

Fast vs. slow-reacting non-modified tannin extracts for exterior particleboard adhesives

A. Pizzi, A. Stephanou

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Non-fortified, non-modified tannin extracts, rather than chemically-modified tannin adhesives intermediates, can be used as effectively to produce excellent exterior grade particleboard at fast pressing times of industrial significance, with considerable advantages in both handling and cost. This can be achieved by simple pH-controlled reactivity adjustments of the tannin extract in the glue mix. This new concept is shown to be applicable to all types of polyflavanoid tannin extracts: Pine, mimosa, pecan, quebracho, and gambier tannin extracts are compared as regards their performance and capabilities. The new concept applied to the faster-reacting tannins, such as pine tannin and pecan tannin extracts, is shown to produce fast hot-pressing rates and excellent high moisture content tolerances which are undreamed of for the slower-reacting tannins, modified or non-modified, which are traditionally associated with thermosetting wood adhesives. Industrially used techniques to control well viscosity and pot-life of the glue-mixes of accelerated tannin adhesives are also presented.

Schnell- statt langsam-reagierende, nicht-modifizierte Tanninextrakte zum Verleimen von Spanplatten für die Außenverwendung

Nicht-verstärkte und unmodifizierte Tanninextrakte können anstelle von chemisch modifizierten Tanninen zur Herstellung hochwertiger Spanplatten für die Außenverwendung eingesetzt werden. Sie ermöglichen kurze Preßzeiten und bieten sowohl auf der Kostenseite als auch in der Handhabung bedeutende Vorteile. Dies kann durch einfache pH-Steuerung der Reaktivität des Tanninextraktes im Klebstoffgemisch erreicht werden. Das neue Konzept konnte für alle Typen von Polyflavonid-Tanninen bestätigt werden. So wurden Tanninextrakte aus Kiefern, Mimosa, Hickory, Quebracho und Gambir (Katechu) auf ihre Verwertbarkeit untersucht. Mit den schnell-reagierenden Tanninen aus Kiefern und Hickory konnten sehr kurze Preßzeiten und ausgezeichnete Feuchtetoleranzen gegenüber den Spänen erzielt werden, die bisher bei herkömmlichen, langsam-reagierenden Tanninlebern unvorstellbar waren. Weiterhin werden industrielle Verfahren zur Kontrolle der Viskosität und der Topfzeit der Klebstoffgemische aus diesen beschleunigt reagierenden Harzen vorgestellt.

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Introduction

Tannin-based adhesives for exterior grade particleboard based on mimosa tannin extract and to a lesser extent on quebracho Tannin extract have been in commercial operation in several Southern Hemisphere countries since 1971 (Pizzi 1991). The adhesives based on these two tannins are much slower reacting with formaldehyde than the newer generation of adhesives based on the faster reacting procyanidin/prodelphinidin-type tannins such as pine bark and pecan nut pith tannin extracts, introduced since

1990 (Pizzi 1991; Pizzi et al. 1993). The structural reasons for the differences in reactivity towards formaldehyde of the two slow reacting tannins, mimosa and quebracho, and two faster-reacting tannins, pine and pecan, are well known. They are based on the structure of the A-rings of the majority of flavonoid units composing the two sets of tannins: resorcinol-like in mimosa and quebracho, phloroglucinol-like in pine and pecan nut tannins. (Pizzi 1983; Pizzi and Stephanou 1993a). Their reactivity has always been thought to put the fast tannins at a disadvantage in relation to the slower-reacting tannins traditionally used for wood adhesives (Pizzi 1983). This because hardening reactions under industrial conditions were thought to be not easily controllable, leading to problems of precuring, and particularly because of the traditional pHs of operation of tannin adhesives, these because of the pot-life of the adhesive glue mix would be too short (Pizzi 1983). Furthermore, because the main tannin adhesive based on mimosa was based on structural modifications of the tannin extract itself (Pizzi 1978), without any synthetic resins fortification the ineffectiveness of such modifications on the fast tannins appeared to deny their use for lower-cost nonfortified exterior grade adhesives.

This paper describes instead how the high reactivity of these tannins permits application feats in wood adhesives which are impossible to achieve with the slower, traditional tannins and introduces the solutions now used industrially to overcome the problem of pot-life of the glue-mix which has been perceived as a blocking problem in the industrial exploitation of the fast tannins. It also addresses and corrects the misconception that any flavonoid tannin slower or faster-reacting, needs to be chemically or structurally modified, or fortified by addition of synthetic resins, to perform as an excellent thermosetting wood adhesive.

