extracted	13759	1344	11342	15961	4619	403	10

Table 14b: Average Wood Failure of Blocks Bonded with PRF (BOIL TEST).

Treatments (Wood)	Mean Wood Failure (%)	Standard Deviation	Minimum Wood Failure (%)	Maximum Wood Failure (%)	Range (%)	Standard Error of Mean	Coeff. of Variation (%)
Unextracted (Control)	56	31	10	95	85	9	55
Hot Water Extracted	87	15	60	100	40	4	17
Nitric acid Treated	13	18	0	50	50	5	136
10% NaOH Treated	45	37	5	95	90	11	82
A.–Benzene Extracted	100	0	100	100	0	0	0

Table 15a: Analysis of Variance for Bond Srength of Blocks Bonded with PRF (BOIL TEST) .

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	Computed f
Wood	4	149574795.732	37373698.933	13.20
Error	70	138824944.581	2833162.134	
Total	74	288399740.315		

Table 15b: Analysis of Variance for Wood Failure of Blocks Bonded with PRF (BOIL TEST) .

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	Computed f
Wood	4	52548.182	13137.045	23.19
Error	70	28322.727	566.455	
Total	74	80870.909		

Table 16: Statistical Ranking by Duncan's Multiple Range Test for Bond Strength and Wood Failure of Blocks Bonded with PRF.

STRENGTH (kPa)

RANKING OF TREATMENT MEANS

Dry Test	NAO. 14629	BEN. 14543	UNX. 12924	WAT. 10411	NIT. 9709

Cold Soak Test	BEN. 14609	NAO. 12708	UNX. 11509	WAT. 10421	NIT. 1321

Boil Test	BEN. 13759	UNX. 12926	NAO. 10103	WAT. 9920	NIT. 9523

WOOD FAILURE (%)

RANKING OF TREATMENT MEANS

Dry Test	BEN. 98	NAO. 75	WAT. 60	UNX. 30	NIT. 0

Cold Soak Test	BEN. 100	WAT. 91	UNX. 60	NAO. 42	NIT. 0

Boil Test	BEN. 100	WAT. 87	UNX. 56	NAO. 45	NIT. 13

N.B. 1. Means underscored by the same line are not significantly different at the 5% level of significance.

2. BEN.=A.–Benzene extracted; NIT.=Nitric acid treated; NAO.=NaOH treated; WAT.=Hot water extracted ; UNX.=Unextracted(Control).

Table 17: Comparison of Bond Strength Test Results of blocks bonded with PRF .

Treatments (Wood)	DRY TEST		COLD SOAK TEST		BOIL TEST	
	Mean Bond Strength (kPa)	Mean Wood Failure (%)	Mean Bond Strength (kPa)	Mean Wood Failure (%)	Mean Bond Strength (kPa)	Mean Wood Failure
Unextracted (Control)	12924	30	11509	60	12926	56
Hot Water Extracted	10411	60	10421	91	9920	87
Nitric acid Treated	9709	0	1321	0	9523	13
10% NaOH Treated	14629	75	12708	42	10103	45
A.–Benzene Extracted	14543	98	14609	100	13759	100

Table 18a: Average Bond Strength of Blocks bonded with UF (DRY TEST).

Treatments (Wood)	Mean Bond Strength (kPa)	Standard Deviation	Minimum Bond Strength (kPa)	Maximum Bond Strength (kPa)	Range (kPa)	Standard Error of Mean	Coeff. of Variation (%)
Unextracted (Control)	13654	2409	1008	20181	10101	622	18

Table 18b: Average Wood Failure of Blocks bonded with UF (DRY TEST).

Treatments (Wood)	Mean Bond Strength (kPa)	Standard Deviation	Minimum Bond Strength (kPa)	Maximum Bond Strength (kPa)	Range (kPa)	Standard Error of Mean	Coeff. of Variation (%)
Unextracted (Control)	97	6	80	100	20	2	6

Table 19a: Average Bond Strength of Blocks bonded with UF (COLD SOAK TEST).

Treatments (Wood)	Mean Bond Strength (kPa)	Standard Deviation	Minimum Bond Strength (kPa)	Maximum Bond Strength (kPa)	Range (kPa)	Standard Error of Mean	Coeff. of Variation (%)
Unextracted (Control)	6898	2825	1579	11569	9990	729	41

Table 19b: Average Wood Failure of Blocks bonded with UF (COLD SOAK TEST).

Treatments (Wood)	Mean Bond Strength (kPa)	Standard Deviation	Minimum Bond Strength (kPa)	Maximum Bond Strength (kPa)	Range (kPa)	Standard Error of Mean	Coeff. of Variation (%)
Unextracted (Control)	66	31	15	100	85	8	47

Table 20: Summary of Bond Strength and Wood Failure Results.

Treatments (Wood)	PVA		Casein		PRF		UF	
	Mean Bond Strength (kPa)	Mean Wood Failure (%)	Mean Bond Strength (kPa)	Mean Wood Failure (%)	Mean Bond Strength (kPa)	Mean Wood Failure (%)	Mean Bond Strength (kPa)	Mean Wood Failure (%)
Unextracted (Control)	D 13209	D 56	D 9933	D 15	D 12924 C 11509 B 12926	D 30 C 60 B 56	D 13654 C 6898	D 97 C 66
Hot Water Extracted	D 12164	D 18	D 11520	D 78	D 10411 C 10421 B 9920	D 60 C 91 B 87		
Nitric acid Treated	D 14692	D 49	D 5911	D 0	D 9709 C 1321 B 9523	D 0 C 0 B 13		
10% NaOH Treated	D 12745	D 70	D 14297	D 51	D 14629 C 12708 B 10103	D 75 C 42 B 45		
A.–Benzene Extracted	D 7452	D 6	D 12955	D 45	D 14543 C 14609 B 13759	D 98 C 100 B 100		

N.B: B=Boil test ; C=Cold soak test ; D=Dry test.

Figure 1. Average Bond Strength and Wood Failure of Blocks bonded with PVA (DRY TEST).

N.B   BEN.=Alcohol−Benzene extracted ; NAO.=NaOH treated ; NIT.=Nitric acid treated ;
      WAT.=Hot water extracted ; UNX.=Unextracted(Control).

Figure 2. Average Bond Strength and Wood Failure of Blocks bonded with Casein (DRY TEST).
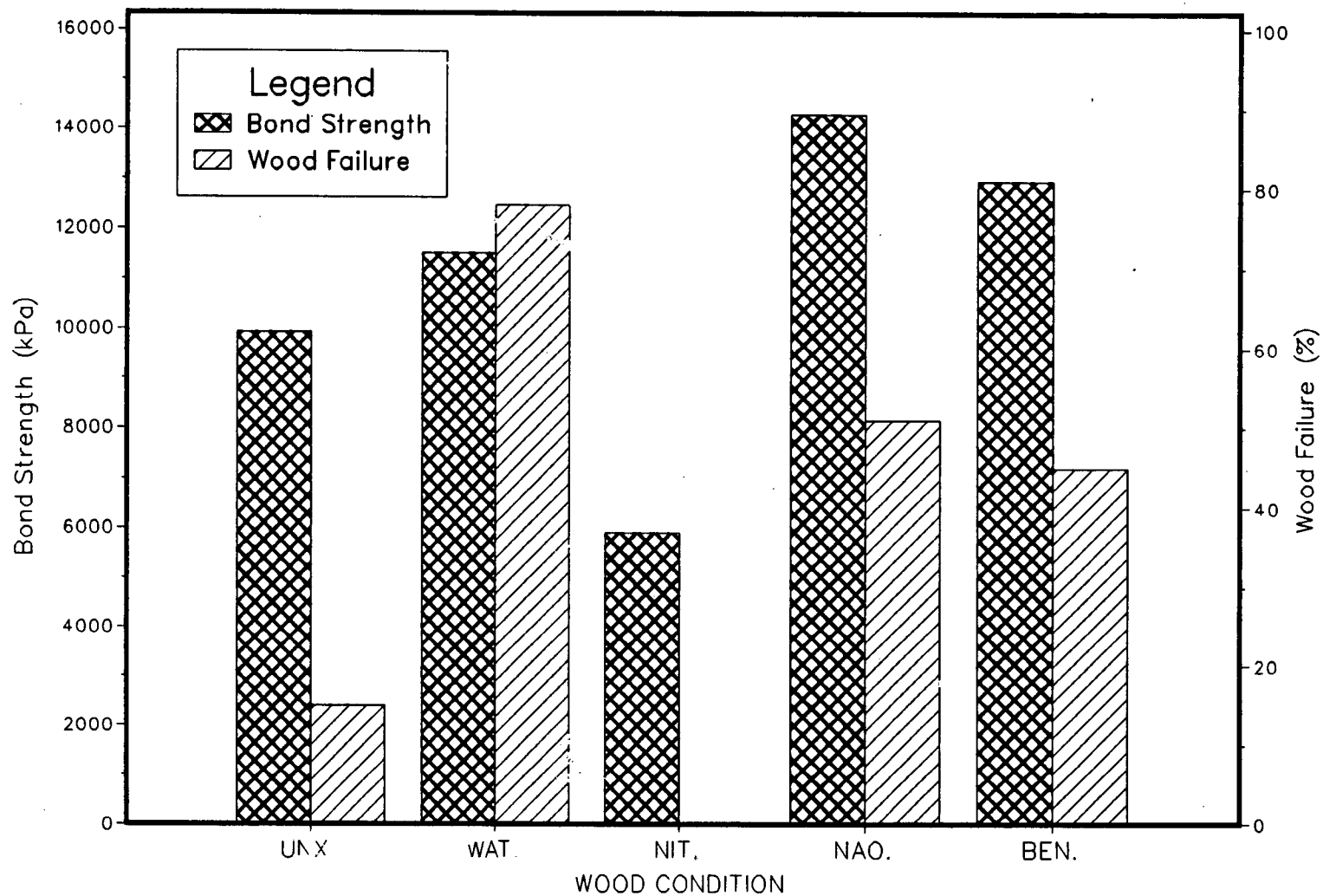
N.B BEN.=Alcohol–Benzene extracted ; NAO.=NaOH treated ; NIT.=Nitric acid treated ;
     WAT.=Hot water extracted ; UNX.=Unextracted(Control).

