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Japanese desiccator method JIS A 5905

A study into the factors causing inter-laboratory differences

SUMMARY

Inter-laboratory trials were used to quantify between-laboratory differences for the Japanese desiccator formaldehyde emission method. The differences in equipment and methodology were determined by visiting a number of laboratories. The visits were followed by laboratory trials to ascertain the significant factors.

With this knowledge, further inter and intra-laboratory trials were conducted to determine if differences could be reduced. Recommendations are made for future changes to the standard.

Introduction

There are three Japanese Standards documenting formaldehyde emission testing by the Japanese desiccator method. Japanese Industrial Standards (JIS) A 5905 and A 5908 cover testing of fibreboard and particleboard. The Japanese Agricultural Standard 1515 covers plywood testing. This report is based on data from medium density fibreboard (MDF), 2.5mm to 18mm thicknesses, of between 700 and 800 kg/m^3 made using urea formaldehyde (UF) resins.

The objective of this work was to improve the repeatability and reproducibility of the Japanese desiccator method and to make recommendations to the JANS (Japan, Australia, New Zealand Standards) Committee. Other researchers have carried out fundamental work examining the factors affecting formaldehyde emission (Roffael, 1993). However the objective of this work was not to carry out fundamental studies but to ascertain the significance of specific differences in equipment and methodology using a specific product, namely MDF made with current UF resins.

The three Japanese desiccator standards are similar, employing a glass desiccator as a test chamber. Test-pieces are cut into rectangles150mm by 50mm. A number of test-pieces, corresponding to approximately 1800cm^2 total surface area, are supported above a crystallising dish containing water inside the desiccator. The lid is placed on the desiccator and the samples are sealed inside for 24 hours at 20±1°C. The emitted formaldehyde is absorbed by the water in the crystallising dish in proportion to the emission rate from the sample. The concentration of formaldehyde in the water is determined by reaction with acetylacetone, followed by spectrophotometric determination of the developed colour.

Over the past two years, Pacific Rim manufacturers of Panelboards have been producing increasingly lower formaldehyde emitting products. Product made in, or exported to, Asia, New Zealand and Australia is often tested by the Japanese Desiccator method to determine its emission category. The emission ratings for the test are, according to JIS A 5905: E0 for <0.5mg/l, E1 for <1.5mg/l and E2 for <5mg/l. As with the perforator method EN120, this rating determines the end use of the product and in many cases the buyer specifies the emission rating in the purchase contract.

To produce low emission values whilst still maintaining mechanical test qualities the manufacturers must use different and in some cases more expensive technology. There is considerable competition to produce low emission product and some scrutiny both by other manufacturers and by the purchaser to ensure that product truly conforms to specification. As the demand for low emission product has increased it has become apparent that different laboratories have been rating product differently. Different laboratories can rate a single product E0, E1 or E2. This has led to problems with sale of product, loss of potential markets and in some instances has led to lack of confidence by the manufacturers in the test method and in the laboratories performing the test.

To investigate and rectify this problem, TimberTest conducted a

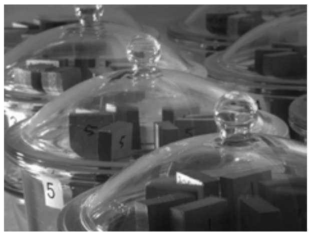

Figure 1: The Japanese desiccator test equipment.

five-part study:

1. Inter-laboratory trials were conducted to ascertain the actual differences between laboratories.
2. Laboratories around the Pacific Rim were contacted and in many cases visited to ascertain the differences in equipment and methodology used in each laboratory.
3. Differences in methodology and equipment were simulated in laboratory trials. This information in combination with literature searches was used to determine the most significant factors.
4. Within and between laboratory trials were then conducted specifying the test procedure exactly based on these factors.
5. Recommendations were made to the JANS standards committee.

