A comparison of artificial ageing with 27 years of natural ageing

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Abstract

In 1980, a project involving the testing of physical and chemical properties of six papers before and after artificial ageing was conducted. It was decided at the time that a very useful extension of this project would be to compare artificial ageing results with those obtained after a period of natural ageing in order to provide some useful data on previous estimates of equivalencies between artificial and natural ageing. Comparative testing for folding endurance and pH of paper samples naturally aged for 27 years has now been carried out and compared with the testing results of the same paper samples from 1980.

Introduction

The origins of this project go back to 1980 when Ian Batterham, Bill Hamilton and Maurice Weightman carried out a paper quality testing programme as their major research project for the Canberra College of Advanced Education (CCAE) Materials Conservation course (Batterham et al. 1980). The project was sponsored by the then Australian Archives (now the National Archives of Australia, NAA) who provided the paper samples and the testing facility. Ian Cook, then of the National Library of Australia (NLA) was also on hand assist with the scientific elements of the project. The project resulted in some useful data and results of the testing were eventually published in *Archives and Manuscripts* in December 1982 (Batterham et al. 1982).

Towards the end of the project the idea presented itself to store samples of the test papers to enable retesting in a period of years (we aimed for 25) and retest the paper to see how our artificial ageing procedure compared with natural ageing.

The Technical Association of the Pulp and Paper Industry (TAPPI) Standard Test Method *T453 Effect of dry heat on properties of paper and board* suggests that for folding endurance results there is an equivalence of 72 hours of dry ageing at 105˚C to 25 years of natural ageing. This is a figure that was established through the work of a variety of researchers, from Rasch and Stone (1932), to Wilson and Parks (1980).

One intention of this project was to see how our results would compare with previous studies. It is a rare thing to be able to carry out such comparative tests so data is scant and new data to assist with making predictions of longevity is welcomed.

Our testing also had some unique features: the inclusion of papers classed as archival along with ‘standard’ grade papers and the placement of samples in both air-conditioned (A/C) and non air-conditioned (non-A/C) environments, although as will be discussed, control of this was far from perfect.

In 1980 samples of the test papers were packed into standard archives boxes, which at that time were of poor quality brown board, and sent to the (now) NAA offices in each of the state capitals. Samples were also lodged at the NLA. Two boxes were sent to each office, one designated for A/C storage and one for non-A/C storage.

In 2007, 27 years later the decision to recall these samples and carry out the follow-up project was made. Luckily Ian Batterham was still with the NAA in Canberra and the organisation still maintained a paper testing facility. There was the added bonus of having a chemist on staff, who assisted with the testing and analysis of results. Accordingly, a letter was sent out to each NAA state office asking for the samples to be returned to Canberra.

Natural ageing conditions

When samples were sent to the state offices in 1980, the request was made that one box be placed in A/C storage and one be placed in non-A/C storage. We believe that in general this was done but the process was far from controlled so we don’t actually know what conditions prevailed for either the A/C samples or the non-A/C samples. We do know that boxes in A/C storage were stored amongst actual archives on standard shelving. The air conditioning would therefore have generally been on 24 hours per day and been aimed at maintaining the standard conditions of 50% RH and 23°C. We are less certain as to the conditions to which the non-A/C samples were subjected, but believe they would have reflected the external conditions prevailing in the particular locations. All boxes were moved around over time as offices relocated or downsized so conditions may also have changed at these times. This uncertainty has somewhat limited the conclusions we can draw from the data.

Retrieval of samples

Unfortunately over time some of the boxes had been lost. However, boxes were received back in time for testing from Melbourne, Hobart, Perth and Brisbane, which gave us a good range of environments: subtropical, warm temperate and cool temperate.

An initial look at the boxes themselves was interesting. All boxes that had been stored in non-A/C conditions had discoloured more than those in A/C. Also, a red felt tip pen used in labelling had faded entirely on the non-A/C boxes but still remained visible on the A/C boxes (Figure 1). Opening the boxes revealed nothing obviously out of the ordinary (Figure 2).