Figure 3. Average Bond Strength and Wood Failure of Blocks bonded with PRF (DRY TEST).
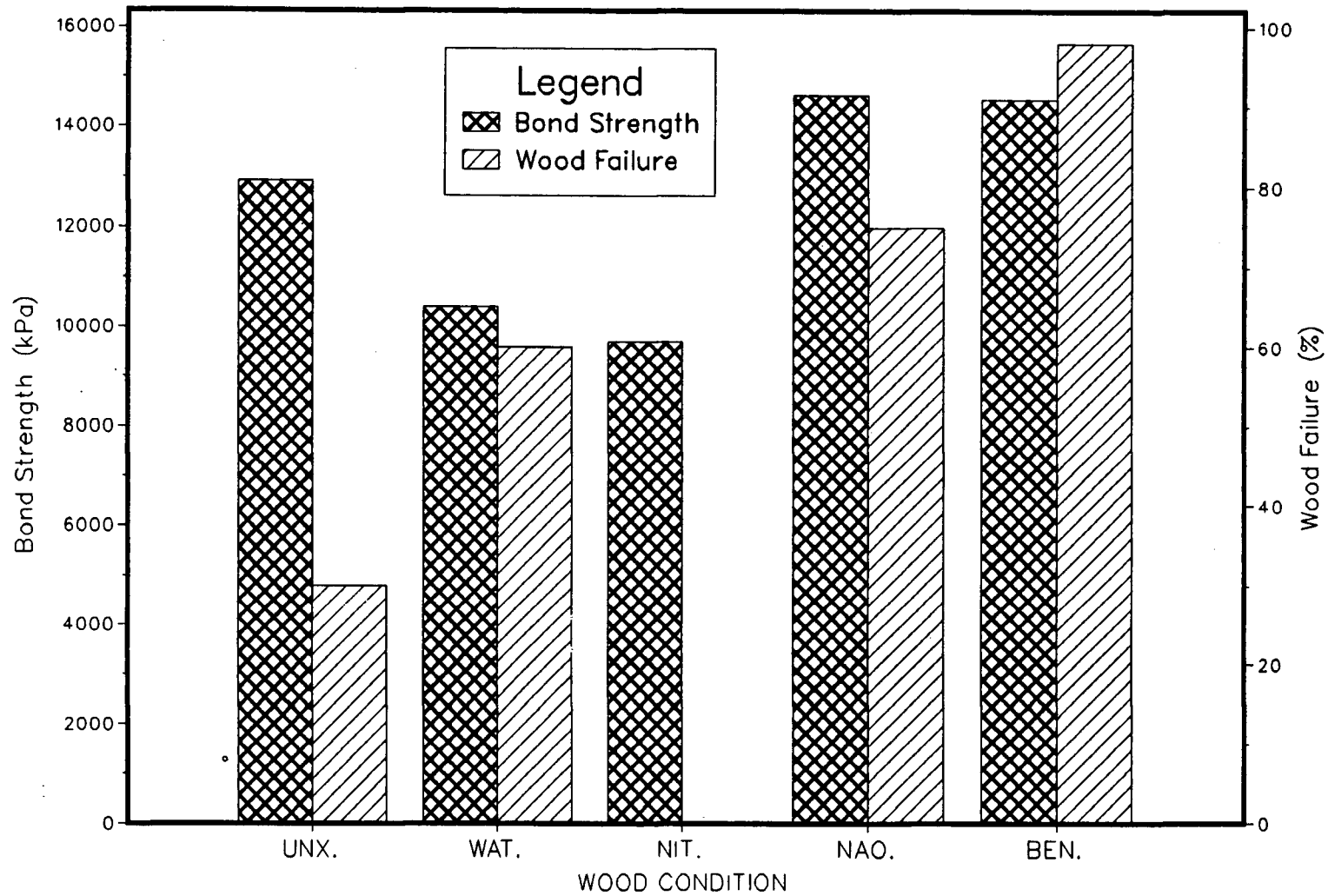
N.B BEN.=Alcohol−Benzene extracted ; NAO.=NaOH treated ; NIT.=Nitric acid treated ;
WAT.=Hot water extracted ; UNX.=Unextracted(Control).

Figure 4. Average Bond Strength and Wood Failure of Blocks bonded with PRF (COLD SOAK TEST).
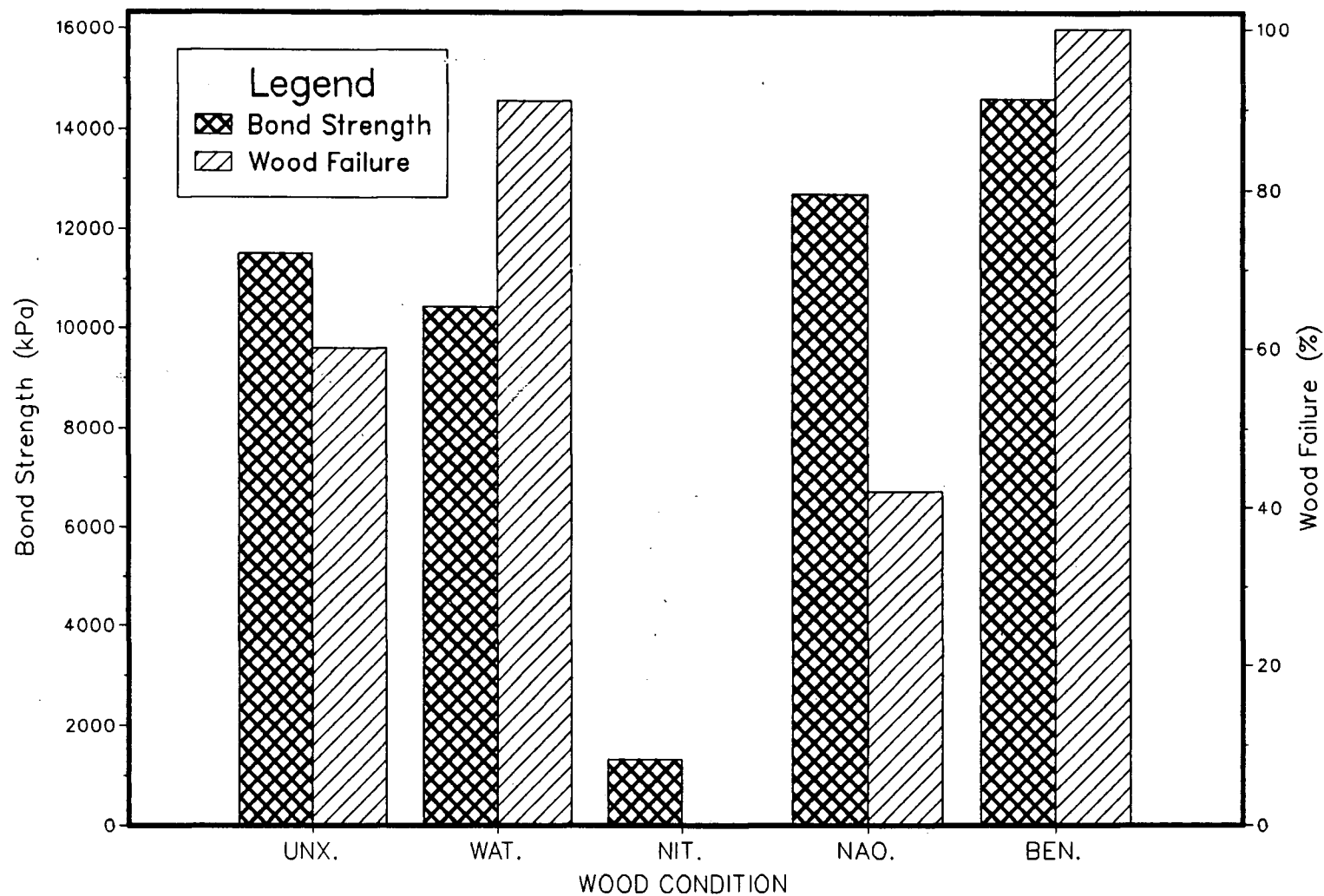
N.B BEN.=Alcohol−Benzene extracted ; NAO.=NaOH treated ; NIT.=Nitric acid treated ;

WAT.=Hot water extracted ; UNX.=Unextracted(Control).