Inter-laboratory trials

Four inter-laboratory trials were conducted, the results of one of these is presented here. The laboratories included commercial testing laboratories, Government laboratories and factory laboratories from Australia, New Zealand and Asia including Japan. The trials used samples of 4.75mm and 2.5mm MDF, UF resin. Samples for five formaldehyde tests were randomised and sent to the participating laboratories. One set of samples was flown to Singapore and back to New Zealand to test the effect of flying samples to different locations. The results indicated little or no effect from flying samples to Singapore but showed a wide range of values from the different laboratories. The difference between the lowest and the highest value was 0.9 mg/l

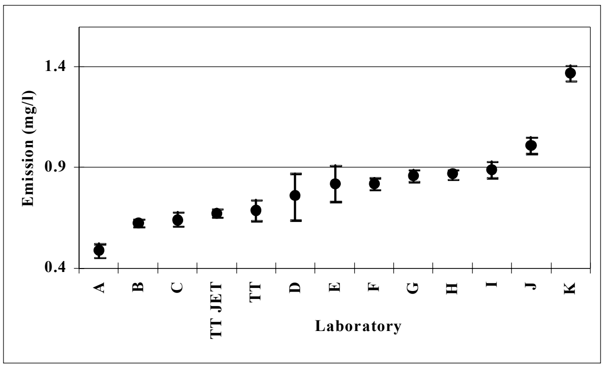

Figure 2: Inter-laboratory trial. Limit bars are the 95% confidence limits for the mean of five samples.

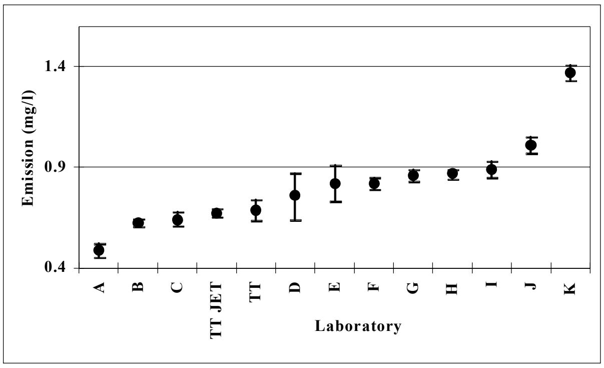

Figure 2: Inter-laboratory trial. Limit bars are the 95% confidence limits for the mean of five samples.

Laboratory visits

Between June 1998 and April 1999 12 companies in the Pacific region were contacted or visited. The objective was to determine the differences in the interpretation of the standard, which could be leading to the wide range of results, found in the inter-laboratory studies.

A number of differences were found in sample handling, test equipment and methodology. These differences are summarised below:

Pre-conditioning
Samples were cut and then pre-conditioned for between zero and 10 days. The pre-conditioning environment varied with some being controlled at 65%RH and 20°C while others used uncontrolled laboratories for pre-conditioning. In the later cases the room temperatures ranged from 18 to 30°C (temperate to tropical situations).

Laboratories pre-conditioned samples either in plastic bags, stacked together without separation or in racks which ensured separation. The racked samples were exposed to either still or moving air (fans).

Background Formaldehyde in the Pre-Conditioning Environment
Although it could not be quantified it was suspected that the background formaldehyde in the pre-conditioning environment differed between laboratories. Samples were conditioned in a range of enclosed cabinets or rooms, some of which had clean air and some airflow from the manufacturing facilities. Some laboratories conditioned the samples inside plastic bags.

Number of Test-pieces
When asked to test one product laboratories made different decisions on how many test pieces to test. Some chose less than $1800cm^2$, others chose more than $1800cm^2$.

Supporting Plates
Plates of various designs were used to support the sample above the crystallising dish. The most common plate types were those supplied by desiccator manufacturers.

Supporting Clamps and Racks
Clamps and racks were also used to keep thin samples upright in the desiccator. These systems included stainless steel wire baskets and carousel arrangements from which the samples where hung using alligator clips. Some laboratories used staples to attach the samples together which held the samples upright.

Desiccators
A range of desiccator designs were in use. These included Japanese manufactured desiccator's with convex bottoms, German designs with flat bottoms and a range of different enclosed volumes. The distance between the sample and the top of the crystallising dish varied with each design.

Crystallising Dish
The formaldehyde is collected into water in a crystallising dish. These dishes ranged from 110mm to 120mm inside diameter.