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Experimental

2.1

Gel time and reactivities

Gel time of the reaction of tannin extracts with formaldehyde are an indication of the reactivity of the tannin sites and hence of some of the likely structural characteristics of the tannin repeating units important for the preparation of adhesives and resins (Pizzi and Stephanou 1993; Pizzi et al. 1993e). The gel time dependance on pH at 94°C was determined over the pH range 4, 5 to 10 for five commercial tannin extracts, namely mimosa (*Acacia Mearnsii*, de Wildt) bark extract, quebracho (*Schinopsis balansae*) would extract, pine (*Pinus radiata*) bark extract, pecan (*Carya illinoensis*) nut pith extract and Gambier (*Uncaria Gambir*) shoots extract and for two modified, non-fortified, tannin adhesives (Pizzi and 1978; Pizzi and Stephanou 1993b) based respectively on mimosa and quebracho tannins. The results are shown in Figs. 1 and 2. Gelation is defined as the point at which the polycondensates formed by the reaction of tannin extract and formaldehyde ceases to be a viscous liquid and becomes a soft, elastic, rubbery solid. 10 g of liquid tannin extract 40% (quebra-

Table 1. One layer laboratory particleboard results of different tannin extracts and tannin adhesives intermediates at their pHs of equivalent reactives, at press time of 10 seconds/milimetre board thickness

| Tannin binder type | pH | Board density (kg/m ³) | I.B. dry (MPa) | I.B. V100 (MPa) |
|--------------------|-----|------------------------------------|----------------|-----------------|
| Mimosa Adhesive | 7.7 | 703 | 0.76 | 0.25 |
| Mimosa Extract | 8.3 | 700 | 0.83 | 0.25 |
| Quebracho Adhesive | 8.4 | 698 | 0.78 | 0.25 |
| Quebracho extract | 9.5 | 702 | 0.66 | 0.20 |
| Pine Extract | 5.7 | 700 | 0.78 | 0.23 |
| Pecan Extract | 4.8 | 702 | 0.87 | 0.32 |

cho, pine, pecan) and 50% (mimosa) water solution is weighed in a glasstube. 7.5% by mass of fine powder 96% paraformaldehyde based on tannin extract solids is then added to the test tube. A wire spring is placed in a test tube and the paraformaldehyde gently mixed at ambient temperature into the tannin extract or tannin adhesive intermediate solution. The test tube is then placed in a boiling water bath (at boiling point = 94°C, location altitude = 2000 m on sea level), the wire spring is moved rapidly up and down in the tube, and the time taken to reach gel point measured using a stopwatch. The end point at gel is very sharp and easily sensed in the case of tannins. The test is done in duplicate. The gel time measurement is used to relate to the speed of gelling of the tannin/paraformaldehyde mixture under actual application conditions.

2.2

Glue mixes and particleboard preparation

The glue mixes were prepared by adding 10% paraformaldehyde to 40% solutions of the tannin extract or adhesive intermediates at the pH indicated in Tables 1 and 2. Duplicate single layer pine wood chips 350 × 300 × 12 mm laboratory particleboard were prepared with the glue mixes described to obtain 10% tannin extract solids load on dry wood particles. Moisture contents of 20–22% of the resinated wood particles were used for the panels in Tables 1 and 2, except for pecan where moisture contents of 26% were used. The pressing times used were of 15 sec/mm and 10 sec/mm board thickness for all the cases reported in the Tables. The pressing temperature used was of 195°C. The pressing cycles used were for 15 sec/mm: 5 sec/mm from platen contact to high pressure (24 kg/cm²) and higher pressure maintenance, plus 2.5 sec/mm at an intermediate pressure of 10–12 kg/cm², plus 7.5 sec/mm at a low pressure of 2 kg/cm². For 10 secs/mm: 5 sec/mm from platen contact to max pressure and higher pressure maintenance, plus 5 sec/mm at a low pressure of 2–3 kg/cm². All the boards prepared were tested for internal bond (I.B) strength dry

and after 2 hours boiling tested wet (V100 test, DIN 68763). The results obtained are shown in Tables 1 and 2.