Figure 5. Average Bond Strength and Wood Failure of Blocks bonded with PRF (BOIL TEST).
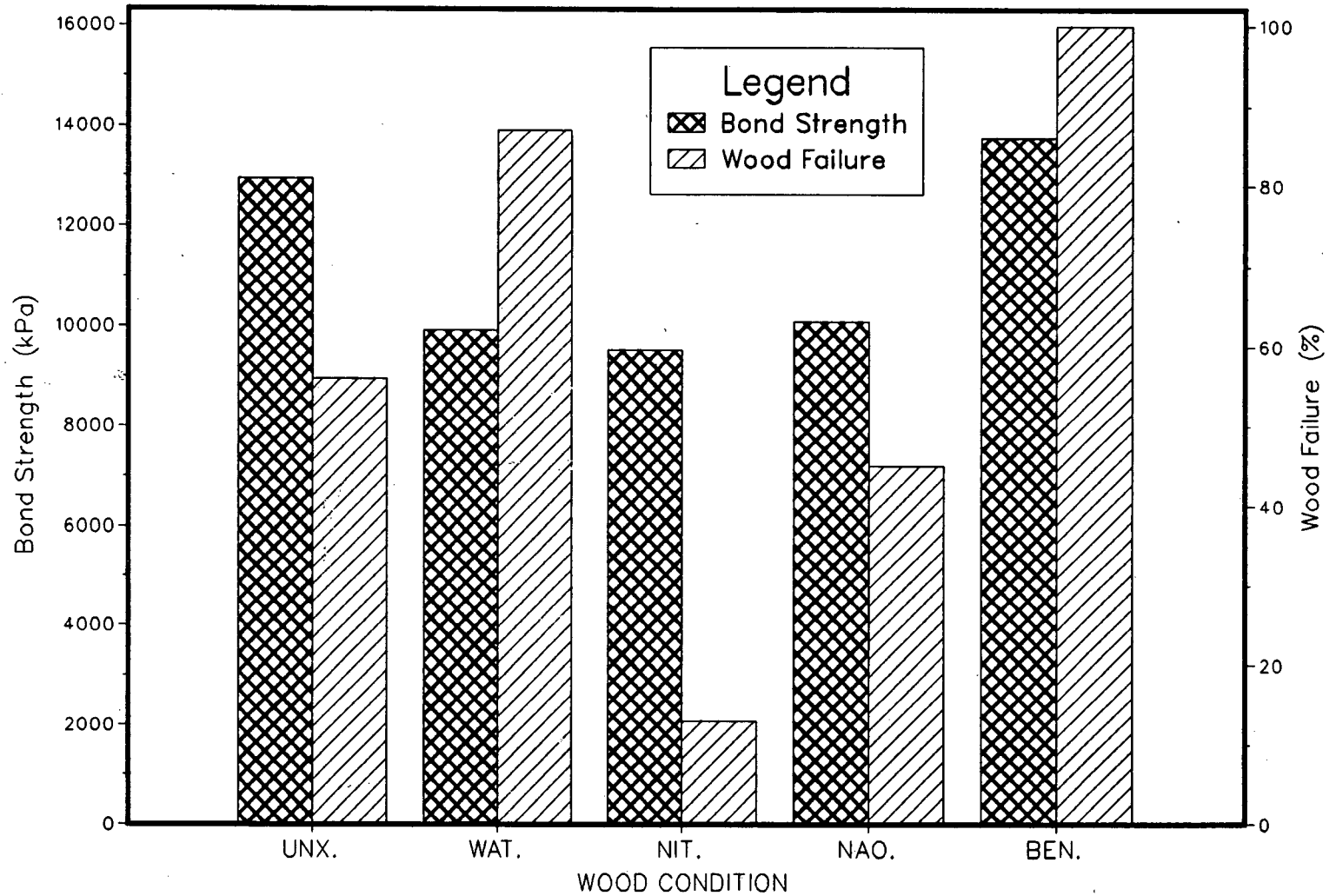
N.B BEN.=Alcohol–Benzene extracted ; NAO.=NaOH treated ; NIT.=Nitric acid treated ;
WAT.=Hot water extracted ; UNX.=Unextracted(Control).

