# How mole ratio of UF resin affects formaldehyde emission and other properties: A literature critique

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**Abstract**

A critical review was made of the literature concerned with how the formaldehyde to urea mole ratio (F/U) affects formaldehyde emission from particleboard and plywood bonded with urea-formaldehyde (UF) adhesives, and how this ratio affects certain other adhesive and board properties. It is difficult to quantify the dependence of various properties on mole ratio or determine lower limits of mole ratio for a particular property because of the range in resin, board, and testing parameters used in the cited studies. However, the available data do at least suggest the following limitations on F/U in conventional UF particleboard systems for maintenance of acceptable properties: 1) F/U < 1.2 or even < 1.1 to meet the German El emission standard, 2) F/U < 1.3 or possibly < 1.2 to meet the tentative NPA emission standard for U.S. mobile homes, 3) F/U ⩾ 1.2 for bending strength and modulus of rupture, 4) F/U ⩾ 1.1 or possibly ⩾ 1.2 for internal bond, 5) F/U ⩾ 1.2 or possibly ⩾ 1.3 for 24-hour thickness swell. These conflicting limitations strongly indicate that problems exist in depending solely on mole ratio reductions to produce low emission UF particleboard with acceptable physical properties.

During the past two decades, an extensive body of literature has developed concerned with the problem of formaldehyde emission from bonded wood products, particularly with those bonded by urea-formaldehyde (UF) adhesives. This literature, composed of patents, reports, and journal articles, describes the results of investigations into the effects of resin and board processing parameters on formaldehyde emission and of investigations into a variety of approaches to reduce emissions. Reviews of some of this literature have appeared at intervals (14, 16, 17). However, because of an accelerating number of studies in recent years and a growing maturity of understanding, a strong need was felt for an up-to-date, critical review of the state of knowledge for some of the more important aspects of the problem.

Accordingly, this paper represents the first in a series of six such critical summaries, each to be concerned with a different aspect of the problem: 1) effects of formaldehyde to urea mole ratio (F/U), 2) ventilation rate and board loading, 3) temperature and humidity, 4) furnish pretreatment, 5) board post-manufacture treatment, and 6) resin hydrolysis. The first five of the critiques are scheduled for completion within the next year and the sixth within the next 2 years. The critiques will be based upon citations in separately compiled bibliographies covering the period from 1960 to the date of each critique's preparation (January 1983 in the present case).[1] The bibliographies in turn will be derived from a variety of sources including Chemical Abstracts, Abstract Bulletin of the Institute of Paper Chemistry, National Technical Information Service, and Pollution Abstracts.

In this review of mole ratio effects, both the data examined and the conclusions drawn therefrom refer, as far as possible, to the influence of that variable in the

[1]Copies of each bibliography may be requested from the Formaldehyde Institute, 1075 Central Park Ave., Scarsdale, NY 10583, after the corresponding critique has been published. Each citation will include keywords and usually a short abstract.

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absence of changes in other resin or board variables that might alter board properties. By the time this review is published, other complementary variables undoubtedly will have been introduced into industrial practice.

### General comments on mole ratio effects

The bibliography (15) compiled for this critique contains 79 citations. Most of the more useful citations deal with particleboard, and unless stated otherwise, the ensuing discussion refers to particleboard, not hardwood plywood or other UF-bonded products. Unfortunately, many of the most relevant citations do not completely specify all the variables that can strongly influence formaldehyde emission and the mechanical properties of particleboard – e.g., resin synthesis details, cure catalyst, press time and temperature, moisture content, board conditioning or aging. One criterion used in selecting data to emphasize in this critique was the degree to which those variables were specified. Fortunately, however, that criterion did not have to be rigidly adhered to since we are interested here primarily in patterns of behavior with changing mole ratio and not necessarily in absolute values of properties. A second criterion, therefore, allowed the use of data that appeared to represent an average or a summary of experience within a given industry or country (13, 29, 30), despite the absence of some specifications. Finally, those citations were emphasized that included data down to lower mole ratios (F/U $\leq$ 1.5), since the range between 1.0 and 1.5 is of primary interest for low emission.

Table 1 lists the citations from which data were actually extracted for this summary based on the above criteria. The table includes information about each study and also lists the relevant symbols used to designate each citation in plots of selected properties versus mole ratio (Figs. 1-5). Note that many of the data points shown in Figures 1 to 5 were read from graphs in the original literature and are subject to small inaccuracies.