Figure 1: Comparison of box stored 27 years in non-A/C environment (left) and box stored in A/C environment (right)

Figure 2: Open box showing packing of the samples

The paper samples

The papers selected in 1980 for testing were a combination of papers deemed 'archival' quality and those not intended for the archival market classed as 'standard'. They were as follows:

i. Permalife® Bond (73 gsm) - Archival

A paper produced by the Howard mill in the USA for the archival market.

ii. Archive Text (85 gsm) - Archival

The manufacturer and supplier details of this paper are not known. Archive Text was a commonly used archival paper at the time and is still produced, currently sold from the UK by Conservation by Design.

iii. Dry Process Copying (80 gsm) – Standard

A typical paper produced at the time for electrostatic copying. Produced by Associated Pulp and Paper Mills (APPM) in Australia.

iv. Plus Fabric Bond (70 gsm) – Standard[1]

A paper produced by APPM using cotton fibre, for the 'fine paper' market.

v. Burnie Bank (44 gsm) – Standard

A light weight 'manifold' paper in use at the time for the production of carbon copies. Produced by APPM.

vi. Canary Bank (44 gsm) – Standard

Identical to Burnie bank except the paper is dyed yellow. Also produced by APPM. Included to see whether the dye would have any effect on longevity.

Testing carried out in 1980

A range of testing had been carried out in 1980 (Batterham et al. 1980) to determine the quality and makeup of the papers, as follows:

- Basis weight
- Thickness
- Spot tests for lignin, alum, rosin and starch
- Folding endurance
- Brightness
- Optical whiteners
- pH levels

Papers had also been artificially aged at 105°C as per TAPPI Standard Test Method *T453 su-70 Effect of dry heat on properties of paper and board*. Samples were aged for periods of 1, 3, 6, 12, 18, 24, 30 and 36 days.

Experimental

For the current testing of the naturally aged samples, folding endurance testing and pH analysis were repeated. Fourier transform infrared (FTIR) spectroscopy was also carried out as the NAA now owns an FTIR instrument.

Folding Endurance Test

Folding endurance was the primary strength test utilised in 1980 and so was repeated for the follow-up testing. The machine used for testing was the very one used in 1980, a Torsee M.I.T. type folding endurance tester manufactured by the Tokyo Testing Machine Manufacturing Company. Testing was carried out as per TAPPI Standard Test Method *T511 su-69 Folding Endurance of Paper (MIT Tester)*. Ten sheets of each type of paper were randomly selected and then five folding strips of 15 x 110 mm were cut from each sheet. Samples were folded across the paper's machine direction.

Results

When retesting of the papers began it soon became obvious that the results for Burnie Bank and Canary Bank were identical, therefore to save time, Canary Bank paper samples were removed from testing. All results and discussions in the remainder of the paper only relate to the five remaining papers.

Folding endurance results for Permalife® Bond, Archive Text, Dry Process Copy, Plus Fabric Bond and Burnie Bank from air-conditioned (A/C) and non air-conditioned (non-A/C) after 27 years are shown in Table 1. Also given is the loss of strength expressed as a percentage of original strength.

Discussion

These results are highly informative. Clearly all papers lost strength over the 27 year period. However, strength loss was not consistent for all papers. By far the best performer was Archive text which at worst lost 15.02% of its strength – stored in non-A/C conditions in Perth - in other storage conditions it performed much better. The other archival paper, Permalife®, performed disappointingly, at worst losing 71.8% of its strength (stored in non-A/C conditions in Brisbane). One of the 'standard' papers, Dry Process Copying, actually performed better than Permalife® paper, consistently losing less strength. The other 'standard' grades of paper performed quite poorly with Plus Fabric Bond performing slightly better than Burnie Bank.