Temperature of Test
All the laboratories attempted to maintain test temperature between 19°C and 21°C as specified in the standard. Some laboratories did not log the temperature throughout the test and were therefore not able to determine if the temperature remained within the limits. Where the test temperature was known to have been high or low throughout the test some laboratories applied correction factors to the data using published temperature - emission relationships.

Background Formaldehyde in the Test Process

Some laboratories did not monitor the background formaldehyde in the desiccators or in the water used for the test.

Orientation of Test-pieces
In most cases test-pieces were tested "upright". However some laboratories tested samples on their edges.

Laboratory trials
The effect of each of the equipment methodology differences was determined by simulating the differences in controlled laboratory trials.

Materials
All trials used UF-MDF panels of 3mm to 18mm thickness. All sample material was more than three months old. In all cases test-pieces were cut and randomised to reduce the effect of variation in the material. Unless otherwise specified all treatments were replicated and the mean of the two values has been reported.

Days After Cutting
An uncut MDF panel, 3mm thick, was pre-conditioned to constant weight at 65%RH and 20?C. Then after pre-conditioning, on day "zero", all samples were cut into test-pieces. The test-pieces were then further conditioned for between zero and 10 days at 65%RH and 20?C, in a well ventilated controlled environment room. The results indicated that the number of days conditioning from the time of sample cutting is important. Since the samples were more than three months old and the samples had been conditioned to constant weight prior to cutting, the emission reduction with time is assumed to be due to freshly cut samples producing high initial emissions.

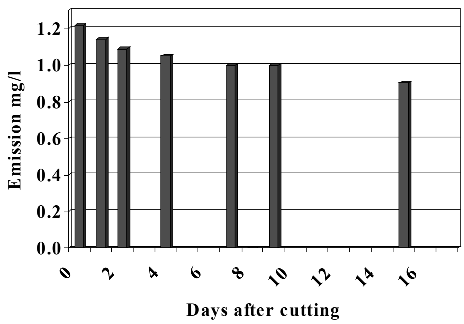

Figure 3: Days after cutting.
Each point represents a single test value.

Pre-conditioning Temperature – Part 1

The effect of the temperature of the pre-conditioning environment was examined by conditioning two thicknesses of cut test-pieces at 10?C, 20?C and 28?C (uncontrolled RH) for seven days. After removal from the pre-conditioning environment the emissions were measured at 20?C.

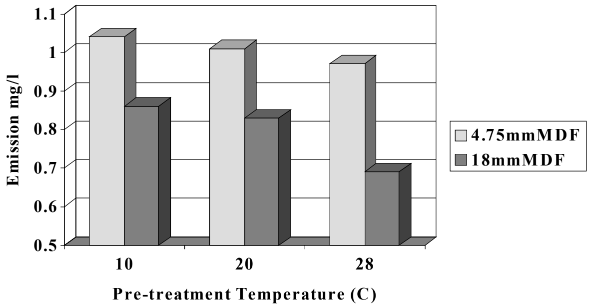

Figure 4: Pre-conditioning temperature.

Pre-conditioning Temperature Part 2

To investigate whether this effect can be negated by subsequent conditioning for seven days at 65%/20?C the same samples were re-conditioned at 65%RH/20?C. The re-tested samples gave similar results despite their pre-history.

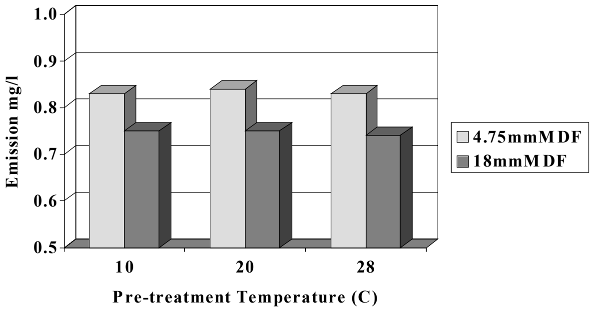

Figure 5: Reconditioning samples at 65% 20?C to remove pre-history.