Figure 6. Bond Strength and Wood Failure of Unextracted Blocks Bonded with PVA, Casein, PRF and UF Adhesives (DRY TEST).
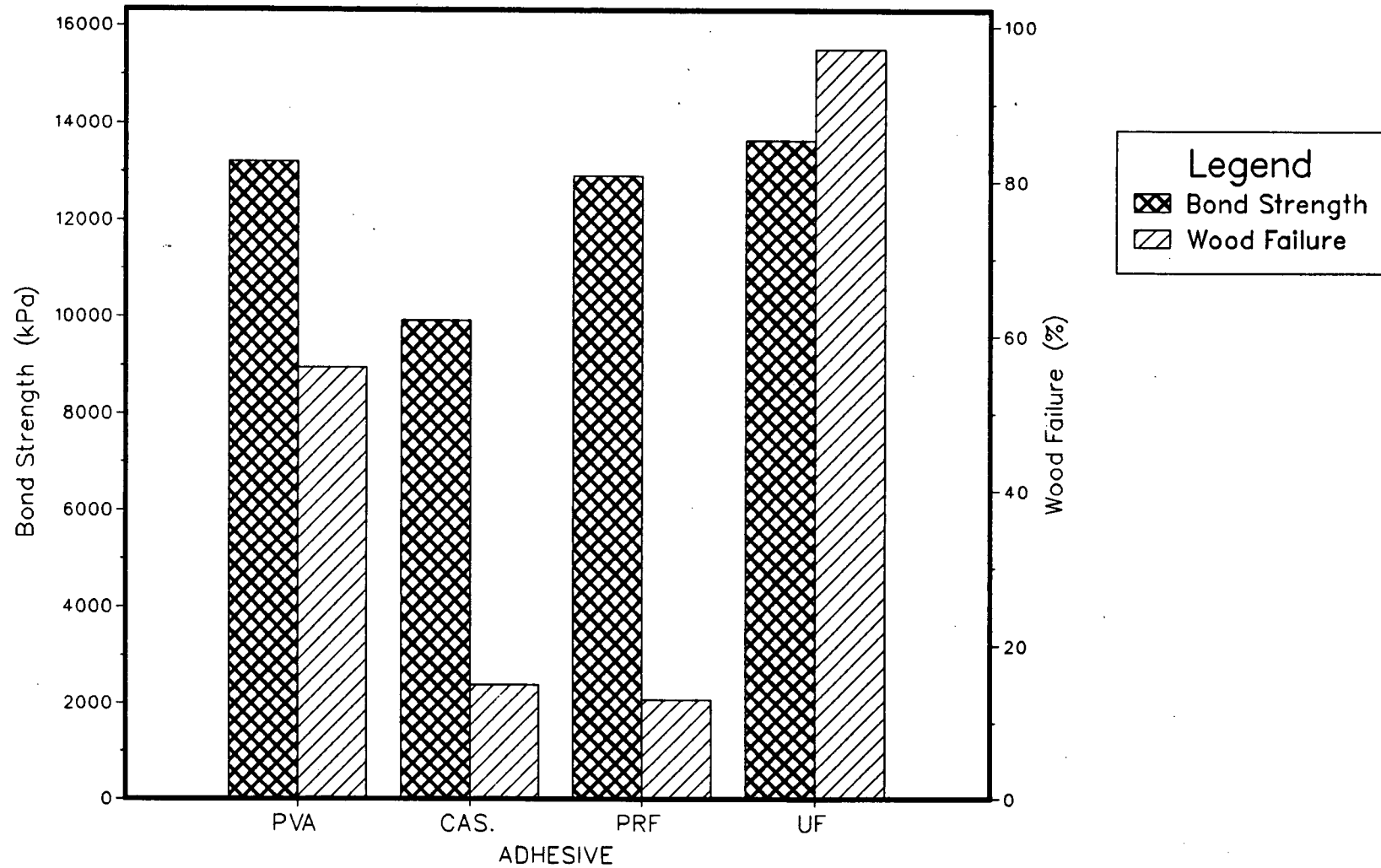
Figure 7. Bond Strength and Wood Failure of Hot Water Extracted Blocks
Bonded with PVA, Casein and PRF Adhesives (DRY TEST).
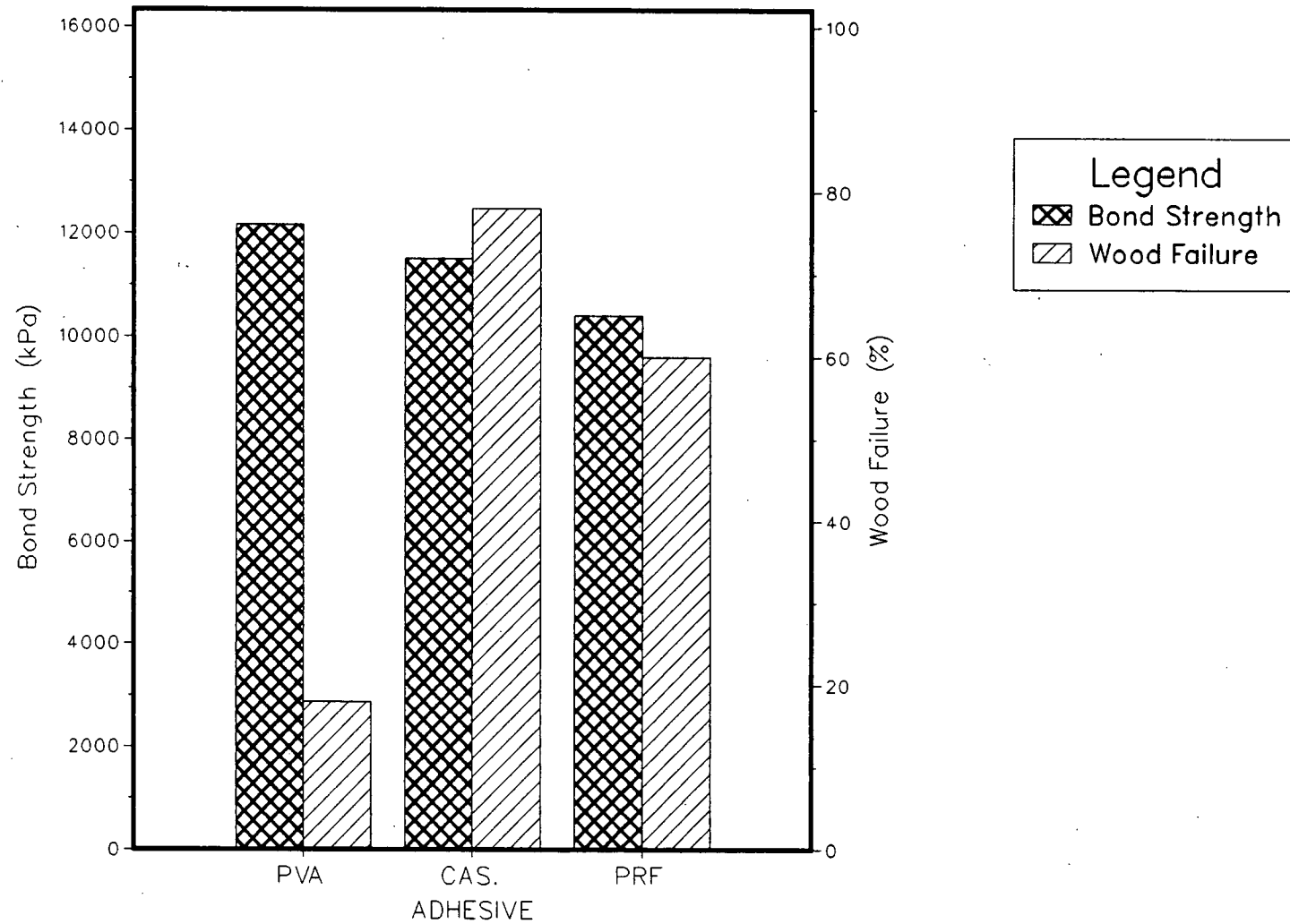
Figure 8. Bond Strength and Wood Failure of Nitric Acid Treated Blocks Bonded with PVA, Casein and PRF Adhesives (DRY TEST) .
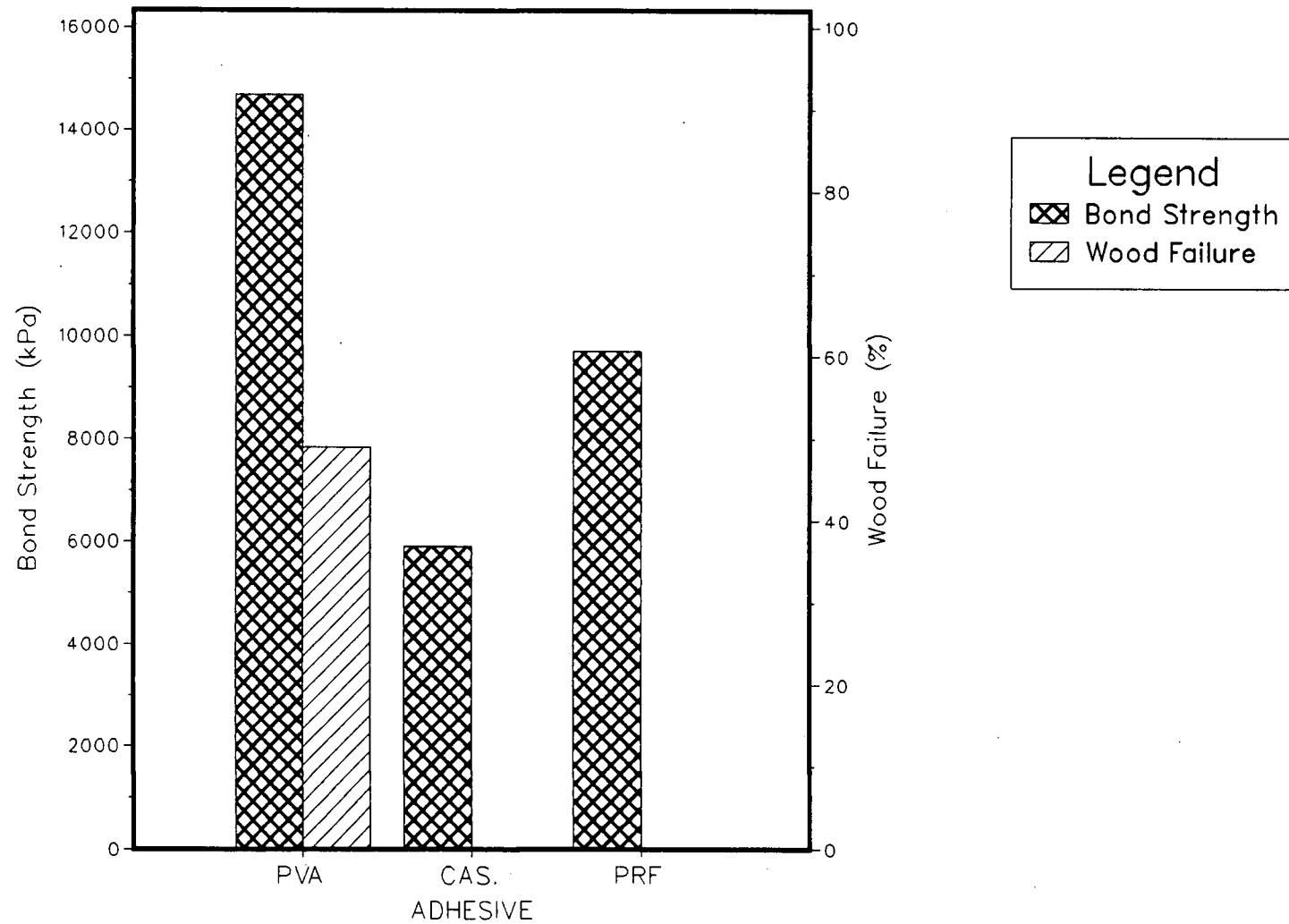