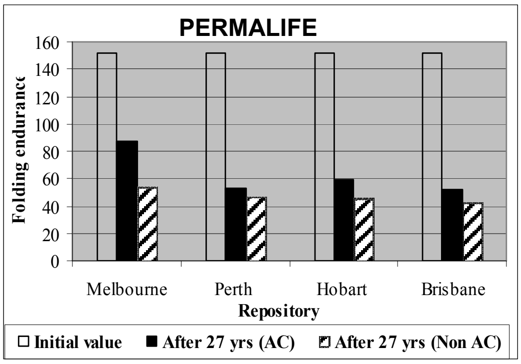
When comparing the different storage locations, given that we are uncertain as to the actual conditions in which the papers were stored, care is required in interpreting results. However, the first thing to be noted is that in almost all cases papers performed better in A/C storage than in non-A/C storage. This confirms that A/C storage is preferred for the long-term preservation of paper materials. However, a look at the results for non-A/C storage across the various cities gives some interesting results. The worst strength retention results were obtained for papers stored in non-A/C conditions in Perth and Brisbane. Both these cities have hot and moist climates, and given that these conditions would generally transfer into a non air-conditioned building, the greater degree of deterioration can be attributed to these conditions. This premise is supported when looking at results for Hobart, the coldest city, which actually achieved non-A/C storage results close to those for A/C.

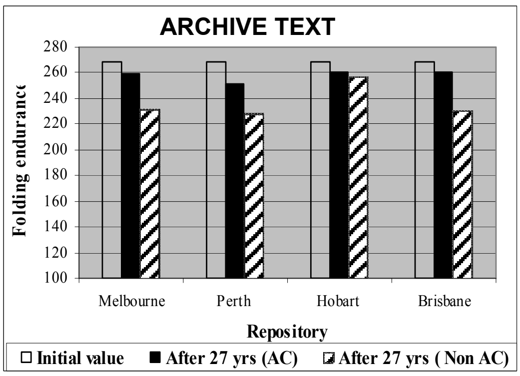
Table 1: Average folding endurance results for fresh papers in 1980 compared with those for naturally aged samples stored in both A/C and non-A/C environments

SN	Paper Type	Repository	Initial value (1980)	After 27yrs in AC		After 27yrs in non-AC	
				Average Fold value	Average % lost	Average Fold Value	Average % lost
1	Permalife	Melbourne	152	87.7	42.3	53.9	64.5
		Perth	152	53.2	65.0	46.5	69.4
		Hobart	152	59.5	60.9	45.6	70.0
		Brisbane	152	51.6	66.0	42.9	71.8
2	Archive Text	Melbourne	269	259.8	3.4	232	13.8
		Perth	269	251.2	6.6	228.6	15.0
		Hobart	269	260.2	3.3	257.1	4.4
		Brisbane	269	260.6	3.1	230.8	14.2
3	Dry Process Copying	Melbourne	100	39.6	60.4	39.2	60.8
		Perth	100	43.0	57.0	42.5	57.4
		Hobart	100	42.7	57.3	41.6	58.4
		Brisbane	100	46.7	53.3	37.7	62.2
4	Plus Fabric Bond	Melbourne	83	36.3	56.2	35.6	57.1
		Perth	83	16.6	80.1	26.0	68.7
		Hobart	83	31.5	62.1	30.0	63.9
		Brisbane	83	22.5	72.9	21.7	73.9
5	Burnie Bank	Melbourne	96	23.9	75.1	23.5	75.5
		Perth	96	23.7	75.3	23.2	75.8
		Hobart	96	27.3	71.5	27.2	71.7
		Brisbane	96	25.9	73.0	12.9	86.6

This seems to indicate that the variations observed in the non-A/C results for each city are due to the conditions prevailing outside the storage space. One could argue here that for particularly cold and dry climates there may be no need or advantage in air conditioning storage spaces. There are anomalies however; some papers displayed the biggest difference between results for A/C and non-A/C in Melbourne, which presumably should have milder ambient conditions that Perth or Brisbane. This is where our lack of exact knowledge of the conditions in which the samples were stored over the past years makes it difficult to draw firm conclusions.