TABLE 1. –*Citation summary.*

| Investigator (yr.) | Reference No. | Plotting symbol[b] | Mole ratios, F/U | Measurements[a]: Free F | Gel time | Formaldehyde emission | Bending strength | Modulus of rupture | Internal bond | Thickness swell | Comments[c] |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Berger (1964) | 1 | ▽ | 1.45,2.0 | + | – | – | + | – | – | + | Low |
| Brinkmann (1978) | 2 | ◠ | 1.27,1.5 | – | – | + | + | – | + | + | Medium. Two press temperatures. |
| Brunner (1980) | 3 | ▷ | 1.25,1.6 | – | – | + | – | – | + | + | High. |
| Dunky, et al. (1981) | 6 | ■◧◩[d] | 1.26,1.36,1.53 | + | + | + | + | – | + | + | High. Three degrees of condensation; three press times. |
| Hse (1974) | 9 | ⬭ | 1.5,1.7,1.9, 2.1, 2.3 | + | – | – | – | + | + | – | High. Three reaction temperatures; three reactant conc. |
| Marutzky, Ranta (1979) | 10 | ◒ | 1.2,1.25,1.27, 1.3,1.36,1.4 1.43,1.5,1.54, 1.55,1.6,1.8, 2.1 | + | + | – | – | – | – | – | Low. |
| Marutzky, Ranta (1980) | 11 | ● | 1.2,1.25,1.27 1.3,1.36,1.4, 1.6,1.8 | – | – | – | + | – | + | + | High. Two board densities. |
| Marutzky, et al. (1979) | 12 | ○ | 1.2,1.25,1.3, 1.36,1.4,1.6, 1.8,2.1 | – | – | + | – | – | – | – | High. Two board densities. |
| Mayer (1979) | 13 | ═══[e] | Curves 0.8-1.8 | + | + | + | – | – | + | + | Low. "Conventional" and "optimized" condensation procedures. |
| Petersen, et al. (1972) | 20 | ◇ | 1.4,1.6,1.8 | + | – | + | – | – | – | – | Medium. |
| Petersen, et al. (1973) | 21 | ▽ | 1.4,1.6,1.8 | – | – | + | – | – | – | + | Medium. Two press times. |
| Pshenitsyna, et al. (1979) | 22 | ◨ | 1.33,1.4,1.66, 1.8,2.0 | + | + | – | – | – | – | – | High. |
| Roffael (1976) | 23 | △ | 1.4,1.6,1.8 | – | + | + | – | – | + | + | Low. |
| Roffael (1978) | 24 | ◊ | 1.27,1.4,1.55, 1.6,1.8 | + | + | + | – | – | – | – | High. Several conditioning times. |
| Roffael (1980) | 25 | 0 | 1.27,1.4,1.6,1.8 | – | – | + | – | – | – | – | Low. |
| Roffael, et al. | 26 | ▲ | 1.2,1.4,1.6,1.8, 2.0 | – | – | + | – | – | – | – | Low. Two conditioning times. |
| Slonim et al. (1978) | 27 | × | 1.33,1.66,1.8, 2.0 | + | – | – | – | – | – | – | High. |
| Steiner (1973) | 28 | ⬒ | 1.4,1.6,1.8,2.0 | – | + | – | – | – | – | – | High. Cyclic aging of laminates. |
| Sundin (1979) | 29 | ⬡ | 1.25,1.32,1.45 | + | – | + | – | + | + | – | Low. Average results one plant. |
| Tinkelenberg, et al. (1982) | 30 | ——— | Curves, 0.8-1.6 $F/(NH_2)_2$ | – | – | + | – | – | – | – | Low. V100 test. $F/(NH_2)_2$ = effective ratio. |
| Williams (1982) | 32 | ◆ | 1.0,1.2,1.3,1.6 | – | – | + | – | + | + | – | High. Desiccator test. Conditioning time. |

[a]F = formaldehyde, MOR = modulus of rupture, IB = internal bond, + or – indicate whether the property was, or was not, measured.
[b]Figures 1-6.
[c]Low, Medium, High, = qualitative measure of amount of detail supplied about resin and board.
[d]Low, Medium, High degree of condensation.
[e]Conventional (---) and optimized (————) condensation procedures. Unspecified (——).