Figure 9. Bond Strength and Wood Failure of NaOH Treated Blocks Bonded with PVA, Casein and PRF Adhesives (DRY TEST).
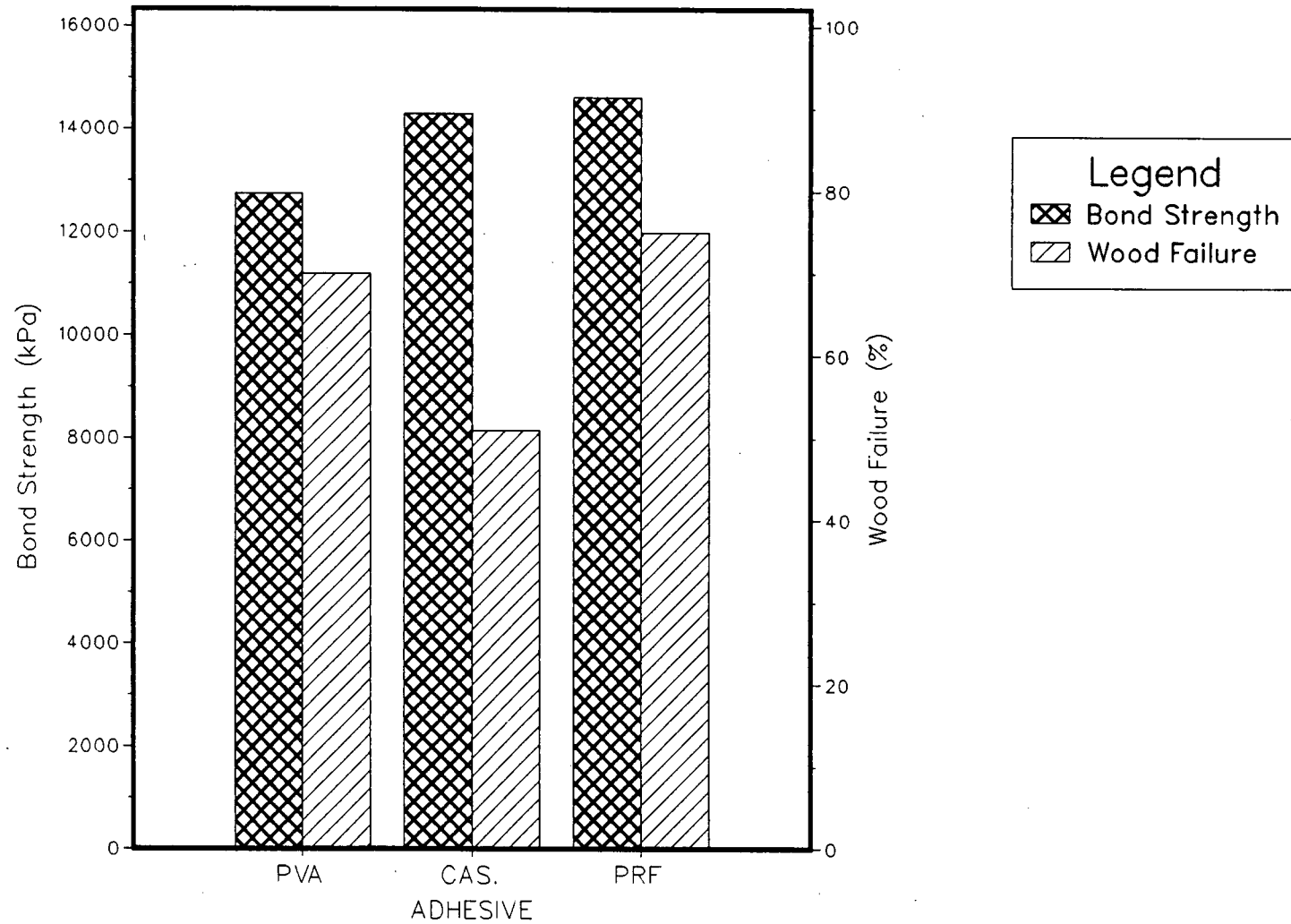
Figure 10. Bond Strength and Wood Failure of Alcohol—Benzene Extracted
Blocks Bonded with PVA,Casein and PRF Adhesives(DRY TEST).
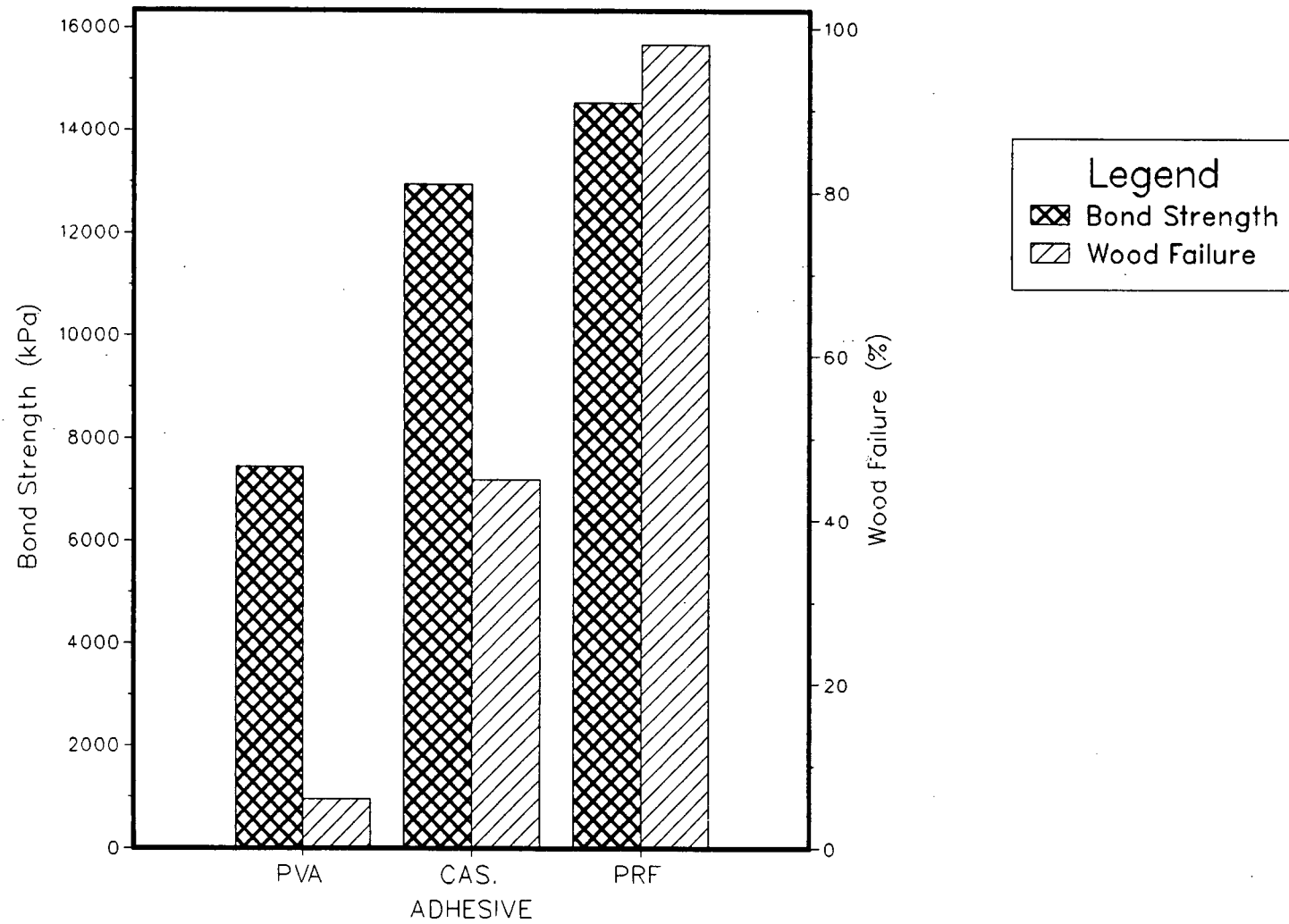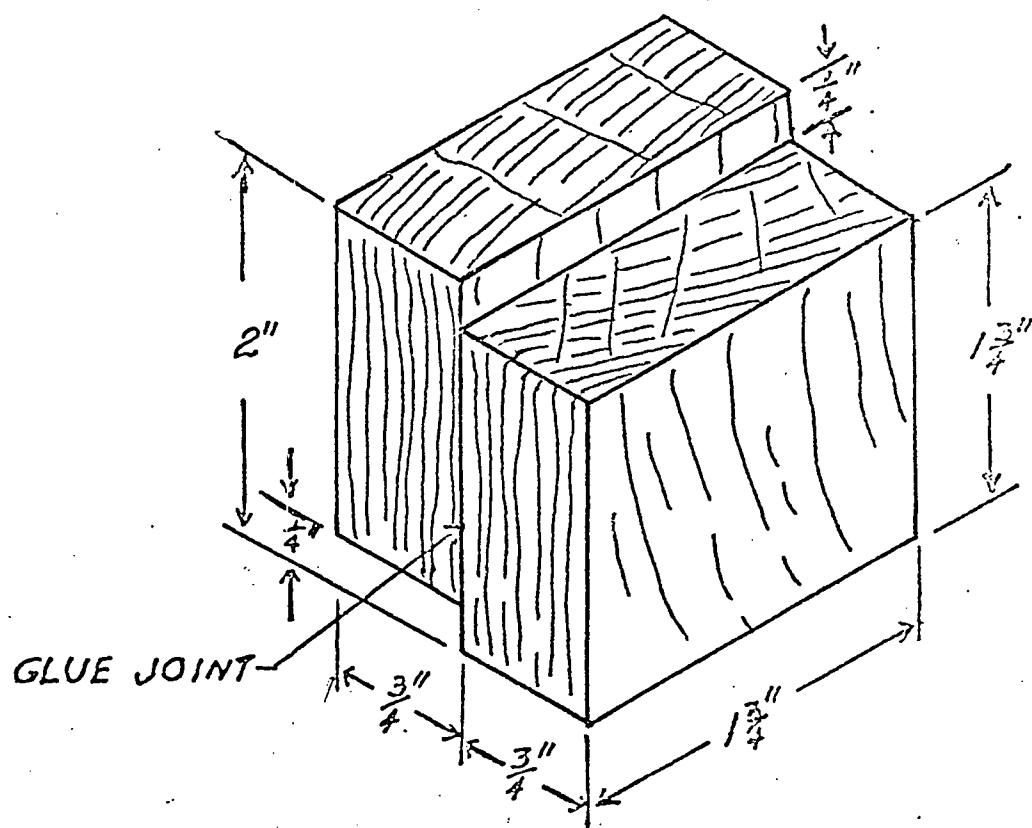
Figure 11 Form and dimension of block shear test specimen
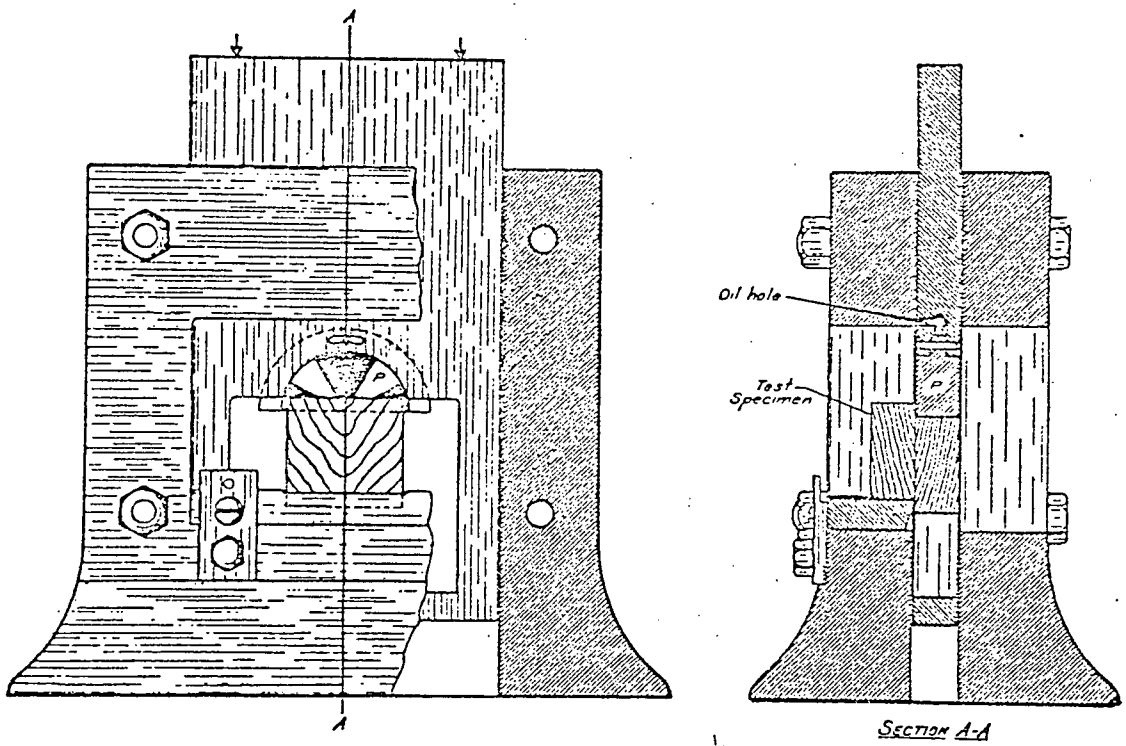
Figu. 12. Shearing tool