A factorial ANOVA analysis[2] of this data was carried out to determine if there was a significant difference between results for samples stored in A/C and non-A/C. This analysis found that for both archival papers, Archive Text and Permalife®, there was a significant difference in percentage loss, whereas there was no significant difference for Burnie Bank, Plus Fabric Bond and Dry Process Copying paper. The analysis also found significant differences between cities. A set of graphs comparing the folding values of all papers before and after naturally ageing is shown in Figure 3.





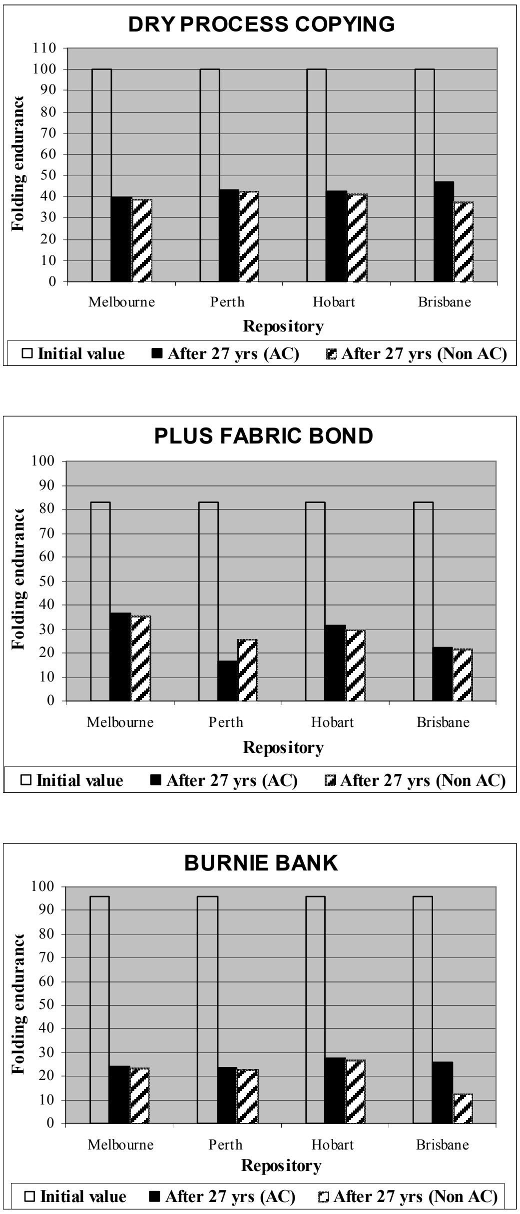


Figure 3: Folding endurance values for each paper before ageing and after natural ageing in both A/C and non-A/C conditions

pH analysis of the papers

For the purposes of pH analysis, samples of the same paper from different locations were grouped together, giving an average result for all environmental conditions. The pH was measured as per Australian/New Zealand Standard™ *AS/NZS 1301 421s:1998 Determination of the pH value of aqueous extracts of paper, board and pulp – cold extraction method.* Table 2 gives these results along with other information for the purposes of comparison: pH as measured in 1980, the folding endurance measured 1980 and the average folding endurance for the naturally aged papers after 27 years.

Table 2: Average pH and folding endurance results before and after natural ageing for each paper sample

Paper Types	Initial pH (1980)	pH after 27 yrs	Initial Folding Endurance (1980)	Folding Endurance after 27 yrs
Archive Text	7.1	8.7	269.0	258.0
Permalife Bond	8.3	9.0	152.0	63.0
Dry Process Copying	5.6	5.4	100.0	43.0
Burnie Bank	5.1	4.5	96.0	25.2
Plus Fabric Bond	5.0	4.5	83.0	26.7