## UF resin properties

### Free formaldehyde content of resins

Resin free formaldehyde is frequently reported because a positive correlation generally exists between it and formaldehyde emission from boards. Several data sets are shown in Figure 1. A wide range of free formaldehyde values is observed at a given mole ratio, particularly at higher F/U. This divergence may be due to differences in resin synthesis procedures or to errors inherent in the sulfite titration method that is usually used for determining free formaldehyde (31). That synthesis differences may not be at fault is indicated by the reports that degree of resin polymerization does not affect free formaldehyde content between F/U 1.26 and 1.53 (6). and that free formaldehyde content is only moderately changed by reactant concentration or reaction temperature between F/U 1.5 and 2.3 (9).

Despite reservations about quantitative accuracy, the general parabolic pattern (Fig. 1) of dependence upon mole ratio is clear. Significant reductions in free formaldehyde can be made by decreasing F/U at relatively high mole ratio, but reductions may be only marginal below F/U 1.3 or 1.2.

### Resin reactivity

The parameter universally used as a measure of relative reactivity (and presumably, therefore, as a measure of relative press time) is resin gel time. The data from several sources (Fig. 2) illustrate the well-known reduction in reactivity of lower F/U resins. Here, the large differences among investigators result from the use of different temperatures and catalyst levels and perhaps from using different criteria (unspecified) for gelation. Although catalyst information was not always supplied, it seemed likely that $NH_4Cl$ or $(NH_4)_2SO_4$ was usually used; a good part of the explanation for longer gel times at low F/U, therefore, resides in the decreased availability of free formaldehyde for reaction with $NH_4Cl$ to liberate HCl for cure catalysis.

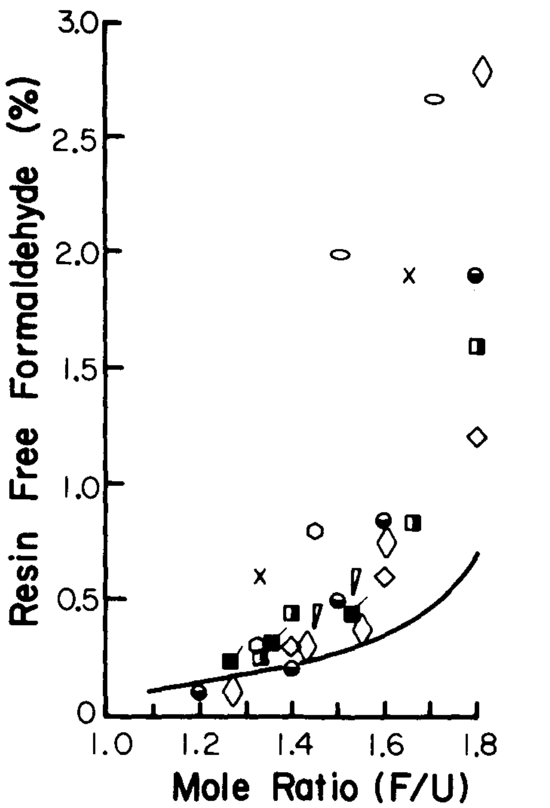


Figure 1. – Effect of mole ratio on resin free formaldehyde content. (See Table 1 for symbol/curve identification).

## Board properties

### Formaldehyde emission

Numerous studies have reported on the effect of mole ratio on formaldehyde emission. A variety of tests were used but the emphasis was upon the perforator (8) and variations of the WKI (25) tests, with very limited data from the desiccator test (18). Figure 3 is a composite of data from the few studies that extended down to mole ratios of 1.3 and below; some of the data sets ( — , 7 ) were multiplied by an arbitrary factor to place them on the same scale. The dashed line is reproduced from Mayer's survey paper (13), and the solid line interestingly, is adapted from a plot of board emission versus "effective mole ratio," $F/(NH_2)_2$ for UF and MUF systems with and without added urea or $NH_3$(30).