Background Formaldehyde in the Pre-conditioning Environment
The effect of differences in background formaldehyde in the conditioning environment was investigated by pre-conditioning 18mm MDF test-pieces in a range of conditions. The cut test pieces were placed in the following environments prior to testing.

Box+ enclosed in an 11 litre box with other high emission samples
Box enclosed in an 11 litre box with no other samples
Lab good ventilation, with 20mm space between samples
Lab-fan as for "Lab" except samples were exposed to moving air

The samples in enclosed boxes gave higher results than the "Lab" samples". The moving air had a minor, if any, effect.

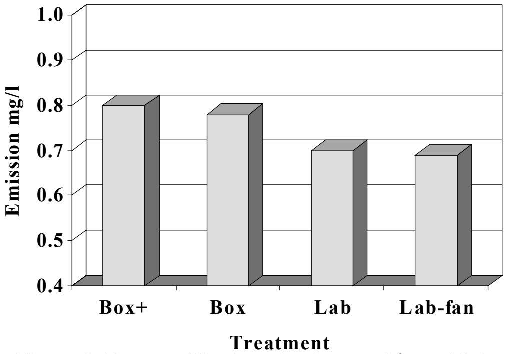

Figure 6: Pre-conditioning - background formaldehyde.

Pre-conditioning in Plastic Bags

A 3mm thick MDF panel was cut into four sets of test-pieces. Two sets of test-pieces were placed in plastic bags for seven days at 20?C. The second two sets of samples were treated using the normal TimberTest technique; on racks to ensure separation, at 65%RH and 20?C.

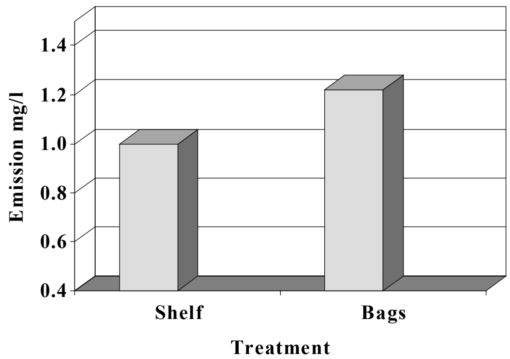

Figure 7: Pre-conditioning - effect of enclosing samples in bags.

Supporting Plates
Three common plate designs were compared with a wire grid and with "no plate". Plate A had three rings of 10mm holes, a total hole area of 10%. Plate B had one large central hole and 6 outer 35mm holes; total hole area was 20%. Plate C had many 3mm holes, a total hole area of 37%. The wire grid was made from stainless steel and had a 20mm grid. The wire grid gave the same values as no plate; the other plates all produced lower results than "no plate".

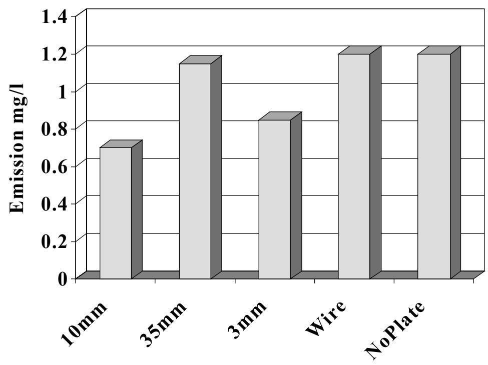

Figure 8: Effect of supporting plates

Desiccators Size and Design

Four desiccator designs were compared. The enclosed volumes were 9,11,13 and 15 litres. The distance from the sample to the top of the crystallising dish varied with each design; this distance is given in brackets in the results. This trial did not indicate a relationship between emission and desiccator volume. However the desiccators in which the samples were supported closer to the crystallising dish did give higher results. It is possible that the distance from the sample to the dish influences the result but more work would be needed to ascertain the influence of different desiccator geometries on the results.