APPENDIX I

Species Description*

Botanical name: *Ocotea usambarensis*

Local name: Muthiti

Trade name: Camphor wood

Family: Lauraceae

Density (at 12% MC):   592.7 kg/m^3 (37 lbs/ft^3)

Specific gravity (at 12% MC): 0.59

Shrinkage (green to air dry):   Radial 2.5%; Tangential 4.0%

Workability: Moderately easy

Gluability: Average

Treatability: Extremely resistant

Seasoning rate: Moderate

Uses: Furniture, joinery, lorry bodies, boats,
                       panelling and overlay veneer

*From Timber Leaflet No. 1 "Commercial Timbers of Kenya",
    Bengough, C.C. (12).

APPENDIX II.

囲卿 Designation: D 905 – 49 (Reapproved 1981)ᵉ¹

Standard Test Method for

STRENGTH PROPERTIES OF ADHESIVE BONDS IN SHEAR BY COMPRESSION LOADING[1]

This standard is issued under the fixed designation D 905; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This method has been approved for use by agencies of the Department of Defense to replace method 1031 of Federal Test Method Standard No. 175a and for listing in the DoD Index of Specifications and Standards.

ᵉ¹ NOTE—Section 2 was added editorially and subsequent sections renumbered in March 1985.

INTRODUCTION

The accuracy of the results of strength tests of adhesive bonds will depend on the conditions under which the bonding process is carried out. Unless otherwise agreed upon by the manufacturer and the purchaser, the bonding conditions shall be prescribed by the manufacturer of the adhesive. In order to ensure that complete information is available to the individual conducting the tests, the manufacturer of the adhesive shall furnish numerical values and other specific information for each of the following variables:

(1) The moisture content of the wood at the time of gluing.

(2) Complete mixing directions for the adhesive.

(3) Conditions for applications of the adhesive including the rate of spread or thickness of film, number of coats to be applied, whether to be applied to one or both surfaces, and the conditions of drying where more than one coat is required.

(4) Assembly conditions before application of pressure, including the room temperature, length of time, and whether open or closed assembly is to be used.

(5) Curing conditions, including the amount of pressure to be applied, the length of time under pressure and the temperature of assembly when under pressure. It should be stated whether this temperature is that of the glue line, or of the atmosphere at which the assembly is to be maintained.

(6) Conditioning procedure before testing, unless a standard procedure is specified, including the length of time, temperature, and relative humidity.

A range may be prescribed for any variable by the manufacturer of the adhesive if it can be assumed by the test operator that any arbitrarily chosen value within such a range or any combination of such values for several variables will be acceptable to both the manufacturer and the purchaser of the adhesive.

1. Scope

1.1 This test method covers the determination of the comparative shear strengths of adhesive bonds used for bonding wood and other similar materials, when tested on a standard specimen under specified conditions of preparation, conditioning, and loading in compression. This test method is intended primarily as an evaluation of adhesives for wood.

2. Applicable Document

2.1 *ASTM Standard:*

D 143 Methods of Testing Small Clear Specimens of Timber[2]

3. Apparatus

3.1 The testing machine shall have a capacity of not less than 6810 kg (15 000 lb) in compression and shall be fitted with a shearing tool containing a self-aligning seat to ensure uniform lateral distribution of the load. The machine shall be capable of maintaining a uniform rate of loading such that the load may be applied with a continuous motion of the movable head to maximum load at a rate of 6.35×10^{-3} mm/s (0.015 in./min) with a permissible variation of ±25 %. The shearing tool shown in Fig. 1 has been found satisfactory. The testing machine shall be located in an atmosphere such that the moisture content of the specimens developed under the conditions prescribed in Section 6 is not noticeably altered during testing.

4. Test Specimens

4.1 Test specimens shall conform to the form and dimensions shown in Fig. 2. The specimens shall be cut from test joints prepared as described in Sections 5 and 6.

4.2 At least 20 specimens shall be tested, representing at least four different joints.

5. Preparation of Test Joints

5.1 Hard maple blocks (*Acer saccharum* or *Acer nigrum*), having a minimum sp gr of 0.65 based on oven-dry weight and volume shall be selected (Note). These blocks shall be of straight grain and free from defects including knots, birdseye, short grain, decay, and any unusual discolorations within the shearing area. The blocks shall be of suitable size preferably so that five test specimens may be cut from one test joint as shown in Fig. 3. Blocks approximately 19 by 63.5 by 304 mm (¾ by 2½ by 12 in.) have been found to be satisfactory for this purpose. The grain direction shall be parallel to the longest dimension of the block. The blocks shall be at the equilibrium moisture content recommended by the manufacturer of the adhesive. In the absence of such recommendation, the moisture content shall be from 10 to 12 % based on oven-dry weight as determined on representative samples in accordance with Sections 122 to 125 of Meth-

ods D 143. The blocks shall be surfaced, just prior to gluing, preferably with a hand-feed jointer, and the blocks weighed and assembled in pairs so that blocks of approximately the same specific gravity are glued together. The surfaces shall remain unsanded and shall be free from dirt.

NOTE—A method for selecting maple blocks of satisfactory specific gravity is described in the Appendix to this method. For referee tests, the specific gravity of blocks may be determined in accordance with Section 115 of Methods D 143.

5.2 The adhesive shall be prepared and applied to the blocks in accordance with the procedure recommended by the manufacturer of the adhesive. The glue-coated blocks shall then be assembled and pressed, likewise in accordance with the recommendations of the manufacturer of the adhesive.

6. Conditioning of Test Joints

6.1 The joints, upon removal from pressure shall be conditioned at a relative humidity of 50 ± 2 % and at a temperature of 23 ± 1°C (73.4 ± 2°F) either for a period of 7 days or until specimens reach equilibrium as indicated by no progressive changes in weight, whichever is the shorter period. The length of this period of conditioning may be extended beyond this limit by written agreement between the purchaser and the manufacturer of the adhesive.

7. Preparation of Specimens

7.1 Cut the specimens as shown in Fig. 3 so that the grain direction is parallel to the direction of loading during test. Take care in preparing the test specimens to make the loaded surfaces smooth and parallel to each other and perpendicular to the height. Take care also in reducing the lengths of the laminations to 44.4 mm (1¾ in.) to ensure that the saw cuts extend to, but not beyond, the glue line. Measure the width and length of the specimen at the glue line to the nearest 0.25 mm (0.010 in.) to determine the shear area.