3 Discussion

In Figs. 1 and 2 are shown the results of gel times at different pHs with formaldehyde, at 94°C, for concentrated water solutions of commercial tannin extracts usable for wood adhesives. The gel times of two non-fortified but modified tannins solutions intermediates based on treatments already presented (Pizzi 1978) and clarified (Pizzi and Stephanou, 1993a–c) are also shown: these are the segmented curves indicated as mimosa adhesive (Figs. 1 and 2). In many applications of the faster-reacting tannins (Pizzi and Stephanou 1993; Pizzi et al. 1993f) indications have occurred several times that only the reactivity, hence the rate of the reaction of tannin with formaldehyde, is the performance-determining factor for tannins as thermosetting wood adhesives. This implies that for all polyflavanoid tannins it is of no or little final importance if modifications to the tannin structure are, (Pizzi and Stephanou 1993b,c) or are not, made. What is important is that the correct reactivity, hence the correct rate of curing, be obtained. This is easier to achieve by simply varying the pH of a non-modified extract than to rather go to the expense and bother of chemically modifying the tannin extract to form an “adhesive intermediate”. To test this hypothesis a pH at which an established modified tanning adhesive is used, was chosen; mimosa tannin adhesives operate, according to the technology involved and the factory using it, in the pH rang 6.3 (Pizzi 1978) to 8.2 (Van Niekerk 1990, 1993; Huber 1993; Pizzi et al. 1993e) at present. Thus the reactivity at pH 7.7 for a non-fortified but modified (Pizzi 1978) mimosa tannin adhesive (Table 1) was chosen as a reference for reactivity. The same reactivity (hence the same gel time) is observed from Figs. 1 and 2 to occur at pHs of respectively 8.3, 9.5, 5.7 and 4.8 for the plain non-modified mimosa, quebracho, pine and pecan nut tannin extracts and pH

Table 2. One layer particleboard results of different tannin extracts and tannin adhesives intermediates at reactivities higher and lower than comparative extracts. (Moisture content of resinated chips = 22%; press temperature = 195°C, max pressure = 24 kg/cm²)

| Tannin binder type | pH | Press Time = 155/mm | | | Press Time = 105/mm | | |
|--------------------|------|------------------------------------|----------------|-----------------|------------------------------------|----------------|-----------------|
| | | Board density (kg/m ³) | I.B. dry (MPa) | I.B. V100 (MPa) | Board density (kg/m ³) | I.B. dry (MPa) | I.B. V100 (MPa) |
| Pine extract | 4.1 | – | – | – | 691 | 0.42 | 0.11 |
| | 7.1 | 745 | 0.76 | 0.18 | 744 | 0.55 | 0.18 |
| | 7.7 | 728 | 0.91 | 0.24 | 721 | 0.80 | 0.24 |
| Pecan nut extract* | 6.6 | 716 | 0.91 | 0.45 | 699 | 0.90 | 0.42 |
| Mimosa extract | 7.7 | 717 | 0.52 | 0.18 | – | BLOWS | – |
| | 8.5 | 725 | 0.93 | 0.29 | 714 | 0.87 | 0.27 |
| Quebracho extract | 7.7 | – | BLOWS | – | – | BLOWS | – |
| | 8.6 | 705 | 0.43 | 0.15 | – | BLOWS | 0.22 |
| | 10.3 | – | – | – | 718 | 0.68 | – |