At first glance it may appear from Figure 3 that the emission/mole ratio curve is approximately parabolic and that the rate ofreduction decreases strongly as F/U approaches unity. While a parabolic pattern appears logical, particularly in view of the resin free formaldehyde behavior (Fig. 1), closer examination makes it less certain. Marutzky, et al. (12), for example, em-

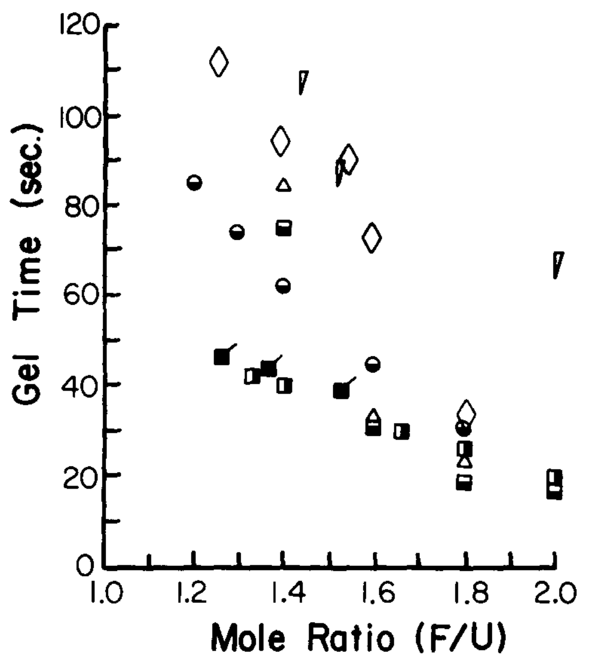


Figure 2. –Effect of mole ratio on resin gel time. (See Table 1 for symbol identification. ◇ = t/3; **T** = t/10; ◪ = t/20).

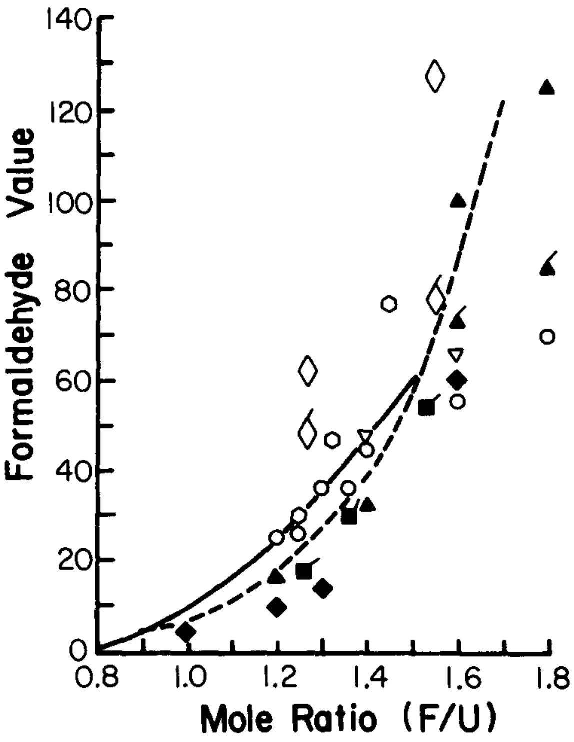


Figure 3. – Effect of mole ratio on particleboard formaldehyde emission (– –) (13). open circles (12); ■ (6); ∇ (21) = perforator value after 4 weeks at 20°C/65% RH; ○ = average perforator for boards from one Swedish plant 1972-1976 (29); ▲ , ▲ = perforator immediately after production and after 8 days (26); ◇ , ◇ = 24-hour WKI test value after 1 day and after 6 weeks at 20°C/65% RH (24); — 21 6-hour WKI test value ÷ 50 versus F/$(NH_2)_2$ for UF and MUF resins with and without added urea or $NH_3$ (30); ◆ = 10X desiccator values after 7 days (32). Perforator and WKI in mg/100 g dry board. Desiccator in µg/mL.

phasize that board conditioning or aging reduces emission more strongly at high F/U than at low, and their data for conditioned boards (open circles, Fig. 3) indicate a linear behavior between F/U 1.8 and 1.2. The aging effect is illustrated further by additional data points in Figure 3 ( ◇ vs. ◇ and ▲ vs ▲) and by the summary in Table 2. In absolute terms, emission is clearly reduced more at high mole ratio, and this is likely true in relative terms as well. Note, moreover, that no conditioning information was supplied for the dashed line (13) in Figure 3.

Other variables may interact with mole ratio to affect emission. Increasing press time or temperature, for example, results in lower board emission, and the available data indicate that the decrease is greater absolutely at higher mole ratios and sometimes greater relatively (Table 3). However, Dunky, et al. report that perforator values do not change with degree of resin conversion at constant mole ratio (6). Resin free formaldehyde content is also affected differently at various mole ratios by reactant concentration and reaction temperature; whether this translates into board emission differences was not examined (9).