7.2 Retain specimens in the conditioning atmosphere described in Section 6 until tested, except during the cutting operations.

8. Procedure

8.1 Place the test specimen in the shearing
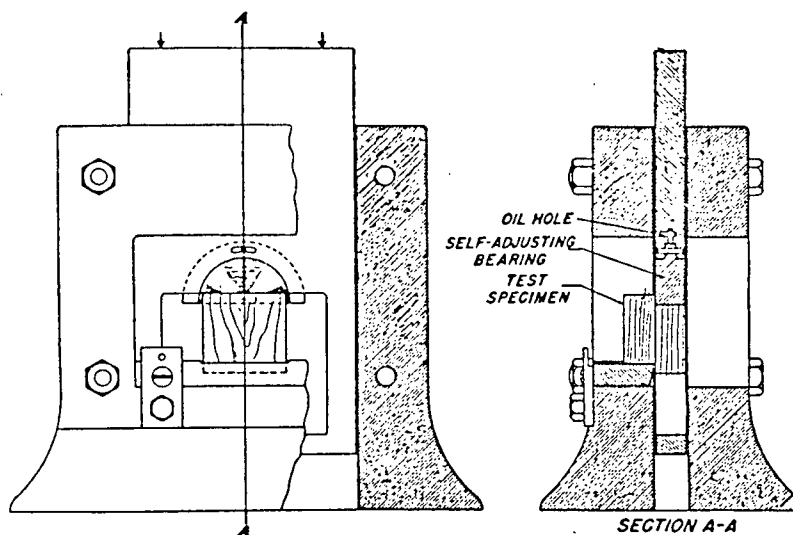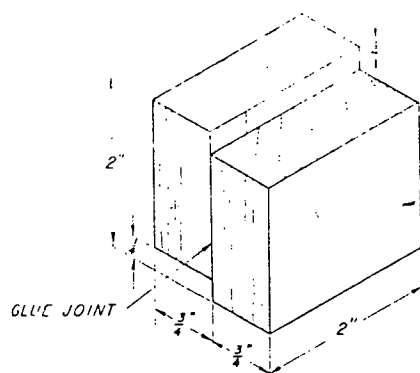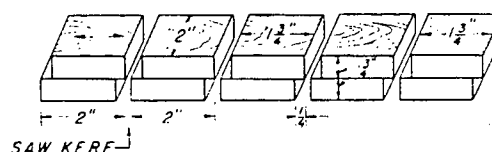
[2] *Annual Book of ASTM Standards,* Vol 04.09.

FIG. 1 Shearing Tool

Metric Equivalents

in.	¼	¾	1¾	2
mm	6.3	19	44.4	50.8

FIG. 3 Test Joint Showing Method of Cutting Five Test Specimens

Metric Equivalents

in.	¼	¾	1¾	2
mm	6.3	19	44.4	50.8

FIG. 2 Form and Dimensions of Test Specimen

APPENDIX

(Nonmandatory Information)

X1. METHOD FOR SELECTING MAPLE BLOCKS OF SATISFACTORY SPECIFIC GRAVITY

X1.1 Cut the hard maple blocks into some standard size such as 19 by 63.5 by 304 mm (¾ by 2½ by 12 in.) after conditioning. Measure the linear dimensions of the blocks in inches, using a suitable caliper or other measuring device. Determine the percentage moisture content of the blocks in accordance with Sections 122 to 125 of Methods D 143. Do not use these samples in further tests.

tool so that the load may be applied as described in Section 3. The position of the specimen in one type of shearing tool is shown in Fig. 1. Apply the loading with a continuous motion of the movable head at a rate of 0.37 mm (0.015 in.)/ min to failure as prescribed in Section 3.

9. Calculation

9.1 Calculate the shear stress at failure in pounds-force per square inch (or kilopascals) based on the glue line area between the two laminations measured to the nearest 0.06 cm^2 (0.01 in.^{2}), and report for each specimen together with the estimated percentage of wood failure.

10. Report

10.1 The report shall include the following:

10.1.1 Complete identification of the adhesive tested, including type, source, manufacturer's code numbers, form, etc.

10.1.2 Application and bonding conditions used in preparing the specimens.

10.1.3 Conditioning procedure used for the specimens.

10.1.4 Temperature and relative humidity in the test room.

10.1.5 Number of specimens tested.

10.1.6 Number of joints represented.

10.1.7 Maximum and minimum shear stresses at failure and percentages of wood failure. The standard deviation or all individual test values, or both, for the failing load values and wood failure values may be included in the report at the option of either the purchaser or the manufacturer of the adhesive.

10.1.8 The average shear stress at failure and the average percentage of wood failure.

TABLE 1 Factors for Corrected Weight in Grams

Moisture Content, %	Factor[a]
4	10.83
5	10.88
6	10.92
7	10.96
8	11.00
9	11.03
10	11.08
11	11.12
12	11.15
13	11.20
14	11.23
15	11.27

[a] These values are the weights of 1 in.^{3} in sugar maple in grams, at the moisture content values indicated, which would have a sp gr of 0.65 on the oven-dry weight and volume basis.

Weigh all blocks to be used in the test at this moisture content; do not use those blocks having a weight less than the above calculated value.

X1.2 Calculate the volume of the blocks to the nearest 1 cm³ (0.1 in.³) and express the average percentage moisture content to the nearest whole number. Determine the numerical value of the factor for this moisture content by reference to Table 1, which is based on hard maple blocks that have a specific gravity of 0.65 on the oven-dry weight and volume basis and

which were conditioned to various moisture content values.

X1.3 Multiply the factor in Table 1 by the volume of the block in cubic inches to obtain the weight of the block in grams which would have a specific gravity of 0.65 on the oven-dry weight and volume basis.

APPENDIX III.

 Designation: D 1107 – 84

Standard Test Method for
ALCOHOL-BENZENE SOLUBILITY OF WOOD[1]

This standard is issued under the fixed designation D 1107; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the alcohol-benzene soluble content of wood, which is a measure of the waxes, fats, resins, and oils, plus tannins and certain other ether-insoluble components.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 4.1.1.

2. Significance and Use

2.1 The alcohol-benzene extract of wood consists of the soluble materials not generally considered part of the wood substance. They are primarily the waxes, fats, resins, and some gums, as well as some water-soluble substances. The amounts are influenced by seasoning and drying.

3. Apparatus

3.1 *Filtering Crucibles*—Alundum or fritted-glass filtering crucibles of coarse porosity.

3.2 *Extraction Apparatus*—A compact form of Soxhlet extraction apparatus, with ground-glass joints, is preferable. The apparatus shall consist of the following items:

3.2.1 *Soxhlet Extraction Flask* having a capacity of 250 mL.

3.2.2 *Soxhlet Extraction Tube*, 45 to 50 mm in inside diameter, having a capacity to the top of the siphon of approximately 100 mL and a siphon tube approximately 55 mm in height. Extraction tubes of these dimensions siphon

more rapidly than extractors with higher siphon tubes.

3.2.3 *Condenser* of the Hopkins inner-cooled type.

4. Reagent

4.1 *Alcohol-Benzene Solution*—Mix 1 volume of ethyl alcohol (95 %) and 2 volumes of chemically pure benzene.

4.1.1 **Caution**—This method requires the use of benzene. It has been established that exposure to benzene may present a serious health hazard to humans. U. S. Government regulations administered by the Occupational Safety and Health Administration are in effect which prescribe rules and regulations on the use of benzene. These regulations must be consulted before experimental programs employing benzene are undertaken.

5. Test Specimen

5.1 The test specimen shall consist of 2 g of air-dried sawdust that has been ground to pass a 425-μm sieve and be retained on a 250-μm sieve.

6. Procedure

6.1 Weigh two 2-g test specimens in tared Alundum or fritted-glass crucibles. Dry one specimen in an oven for 2 h at 100 to 105°C, then place in a loosely stoppered weighing bottle, cool in a desiccator, and weigh. Continue the drying for 1-h periods until the weight is constant. Cal-

[1] This test method is under the jurisdiction of ASTM Committee D-7 on Wood and is the direct responsibility of Subcommittee D07.14 on Chemical Tests.

Current edition approved April 27, 1984. Published June 1984. Originally published as D 1107 – 50. Last previous edition D 1107 – 56 (1979).

culate the proportions of moisture-free sawdust in the air-dry specimen.

6.2 Place the other specimen in a Soxhlet extraction apparatus having a tared Soxhlet extraction flask. Set a small cone of fine-mesh screen wire in the top of the crucible to prevent loss of specimen. Extract with 150 mL of alcohol-benzene solution for 6 to 8 h, keeping the liquid boiling briskly. This should provide four to six siphonings per hour.

6.3 After evaporating the solvent from the extraction flask, dry the flask and contents in an oven for 1 h at 100 to 105°C, cool in a dessicator, and weigh. Continue the drying until there is no further loss in weight.