* Moisture content of resinated chips = 26%

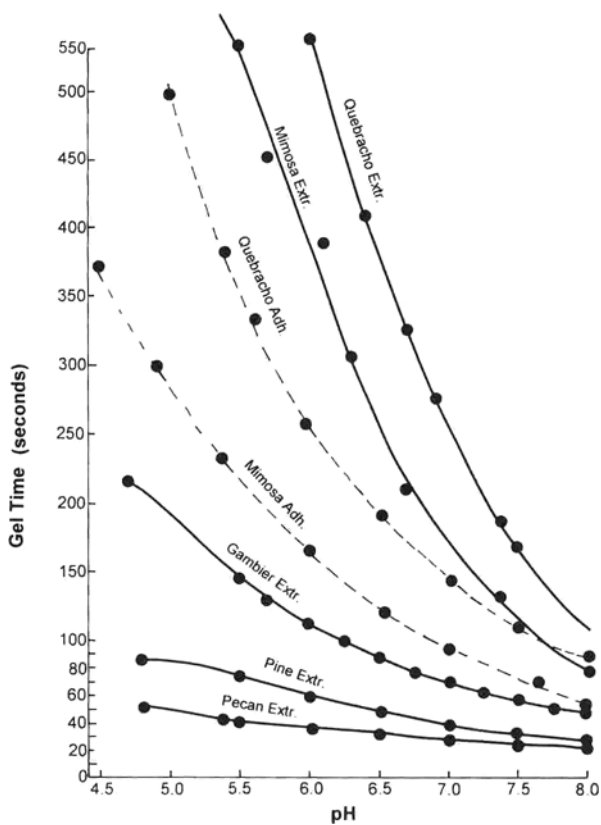


Fig. 1. Gel times in function of pH of five non-modified tannin extracts and two modified tannin adhesive intermediates (segmented lines) in the pH range 4.5 to 8.0

Bild 1. Gelierzeiten in Abhängigkeit vom pH-Wert für fünf nichtmodifizierte Tanninextrakte und zwei modifizierte Tanninkleber-Zwischenstufen (gestrichelt) zwischen pH 4, 5 und 8

8.4 for the modified quebracho adhesive intermediate. These are the pHs and reactivity at which all the particleboard presented in Table 1 were prepared. Pressing times as fast as 10 seconds/millimeter thickness of the panels at moisture contents of 22% were used. It is interesting to note that at the pHs of equal reactivity there is hardly any difference in the internal bond strength (I.B.)

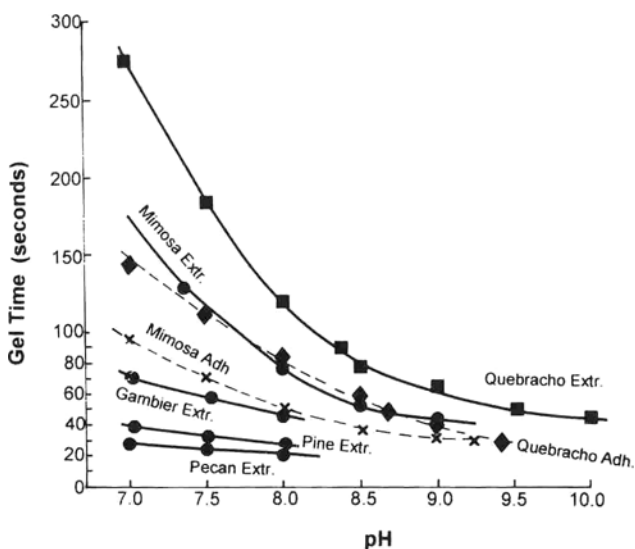


Fig. 2. Gel times in function of pH of five non-modified tannin extracts and two modified tannin adhesive intermediates (segmented lines) in the pH range 7.0 to 10.0

Bild 2. Gelierzeiten in Abhängigkeit vom pH-Wert für fünf nichtmodifizierte Tanninextrakte und zwei modifizierte Tanninkleber-Zwischenstufen (gestrichelt) zwischen pH 7 und 10

results, particularly in the I.B. strength after 2 hours boiling conducted according to the DIN 68763 (V 100) test (Table 1). Thus, there is no difference between a particleboard made with a modified (more expensive) tannin adhesive and just the non-modified, plain tannin extract if its reactivity towards formaldehyde has been adjusted (by pH) to be the same. The only two exceptions to this trend are the pecan nut tannin extract which presents better results, an occurrence depending on the presence of additional cross-linking reactions already described (Pizzi and Stephanou 1993a, b) and the quebracho extracts which is known to be slow reacting.

The results in Table 1 infer that modification of the tannin extract, as done for the slower reacting tannins (Pizzi 1978a, b) such as mimosa and quebracho, is not necessary. To further illustrate this concept, the results presented in Table 2 are of particular interest. In Table 2 comparing pine at pH 7.7 and pecan at pH 6.6 yields identical V100 IB strength, this because the reactivity towards formaldehyde at pH 6.6 is identical to that of pine tannin at pH 7.7. Equally the reactivity of the modified mimosa adhesive at pH 7.7 is equivalent to that of the quebracho adhesive of pH 8.4 (Table 1), and so are the I.B. strengths (Table 1). The same clear trend is noticeable with the non-modified slow extracts where mimosa extract at pH of 7.7 yields similar results of quebracho extract at pH of 8.6.