In view of the resultant uncertainties as to the exact shape of the emission/mole ratio curve, what can we conclude about the potential for meeting proposed board emission standards by simply reducing the UF resin mole ratio? Consider first the German E-1 standard requiring a perforator value of ⩽ 10 mg/100 g for particleboard used in dwellings (7). The only data in Figure 3 that represent perforator values for boards whose conditioning was specified are those symbolized by ▲, ▲ (26); by ■ (6); and by open circles (12). Thus, without employing unconventional press conditions, additives, board treatments, etc., it appears that F/U must be at least below 1.2 and more likely below 1.1 to achieve the 10 mg/100 g E-1 standard.

TABLE 2. – *Effect of aging on particleboard emission.*

| Reference No. | Test | Mole ratio F/U | Aging condition | Aging time | Test value | Test value ratio |
|---|---|---|---|---|---|---|
| 24 | WKI (25) (mg/100 g) | 1.27 | 20°C/65% | 1 day | 63 | |
| | | | | 6 weeks | 49 | 0.78 |
| | | 1.55 | | 1 day | 127 | |
| | | | | 6 weeks | 78 | .61 |
| | | 1.40 | | 7 weeks | 60 | |
| | | | | 15 months | 48 | .80 |
| | | 1.60 | | 7 weeks | 139 | |
| | | | | 15 months | 60 | .43 |
| | | 1.80 | | 7 weeks | 175 | |
| | | | | 15 months | 72 | .41 |
| 26 | Perforator (8) (mg/100 g) | 1.6 | Probably 20°C/65% | 0 | 100 | |
| | | | | 8 days | 73 | .73 |
| | | 1.8 | | 0 | 125 | |
| | | | | 8 days | 85 | .68 |
| 32 | 2-hour desiccator (18) (µg/mL) | 1.0 | Probably ambient | 1 day | 0.8 | |
| | | | | 15 days | 0.4 | .50 |
| | | 1.2 | | 1 day | 1.4 | |
| | | | | 15 days | 0.8 | .57 |
| | | 1.3 | | 1 day | 3.0 | |
| | | | | 15 days | 1.7 | .57 |
| | | 1.6 | | 1 day | 8.0 | |
| | | | | 15 days | 4.3 | .54 |

TABLE 3. – *Effect of press temperature and time on parlicleboard emission.*

| Reference NO | Mole ratio F/U | Varying press temperature: Press temperature/ time | Perforator value | Perforator ratio | Varying press time: Press temperature1 time | Perforator value | Perforator ratio |
|---|---|---|---|---|---|---|---|
| | | (°C/min.) | (mg/100 g) | | (°C/min.) | (mg/100 g) | |
| 21 | 1.40 | 140/8 | 48 | | 180/5 | 41 | |
| | | 180/8 | 37 | 0.77 | 180/8 | 40 | 0.98 |
| | 1.60 | 140/8 | 85 | | 180/5 | 80 | |
| | | 180/8 | 65 | 0.76 | 180/8 | 70 | 0.88 |
| | 1.80 | 140/8 | 157 | | 180/5 | 150 | |
| | | 180/8 | 110 | 0.70 | 180/8 | 110 | 0.73 |
| 2 | 1.27 | 180/3.2 | 27 | | 220/2.1 | 26 | |
| | | 220/3.2 | 18 | 0.67 | 220/3.2 | 18 | 0.69 |
| | 1.50 | 180/3.2 | 59 | | 220/2.1 | 57 | |
| | | 220/3.2 | 40 | 0.68 | 220/3.2 | 40 | 0.70 |
| 6 | 1.25 | | | | 170/2.5 | 28 | |
| | | | | | 170/4.2 | 17 | 0.61 |
| | 1.37 | | | | 170/2.5 | 47 | |
| | | | | | 170/4.2 | 30 | 0.64 |
| | 1.53 | | | | 170/2.5 | 84 | |
| | | | | | 170/4.2 | 55 | 0.65 |

In contrast, the proposed National Particleboard Association (NPA) standard for particleboard in mobile homes is for a 2-hour desiccator value of 1.8 µg/mL. Here the only published data as a function of F/U are given in Figure 3 by ♦ (actual values × 10 (32)). Within the same restrictions as above, these data indicate resins should have a mole ratio below about 1.3, but the limited data preclude certainty.