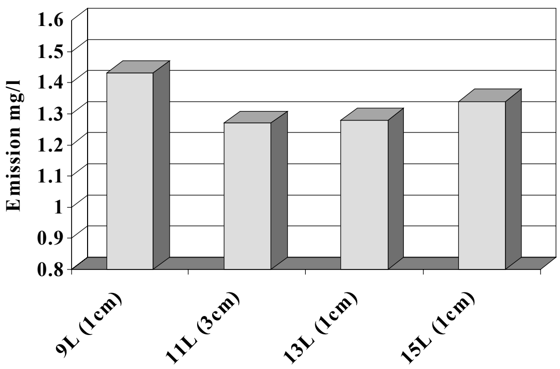

Figure 9: Desiccator size and design.

Crystallising Dish

Dishes of 120mm and 110mm inside diameter were compared; these were the maximum and minimum sizes used in the laboratories surveyed. Two trials were conducted, each trial using each of the two sizes of dish. Each bar on the graph represents the mean of five samples. The larger dish gave approximately 25% higher values for both trials.

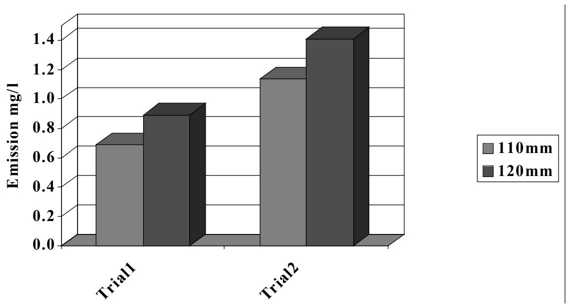

Figure 10: Crystallising dish diameter.

Temperature of Test

The emissions at one degree intervals from 18?C to 22?C were measured using 18mm MDF. The relationship between emission and temperature indicated an approximately 8% change in emission for every degree change in temperature. There was a 16% increase from 19 to 21?C, which is the temperature range allowed by the current standard.

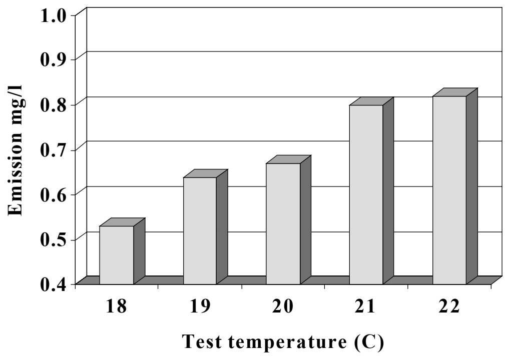

Figure 11: Test temperature.

Number of Test Pieces

To test the effect of different numbers of the 150mm by 50mm test-pieces, tests were carried out using from 6 to 10 test-pieces of 18mm MDF. Eight samples had the closest sample surface area to 1800cm2. There was an increase in emission from the lowest to the highest surface areas, however there was only a small difference between 6, 7, 8 and 9 test-pieces.

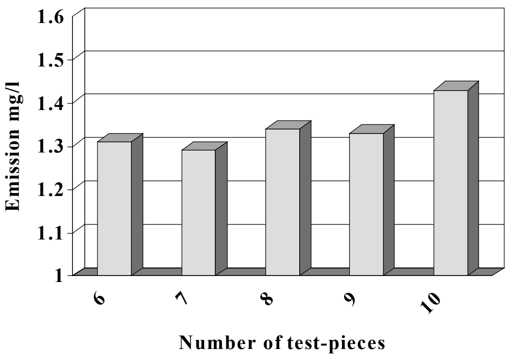

Figure 12: Number of test-pieces.

Inter-laboratory and intra-labortory Trials - similar equipment

To test the difference within and between laboratories using similar equipment, two sets of the same sample were measured in separate "runs", by two different laboratories. Each laboratory tested five samples in each run. In the following graph (Fig 13) the results TT A and B are from the TimberTest laboratory, the results LabX A and B are from the other participating laboratory. The difference between the means within each of the laboratories was 0.01 and 0.11 mg/l. The maximum difference between the means of the two laboratories was 0.17mg/l.