Results and discussion

There are some surprising results from this testing. Of particular interest are the papers that were alkaline in 1980, both of which have become more alkaline after 27 years. The accepted understanding is that papers become more acidic as they age: however, when pH was measured using the cold extraction method, it was found that these papers have become more alkaline. The most obvious explanation that can be given for this is that over time the alkaline elements of the papers, for example calcium carbonate, have become more water soluble, resulting in a higher cold extraction pH. The acidic papers all behaved as expected, dropping in pH and losing strength as time has passed. The initial testing, in 1980 (Batterham et al. 1980, 1982) of these acidic papers showed the presence of alum and lignin. It seems a fair assumption that these elements would have contributed to the increase in acidity. The results overall suggest that alkaline papers age better than acidic papers; however, there is one major anomaly: Permalife®, which despite its high alkalinity does not perform well in terms of folding strength over time.

FTIR analysis

Samples of the naturally aged papers were examined using Fourier transform infrared (FTIR) spectroscopy to see what might be discovered about the chemical reactions contributing to the strength and pH changes observed. Analysis was carried out using a Thermo Nicolet Nexus FTIR spectrometer in attenuated total reflection (ATR) mode.

The ATR technique measures the changes that occur to an infrared beam as it interacts with the test sample when it is reflected inside a diamond crystal. When the beam comes into contact with the sample the internal reflectance creates an evanescent wave in the interface between sample and crystal. When the sample absorbs certain wavelengths of energy in the infrared region based on the molecular vibrational modes of the sample being analysed, the evanescent wave is attenuated. The attenuated energy from each evanescent wave is passed back to the infrared beam, which then exits to the opposite end of the crystal and is passed to the detector in the spectrometer. The system then generates an infrared spectrum characteristic of the material being analysed.

The sample is placed onto the crystal area of 1.5 mm in diameter and pressure is applied to press the sample hard against the crystal surface. Each spectrum shown in this paper is an average of 32 scans over the range of 500 – 4000 cm^{-1}.

Paper is a complex material with cellulose as its main component. Cellulose is a polymer of β glucose linked through 1-4 β glucoside bonds (Figure 4).

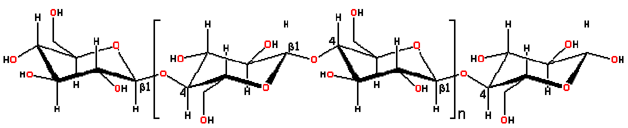


Figure 4: The structure of cellulose

There are different mechanisms that can cause the deterioration of paper, one of the most important being oxidation of the cellulose. The oxidation of cellulose involves the primary and secondary hydroxyl groups of the pyranose ring which results in the creation of carbonyl (C=O) and carboxyl groups (-COOH). These groups are chromophores and their creation is one of the reasons paper yellows as it ages. The formation of carboxyl groups increases acidity and induces depolymerisation of the cellulose, as a result the physical and mechanical strength of the material decreases.

Results and discussion

The FTIR spectral results for all papers were quite similar so the spectra given here are for one paper: Archive Text for A/C and non-A/C conditions in Melbourne. Figure 5 shows these FTIR spectra along with a reference spectrum for cellulose analysed from a Whatman filter paper sample.

Both the A/C and non-A/C samples show a strong absorption in the region 1200 to 950 cm^{-1} which is the 'fingerprint' region of cellulose. This absorption is mainly due to the stretching of C-O and C-C and rocking of CH_2. Thus a drop in the peak height and peak area in this zone suggests a breakdown of some of the cellulose molecules.