Physical and mechanical properties

Researchers generally perceive (11, 13, 29) that, of the more critical properties in this group, reduced mole ratio primarily affects internal bond (IB) and thickness swelling of particleboard. This belief is fairly well supported by the published data. The limited data for bending strength (1, 2, 6, 11), for example, do not extend below F/U = 1.2, but they do not indicate any significant changes between 1.2 and 2.0. Among the few reports containing data on mole ratio and modulus of rupture (9, 29, 32). only one (32) extends below F/U = 1.2 and indicates a loss of modulus with decreasing mole ratio; the significance of that loss is questionable, however, since board density also fell in parallel.

Data for internal bond (IB) and its dependence upon mole ratio are more extensive (Fig. 4). While Marutzky and Ranta (11) indicate little or no loss in IB from F/U = 1.8 to 1.2 (●, plus a parallel series of higher-density boards), most other investigators report significant losses beginning as high as F/U = 1.4 or 1.5. The only findings below F/U = 1.2 are the data ( ♦ ) of Williams (32) and the curves (---, – – ) given by Mayer (13). The apparent loss in IB shown by Williams should probably be moderated because of accompanying board density decreases. The curves from Mayer (13) reportedly demonstrate improvements possible by substituting resins prepared using unspecified "optimized condensation procedures" (---) for those prepared from "conventional condensation procedures" ( – – ), Resin synthesis variations are more clearly defined in other investigations and indicate that degree of condensation does not influence IB between F/U = 1.53 and 1.26 (■, 6), but that increased reactant concentration at F/U = 1.5 may have a small positive influence upon IB (⬭, 9). Finally, the reported effects of press time and temperature upon IB are inconsistent in terms of any interaction with mole ratio (2, 6).

In sum, if we compare the German IB standard of 0.35-0.40 MPa (4) with the European data, a conservative approach would require that "conventional" UF resins should be kept above F/U = 1.1 or possibly above 1.2. On the other hand, it appears (13) that "improved

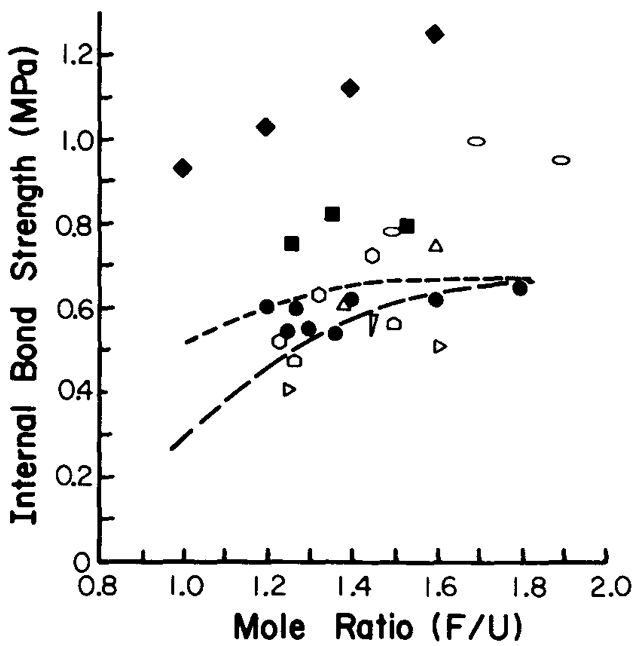


Figure 4. – Effect of mole ratio on particleboard internal bond strength. German Standard (DIN) = 0.35 to 0.40 MPa (4). NPA Standard = 0.55 MPa (19). (See Table 1 for symbol/curve identification.)

UF resins have been developed that will maintain acceptable IB's down to F/U = 1.0.

The available data (Fig. 5) regarding effects of mole ratio on thickness swell are all European, and surprisingly, many of the values do not meet the German standard (4).[2] There is a rather clean consensus, however, that as F/U falls below 1.3, thickness swell begins to rise (deteriorate) at a significant rate. Again, Mayer reports (13) improved behavior for resins from "optimized condensation procedures" (---vs. –). Moreover, Dunky, et al. find that a lower degee of resin condensation provides better thickness swell resistance (% vs. % ),and this is attributed to greater penetration of resin into the particles at the lower degree of condensation (6). Within the relevant studies, no consistent effect of press time or temperature can be observed (2, 6).