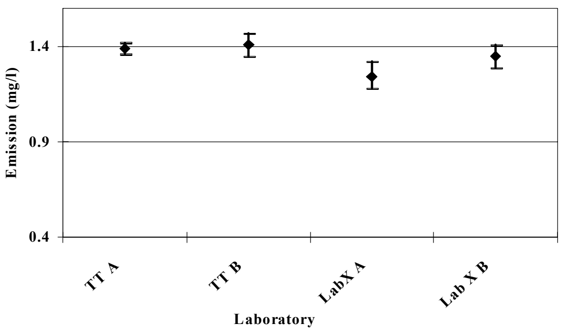

Figure 13: Inter and intra-laboratory trial (same equipment). Limit bars indicate 95% confidence limits for the mean of five samples.

DISCUSSION

Within and between laboratory differences in formaldehyde emission results observed for the Japanese desiccator method has caused, or has the potential to cause, a number of problems for the panel-board industry:

1. Manufacturers are not having their products rated consistently by different laboratories and are therefore not on an equal basis when they sell their product either on the domestic or export markets.

2. Since different technology is required to produce lower emissions there are manufacturing cost difference between producers depending on who is conducting the conformance testing of their product.

3. Bad will (and the possibility of litigation) may occur when the product being sold does not, in the opinion of the purchaser, meet the speciifed level of emission. This is despite the manufacturer making the product in good faith, to perform according to the local interpretation of the test standard.

4. In some cases the manufacturers and purchasers are losing faith in testing laboratory results. There is also potential for favouring one test laboratory over another depending on who provides the most desirable values.

5. Unintentional trade barriers may result, manufacturers being unsure of what is required to sell to a certain market.

Large differences are highlighted by the inter-laboratory trials. The range from lowest to highest value for the inter-laboratory trial presented at the beginning of this report (Fig 1) was 0.9mg/l, more than 200%. These differences are also typical of more recent trials.

The worst case difference between the two laboratories using similar equipment was 0.17mg/l (13%). Although this range is larger than is desirable it is small compared with the range of results from the non-standardised laboratories. The differences are assumed to be due to outstanding inconsistency in the methodology since the test equipment was the same in both laboratories. Some possible causes for the difference have since been identified and these include different background levels of formaldehyde at the two sites. Trials are planned to investigate this further.

RECOMMENDATIONS

At present the Japanese standards are being combined with Australian and New Zealand Standards to produce Japanese, Australian and New Zealand Standards (JANS). During this process the Japanese desiccator method has been under review and it is therefore an opportune time to re-consider the content and requirements of the standard. It is recommended that the following changes be made to the standard:

FactorRecommendationPre-conditioning time7 to 10 days – after cuttingPre-conditioning RH/?C65%RH/20?CPre-conditioning roomWell ventilated room, fresh air supplyPre-conditioning setupRacks to allow air flow around samplesGrids/plates in desiccatorWire grid only allowedDesiccatorsClosely define – or more work neededCrystallising dishSpecify inside dimensionsTemperature of test20?0.2?C, continuous temperature log mandatoryBackground formaldehyde (blanks)Measure, report and subtract from final valueNumber of test-piecesThe number closest to 1800cm2 area

In addition to the above recommendations, quality management techniques from the chemicals and pulp and paper industries are suggested. These techniques include:

1. The use of quality control samples.

2. The use of blanks to separately monitor background levels of formaldehyde inside the desiccator, in the water and in the chemicals.

3. Regular participation in inter-laboratory trials.

Factor	Recommendation
Pre-conditioning time	7 to 10 days – *after cutting*
Pre-conditioning RH/°C	65%RH/20°C
Pre-conditioning room	Well ventilated room, fresh air supply
Pre-conditioning setup	Racks to allow air flow around samples
Grids/plates in desiccator	Wire grid only allowed
Desiccators	Closely define – or more work needed
Crystallising dish	Specify inside dimensions
Temperature of test	20±0.2°C, continuous temperature log mandatory
Background formaldehyde (blanks)	Measure, report and subtract from final value
Number of test-pieces	The number closest to 1800cm^2 area

Table 1: Recommendations for JANS16 standard.

References

EDMONE ROFFAEL (1993). Formaldehyde Release from Particleboard and Other Wood Based Panels:Malayan Forest Records No. 37

Acknowledgements

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