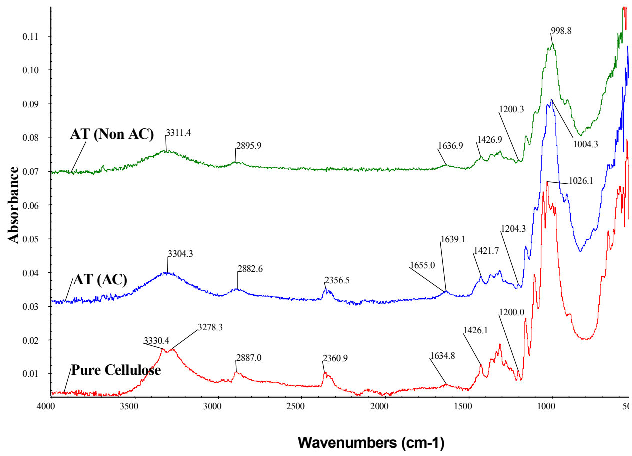


Figure 5: The FTIR spectra of Archive Text from A/C and non-A/C environments in Melbourne, and an FTIR reference spectrum of cellulose

Cellulose is highly hygroscopic, the degree of which depends upon the number of hydroxyl groups (-OH) in the molecule. The greater the number of hydroxyl groups the greater the chance of forming hydrogen bonding with water molecules. If the cellulose is oxidised, its hydration capacity decreases as there will be fewer hydroxyl groups to form hydrogen bonds. Hydroxyl groups vibrate in the region around 3300 cm^{-1} and consequently a smaller peak height and peak area here indicates that a portion of the cellulose has degraded.

Examination of the spectra for A/C and non-A/C papers gave results that are expected: for both the hydroxyl peaks and those in the cellulose fingerprint region, the peak area and peak height are greater for A/C paper than those for non-A/C paper. This confirms that storage in uncontrolled conditions accelerates the degradation of cellulose.

All paper types also had a peak around 1655 cm^{-1} which indicates the presence of carbonyl groups (C=O) which are a product of the degradation of cellulose. Comparing the peak area and peak height for samples from A/C storage and non-A/C storage reveals that in all cases non-A/C storage had a greater number of carbonyl groups and thus this cellulose was more degraded.

Comparing artificially and naturally aged paper

Rasch and Stone (1932) of the U.S National Bureau of Standards carried out one of the first comparison tests between natural ageing and heat ageing. The study resulted in two important conclusions:

- On the basis of folding endurance data there is a rough correlation between 22 years of natural ageing and three days of artificial ageing at 100°C.
- When natural and artificial ageing are compared, there is little correlation between chemical tests but a good correlation between physical tests

This equivalence was also supported by other researchers and was thus incorporated into TAPPI Standard Test Method *T453 Effect of dry heat on properties of paper and board*. For the purposes of simplicity the standard increased the ageing temperature to 105°C which gave an equivalence of three days (72 hours) of accelerated ageing to 25 years natural ageing (from here on referred to as the TAPPI formula). The date we have obtained in this testing gave us a chance to examine this equivalence and see whether it could be confirmed.

Because the testing carried out in 1980 provided folding endurance results for a series of artificial ageing periods it was possible to plot the paper deterioration graphically. At the time, it was realised that these graphs were close to linear when expressed logarithmically (Batterham et al. 1980). An example of these graphs, for Archive Text, is shown at Figure 6.

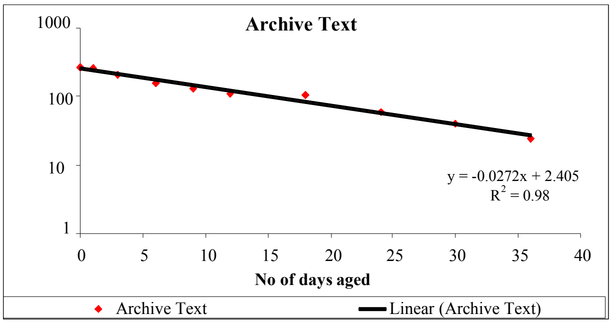


Figure 6: Folding endurance plotted against number of days artificial ageing at 105°C for Archive Text, using results obtained from testing in 1980

This linear form allowed for the simple prediction of folding endurance at any time period, when used in conjunction with the TAPPI formula. If, for example, you wanted to know how much strength Archive Text would retain after 1000 years, you could convert this to artificial ageing days using the TAPPI formula: giving you 120 days at 105°C. When this figure is placed into the formula for the regression line, a fold value of 5.6 is calculated.