Because many of the swelling values exceed the German standard even at high mole ratios, it is difficult to arrive at a limiting F/U. Conservatively, F/U = 1.3 to 1.2 should perhaps be accepted, particularly for "conventional'' resins.

Although durability is not a major consideration for interior applications, several studies have reported results from accelerated aging tests indicating that low mole ratio UF systems possess poorer aging resistance. Such investigations have included exposure of particleboard to humid-dry cycles (11) or to the German V100 test (2-hr. swell in water at 100°C (30)) and exposure of cross-lapped laminates to VPS/dry cycling (28). This greater sensitivity of lower mole ratio systems has been attributed by some to a greater brittleness of UF resins with low mole ratio (5, 11).

**Summary and conclusions**

Published literature on the effects of formaldehyde to urea mole ratio (F/U) of UF resin is rather extensive and covers a wide range of resin, board, and testing variables. This range, plus the frequent lack of details, make it difficult to quantify the dependence of important properties upon mole ratio or to quantify what the lowest mole ratio should be to maintain acceptable levels of a given property. Nevertheless, the following qualitative conclusions appear warranted about the effect of reducing mole ratio on resin and particleboard properties, in the absence of changes in other resin or board variables.

1) Resin free formaldehyde content falls rapidly between F/U 2 and 1.5; reductions may be marginal below F/U 1.3 or 1.2.

2) Resin gel time, using ammonium salt catalyst, increases two-fold or more as F/U decreases from 2.0 to 1.2.

3) Particleboard formaldehyde emission decreases manyfold from F/U 2.0 to 1.0, although the rate of decrease is less for aged boards. To meet the German E-1 standard it appears that mole ratio must be below 1.2 or even below 1.1, in the absence of other emission-reducing steps. Meeting the proposed NPA standard would probably require a mole ratio below 1.3; limited data make this a tentative judgment.

[2]DIN 68-763 limits 24-hour thickness swell to a maximum of 15% with 25 by 25 mm pieces (4). NPA-1-73 imposes a maximum of 8% on 305 by 305 mm pieces (19).

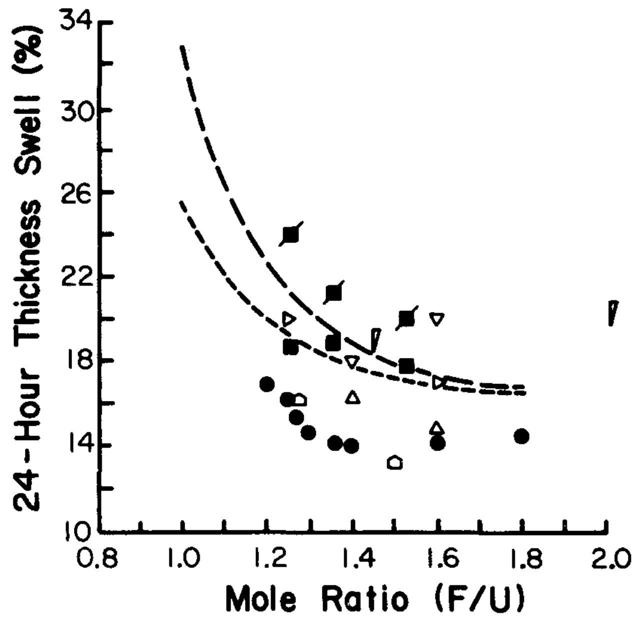


Figure 5. - Effect of mole ratio on particleboard 24-hour thickness swell. German Standard (DIN) = 15 percent (4). (See Table 1 for symbol/curve identification.)

4) Bending strength and modulus of rupture apparently suffer little change down to F/U = 1.2; very little information is available below that point.

5) Internal bond is probably maintained at acceptable levels down to F/U = 1.2, although losses are reported beginning at 1.4 or 1.5. For conventional resins the mole ratio should conservatively not fall below 1.2, possibly 1.1.

6) Thickness swell (24 hr.) begins to rise rapidly as F/U falls to 1.3 or even before. A conservative limit here should probably be set at F/U = 1.3 to 1.2 for conventional systems.

Overall there are obviously potential conflicts between emission (German EI: F/U < 1.1 to 1.2, or proposed NPA: F/U < 1.3) versus IB (F/U > 1.1 to 1.2) and thickness swell (F/U > 1.2 to 1.3). From a conservative viewpoint, one must question whether mole ratio can be the sole variable used to produce particleboard having both acceptably low formaldehyde emission and adequate physical properties.

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