7. Calculation and Report

7.1 Report the results as weight percentage of alcohol-benzene soluble matter in the moisture-free wood, calculated as follows:

Alcohol - benzene soluble matter, %

$$= (W_2/W_1P) \times 100$$

where:

W_2 = weight of dried extract, 6.3,

W_1 = weight of test specimen used in 6.2, and

P = proportion of moisture-free wood in the air-dry specimen (7.1).

7.2 The results shall be based on the average of at least two determinations.

8. Precision and Bias[2]

8.1 Data obtained in a round robin test involving five laboratories indicate a repeatability of 11 % and a reproducibility of 20 %.

8.2 Bias is unknown.

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[2] Data for this section obtained by the Techncial Association of the Pulp and Paper Industry, P.O. Box 105113, Atlanta, GA 30348.

ASTM Designation: D 1110 – 84

Standard Test Methods for
WATER SOLUBILITY OF WOOD[1]

This standard is issued under the fixed designation D 1110; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods[2] cover the determination of the water solubility of wood. Two methods are given, as follows:

1.1.1 *Method A—Cold-Water Solubility*—This method provides a measure of the tannins, gums, sugars, and coloring matter in the wood.

1.1.2 *Method B—Hot-Water Solubility*—This method provides a measure of the tannins, gums, sugars, coloring matter, *and starches* in the wood.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Significance and Use

2.1 Cold water removes a part of such extraneous materials as tannins, gums, sugars, and coloring matters. Hot water removes these *plus* the starches.

METHOD A—COLD-WATER SOLUBILITY

3. Apparatus

3.1 *Filtering Crucibles*—Alundum or fritted-glass crucibles of coarse porosity will be required.

3.2 *Filtering Flask*—A suction filtering flask, equipped with a rubber flange for the crucible and funnel, shall be provided.

4. Test Specimen

4.1 The test specimen shall consist of 2 g of air-dried sawdust that has been ground to pass a 425-μm sieve and be retained on a 250-μm sieve.

5. Procedure

5.1 Place a 2-g test specimen, the moisture content of which has been previously determined, in a 400-mL beaker, and cover with 300 mL of distilled water. Let this mixture digest at a temperature of 23 ± 2°C, with frequent stirring, for 48 h.

5.2 Filter the material on an Alundum or fritted-glass crucible, using suction, wash with cold distilled water, and dry to constant weight at 100 to 105°C. Drying usually requires approximately 4 h. Place the crucible in a loosely stoppered weighing bottle, cool in a desiccator, and weigh.

6. Report

6.1 Report the results as percentage of matter soluble in cold water, on the moisture-free basis, calculated as follows:

$$\text{Cold water solubility, \%} = [(W_1 - W_2)/W_1] \times 100$$

where:

W_1 = weight of moisture-free specimen used in 5.1, and

W_2 = weight of dried specimen after extraction with cold water (5.2).

[1] These test methods are under the jurisdiction of ASTM Committee D-7 on Wood and are the direct responsibility of Subcommittee D07.14 on Chemical Tests.

Current edition approved April 27, 1984. Published June 1984. Originally published as D 1110 – 50 T. Last previous edition D 1110 – 56 (1977).

[2] For further information on these test methods, the following references may be consulted:

Schorger, A. W., "Chemistry of Cellulose and Woods," 1926, p. 506, McGraw-Hill, N.Y.

Hawley and Wise, "Chemistry of Wood," 1926, p. 134, Chemical Catalog Co., N.Y.

METHOD B—HOT-WATER SOLUBILITY

7. Apparatus

7.1 *Digestion Apparatus*—A 200-mL Erlenmeyer flask provided with a reflux condenser shall be used.

7.2 *Water Bath*, so constructed that the water can be maintained at boiling temperature and at a constant-level just above the solution in the flask.

7.3 *Filtering Crucible and Filtering Flask*—See Section 3.

8. Test Specimen

8.1 See Section 4.

9. Procedure

9.1 Place a 2-g test specimen, the moisture content of which has been previously determined, and 100 mL of distilled water in the Erlenmeyer flask and attach the reflux condenser. Place the flask in the boiling water bath, with the solution in the flask just below the level of the water in the bath, and heat gently for 3 h.

9.2 Filter the contents of the flask on a tared Alundum or fritted-glass crucible, using suction, wash with hot water, and dry to constant weight

at 100 to 105°C. Place the crucible in a loosely stoppered weighing bottle, cool in a desiccator, and weigh.

10. Report

10.1 Report the results as percentage of matter soluble in hot water, on the moisture-free basis, calculated as follows:

Hot water solubility, $\% = [(W_1 - W_2)/W_1] \times 100$

where:

W_1 = weight of moisture-free specimen used in 9.1, and

W_2 = weight of dried specimen after extraction with hot water (9.2).

11. Precision and Bias[3]

11.1 All data obtained in one laboratory by testing 20 woods.

	Repeatability, as Solubility, %	Repeatability, % of Solubility
Cold (1.1 to 6.3 %)	0.14	5.7
Hot (1.6 to 9.0 %)	0.15	3.8

11.2 Reproducibility and bias are unknown.

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[3] Data in this section obtained from the Technical Association of the Pulp and Paper Industry, P.O. Box 105113, Atlanta, GA 30348.

Designation: D 1109 – 84

Standard Test Method for
1 % SODIUM HYDROXIDE SOLUBILITY OF WOOD[1]

This standard is issued under the fixed designation D 1109; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the solubility of wood in a hot dilute alkali solution. A 1 % solution of sodium hydroxide (NaOH) is used. One application is in determining the degree of fungus decay that has taken place in a given wood sample. As the wood decays, the percentage of alkali-soluble material increases in proportion to the decrease in pulp yield caused by the decay.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Significance and Use

2.1 Hot alkali extracts low molecular weight carbohydrates consisting mainly of hemicellulose and degraded cellulose in wood. This solubility of wood is an indication of the degree of fungal decay, or degradation by heat, light, oxidation, etc. The more decay or degradation, the higher the solubility.

3. Apparatus

3.1 *Water Bath*—The water bath shall be designed so that the temperature of the material during treatment is uniformly maintained at 97 to 100°C. When a new bath is used the temperature shall be checked to ensure the use of proper conditions. The type of bath recommended is one that is covered and that has holes in the top of such size that beakers may be set down in the bath until they are supported by the flared rim of the beakers. The top of the beaker shall be

nearly level with the cover of the bath. By using this type of bath the sides of the beakers are entirely surrounded by boiling water or steam. The water level in the bath shall be maintained above the level of the liquid in the beakers.

3.2 *Beakers*—The beakers shall be tall-form, 200-mL, alkali-resistant glass[2] beakers.

3.3 *Filtering Crucibles*—Alundum or fritted-glass crucibles of medium porosity are recommended for filtering the treated sawdust.

4. Reagents

4.1 *Sodium Hydroxide Solution (1.0 %)*—Allow a chemically pure NaOH solution (50 %) to stand about 1 week in a stoppered vessel to permit settling of Na_2CO_3 and other insoluble impurities. Dilute the supernatant clear solution with distilled water free of CO_2 and adjust to between 0.9 and 1.1 % NaOH.

4.2 *Acetic Acid (10 %)*.

5. Test Specimen

5.1 The test specimen shall consist of air-dried sawdust that has been ground to pass a 425-μm sieve and be retained on a 250-μm sieve. The weight of the test specimen shall be such that it will be equivalent to 2 ± 0.1 g of moisture-free wood.
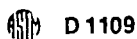
6. Procedure

6.1 Place two test specimens in 200-mL, tall-form beakers and add to each 100 mL of NaOH

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solution (1 %) measured carefully with a graduate. After stirring well, place the covered beakers in the water bath, which shall be boiling steadily. Leave the beakers in the bath for exactly 1 h, stirring the contents three times, at periods of 10, 15, and 25 min after the beakers are placed in the bath.

6.2 At the end of 1 h, filter the contents of each beaker by suction on a tared crucible. Wash the sawdust with 100 mL of hot water, then with 50 mL of acetic acid (10 %), and then thoroughly with hot water. Dry the crucible and contents to constant weight at 100 to 105°C, cool in a desiccator, and weigh in a stoppered weighing bottle.

7. Calculation and Report

7.1 Report the results as weight percentage of matter soluble in 1 % sodium hydroxide solution, on the moisture-free basis, calculated as follows:

Matter soluble in caustic soda, %

$$= [(W_1 - W_2)/W_1] \times 100$$

where:
W_1 = weight of moisture-free wood in specimen prior to test (Section 5), and
W_2 = weight of dried specimen after treatment with the NaOH solution (6.2).

7.2 Base the results on the average of at least two determinations.

8. Precision and Bias[3]

8.1 Results obtained from an interlaboratory study by nine laboratories on four woods indicate a repeatability of 0.45 and reproducibility of 1.96. The solubility of the wood samples ranged from 11.2 to 17.0 %.

8.2 Bias is unknown.

[3] Data in this section obtained by the Technical Association of the Pulp and Paper Industry, P.O. Box 105113, Atlanta, GA 30348.