Both mimosa and quebracho extract at these pHs show considerably more sensitivity to high moisture contents when the faster press times are used. Of particular interest in Tables 1 and 2 is the behaviour of pecan nut tannin extract: the exceptional results obtained (I.B. V100) cannot be simply ascribed to the existing higher reactivity of this tannin. It is quite evident that the novel auto-condensation reaction of this tannin, already partly reported (Pizzi and Stephanou 1993b), greatly contributes to the excellence of the pecan tannin extract results. Such auto-condensation appears to occur, to a much more limited extent, also with mimosa extract judging from the results at pH 8.5 in Table 2. Such auto-condensation reaction is promoted by the high proportion of pyrogallol B-rings in the pecan and mimosa tannins, while it clearly does not contribute in pine and quebracho extracts in which catechol is the exclusive or predominant grouping of the B-ring. Thus, such mechanism is an additional source of I.B. strength in tannins containing a high proportion of prodelphinidins rather than procyanidins, or a high proportion of prorobinetidin rather than profisetinidin. It also justifies the somewhat slightly lower than expected results with quebracho tannin (Table 1).

A few important conclusions can then be drawn:

- (i) Reactivity towards formaldehyde, hence gel time, is all what counts in preparing a tannin adhesive of excellent performance under a given set of application conditions.
- (ii) It is unnecessary to chemically modify the tannin extract as it has now been done for decades (Pizzi 1978a,b) to prepare for instance a mimosa tannin adhesive intermediate. Consequently, modification exclusively determined by model compounds, such as the phlobatannin rearrangement, which have been mistakenly believed to be potentially important in adhesive application (Roux 1989,1992), become instead rather inconsequential to the field of thermosetting tannin adhesives for wood. In most cases it appears that equally good particleboard can be produced by just adjusting the pH of the non-modified tannin extract, in the glue mix, at ambient temperature, to the wanted reactivity. This is valid for all the five tannin extracts investigated.
- (iii) There is no doubt, that in the quest for faster pressing times and improved tolerance to higher moisture contents of the resinated wood furnish the fast-reacting tannins, pine and pecan,

have considerable advantages and flexibility over the slower-reacting ones such as mimosa and quebracho. It is evident from Fig. 2 that even pushing reactivity to its limit even the structurally modified (hence "faster") mimosa and quebracho adhesives can never hope to reach gel-times as short and reactivity as fast as those achievable with pine and pecan nut tannin extracts. This becomes even more evident (Fig. 2) when the slower non-modified tannin extract are compared to the faster ones.

As regards point (iii) above, the curves in Fig. 2 and the results in Tables 1 and 2 indicate then that the results obtainable with pine at pH 7.7 or higher cannot easily be matched by mimosa or quebracho even at over pHs of 8.5 and 10 respectively, where their gel times curves finally flatten out.

Very significant are also the results in Table 2 where laboratory boards bonded with non-modified mimosa extract at pH 7.7 and quebracho extract of pH 8.6 cannot press at 10 sec/mm at percentage moisture contents of the furnish as high as 22% blowing at this high moisture content levels. The results in Table 2 also show that pecan nut tannin extract instead allows pressing times of 10 sec/mm at a moisture content of resinated wood particles of 26% with results which are simply exceptional.

It is clear from the above that only three conditions can then limit industrial application of such a new concept. These are:

- 1) The pot-life of the glue mix
- 2) The viscosity of the glue mix
- 3) The rate of moisture elimination in the form of vapour from the board during pressing.

The first point is easily addressed: it is the combination of the tannin extract with the formaldehyde at the pH needed which is the cause of the problem. It is then sufficient to subtract just one of these elements from the glue mix and add it separately to solve with ease such a problem. This is already done in several of the factories in which tannin adhesives have been used or tried. In some factories the problem is solved by adding separately paraformaldehyde fine powder, by means of a small screw conveyor, to the wood particles before the glue blender in which the liquid tannin extract solution at high pH is blended to the furnish (Van Niekerk 1990, 1993; Pizzi 1979). In other factories instead the liquid glue mix is composed of a solution of the tannin extract at its minimum pH of reactivity (4–4.5, generally the tannin natural pH) mixed with paraformaldehyde powder hardeners: this mix has a pot-life in excess of 30 hours. A measured stream of diluted sodium hydroxide is, however, also added directly to the glue blender through a separate line (Huber 1993), or by mean of a short static mixer to the line carrying the liquid glue mix, just a few centimetres before the glue blender (Dunky 1993) to adjust reactivity. All these are easy industrial practices, the two former ones in daily industrial use now for several years.

The viscosity of the glue mix also does not constitute a problem. On top of factory techniques already reported and used for a long time (Pizzi 1979), several others are now in use. The chemical modification of tannin extracts was originally prompted by the need to have lower viscosity at the highest solid content possible (Pizzi 1978, 1983). With the introduction of the accelerated reaction of tannins induced by higher pHs the faster development of bond strength, at an earlier stage of the cycle during hot-pressing of the board, ensures that the high pressure steam generated within the board at higher moisture contents is not capable of breaking the strong bonds already formed. In short, disruption by steam emission of bonds while they are being formed is minimised. Thus, excess moisture content does not constitute anymore a problem, viscosity can easily be adjusted by adding water, dropping the solids content of the adhesive mix (Valenzuela et al. 1991) (as low as 30% for pecan, and as low as 35% for pine tannin extract): This is possible because these bond-

ing systems have now become very tolerant of very high moisture contents (Table 2). Another system already used industrially (Valenzuela et al. 1991) is to warm the liquid glue mix up to 30°–35°C: its viscosity does then decrease precipitously. Pot-life is not affected because the liquid glue mix does not contain the paraformaldehyde hardener, or if it does, the tannin is at its pH of minimum reactivity anyhow (see above).

The only limiting factor in how fast a pressing time can be achieved is then the rate of elimination of moisture from the board during hot-pressing: after all no one wants too high a moisture content of the finished panel as this would cause dimensional instability, warping and other problems. A particle-board manufacturer using a variant of the high pH system illustrated in this article, uses already moisture contents of the resinated particles in excess of 20% at standard press times of 9 sec/mm (190°C); press times as fast as 7 sec/mm have already been achieved (Van Niekerk 1990, 1993), and these with one of the slower reactivity tannins. It is clear from what has been presented that by the use of higher pHs and of the fast curing tannins, it is quite likely that industrial press times faster than these are achievable. It is also quite clear that the slower reacting tannins could be somewhat upgraded by the addition of percentages of the faster reacting tannins, a fact evident from the curves in Figs. 1 and 2 without the need for any "homogenizing" (Roux 1992) of the different tannins and tannin fractions, a practice which might actually be harmful to final performance. Furthermore, if "homogenizing" of reactivities, the only property where problems might occur, between mimosa and quebracho tannins is needed (Roux 1992) then quebracho adhesive intermediate and plain mimosa extract, the reactivity curves of which are at the needed pHs almost coincident, can be mixed to good effect.

4 Conclusion

In conclusion, non-modified tannin extracts rather than tannin adhesives intermediates can be used as effectively to produce excellent exterior grade particleboards with considerable advantages in both handling and costs. This just by simple pH controlled reactivity adjustments. Furthermore, non-modified faster reacting tannins, such as pine and particularly pecan nut tannins, appear to be able to achieve feats in hot-pressing rate and moisture content tolerance which are undreamed of for the slower-reacting tannins, modified or un-modified, traditionally associated with thermosetting wood adhesives. Lastly, modifications and rearrangements which from model compounds work have been interpreted as being potentially performance-determining for tannin adhesives (Roux 1989, 1992), but the definite yet limited contribution of which has already been placed into its proper perspective by work on the actual polymeric tannins and tannin adhesives (Pizzi and Stephanou 1993b,c), would now anyhow be rendered inconsequential by the new approach presented.

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Kurz-Originalia·Brief Originals

Verbesserte Holznutzung durch neuartige maschinelle Festigkeitssortierung

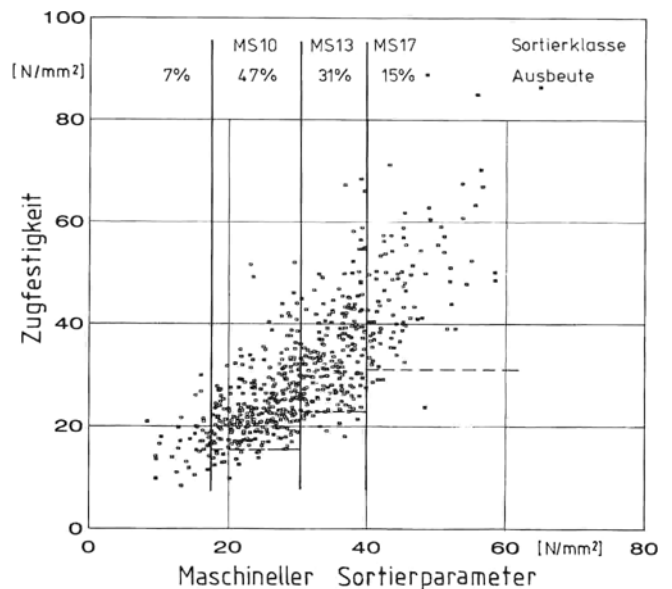
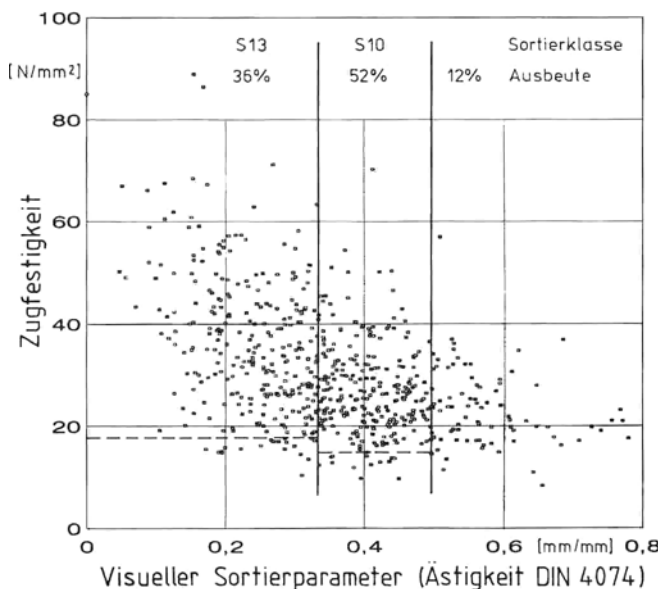
R. Diebold, P. Glos
 Institut für Holzforschung, Universität München, Winzerestr. 45,
 D-80797 München, Deutschland

Subject A new machine strength grading system based on MOE, density and knot measurement provides higher characteristic strength and stiffness values and higher yields in the upper strength classes. This improves the value and competitiveness of timber.

Material und Methode Im Rahmen der Entwicklung eines neuen Verfahrens zur maschinellen Festigkeitssortierung, das die Holzparameter Elastizitätsmodul, Rohdichte und Ästigkeit erfaßt und zur Klassifizierung verwendet, wurde eine Maschine dieses Typs in einem Holzleimbaubetrieb für den Praxiseinsatz installiert. Hier wurden dann 625 Lamellen aus einheimischer und nordischer Fichte mit Breiten von 135–220 mm und Dicken von 33–38 mm bei einer Vorschubgeschwindigkeit von 120 m/min sortiert. Anschließend wurden diese Bretter visuell nach DIN 4074 klassifiziert und die Zugfestigkeit nach EN 408 ermittelt.

Resultate 1. Mit dem neuen, inzwischen in der Praxis eingesetzten maschinellen Sortierverfahren kann Schnittholz bei hohen Sortiergeschwindigkeiten zuverlässig nach seiner Festigkeit klassifiziert werden. 2. Die in den einzelnen Sortierklassen erreichbaren charakteristischen Festigkeitswerte sind bei maschineller Sortierung deutlich höher als bei visueller Sortierung. Die 5%-Fraktilewerte der Zugfestigkeit liegen für MS17 und MS13 bei 30.6 und 22.9 N/mm², gegenüber 17.7 N/mm² bei einer Sortierung des gleichen Holzes in die höchste visuelle Klasse S13. 3. Die Ausbeute in den maschinellen Sortierklassen MS17 und MS13 beträgt 46% und ist damit 10% höher als in der Sortierklasse S13. 4. Bei visueller Sortierung konnten 12% der Hölzer nicht in die Klassen S10 und besser eingestuft werden, bei maschineller Sortierung dagegen nur 7%.

Glos, P.; Diebold, R. 1994: Verfahrensentwicklung und Erprobung zur maschinellen Schnittholzsortierung. Institut für Holzforschung der Universität München, Abschlußbericht 92510



Herrn Prof. Dr. D. Fengel anlässlich seines Ausscheidens aus dem Dienst gewidmet