Now that we have a set of figures for naturally aged papers we are in a position to see how well these artificial ageing graphs predict natural ageing, and whether they support the TAPPI formula that states that three days at 105°C approximates 25 years of natural ageing.

As an example of how this question was examined, the sample of Archive Text kept in A/C storage in Melbourne gave a folding endurance value of 260 after 27 years of natural ageing (see Table 1). When the equivalent number of days for this folding endurance value are calculated using the linear regression plot determined from our 1980 testing and shown in Figure 4, it equated to 0.996 days, which, using the TAPPI formula translates to 8.3 years, quite different from the actual result of 27 years. Put simply, this shows that the regression graph, used together with the TAPPI formula does not give an accurate prediction of ageing behaviour in relation to folding endurance. Further doubt is thrown on the TAPPI formula when looking at the results for the other papers, given in Table 3.

Table 3 clearly shows that each paper has its own unique equivalence value between artificial and natural ageing. The only paper that performed as predicted by the TAPPI formula was Dry Process Copying paper. The rest of the papers aged naturally in quite a different fashion from that predicted by artificial ageing and the TAPPI formula: some papers aged more rapidly than predicted, others less rapidly.

These results tell us that it is impossible to make a simple prediction of the ageing performance of a paper based on artificial ageing. It also tells us that there is no 'typical' ageing performance of a paper: each paper ages in its own way, both naturally and artificially. It would therefore be unwise to make predictions of expected longevity of paper based on this artificial ageing regime, at least in terms of folding endurance.

Table 3: Comparing folding endurance values (average of four repositories) for fresh papers, and for papers after 27 years of natural ageing and equating in terms of years by applying the formula from TAPPI *T453*.

	Archive Text	Permalife Bond	Dry Process	Plus Fabric Bond	Burnie Bank
Folding Endurance value for fresh paper tested in 1980	269	152	100	83	96
Average Folding Endurance after 27 years of natural ageing	258	63	43	27	25
Predicted Folding Endurance value after 27 yrs*	46.77 (y = -0272x + 2.41) R^2 = 0.98	26.30 (y = -0.023x + 2.05) R^2 = 0.92	3.10 (y= -0.045x + 1.7) R^2 = 0.81	11.88 (y= -0.021x + 1.64) R^2 = 0.71	2.04 (y= -0.066x + 2.099) R^2 = 0.91
Actual ageing formula†	27 years NA‡ = 1 day AA# at 105°C	27 years NA = 9 days AA at 105°C	27 years NA = 3 days AA at 105°C	27 years NA = 8 hours AA at 105°C	27 years NA = 16 hours AA at 105°C

* from 1980 artificial ageing, based on linear regression equation (regression equation given); † based on these natural ageing results and the linear regression from 1980; ‡ Natural ageing; # Artificial ageing

Conclusions

The formula given in TAPPI Standard Test Method *T453 Effect of dry heat on properties of paper and board*: 3 days artificial ageing at 105 ± 2°C equates to 25 years natural ageing, has been found to be significantly inaccurate for folding endurance. Each paper seems to have its own individual ageing performance and papers can perform much worse or much better than predicted by the formula. Folding endurance results were also found to be highly variable for papers from the same ream or even across the same sheet.

The degradation of paper depends to some degree on its storage environment. This is indicated by the results obtained for the papers stored in non-A/C conditions which showed better strength retention for papers stored in milder climates than those stored in more fluctuating climates.

In this study the two archival, higher quality papers were shown to degrade more slowly in A/C than in non-A/C. However, the three lower quality 'standard' papers tested appear to degrade at a rate that is more independent of environment.

The degradation of paper depends to some extent upon the acidity level (pH) of the paper, with acidic papers tending to degrade faster than alkaline papers although there are some exceptions to this rule.

In this study, as expected, the three papers which were initially acidic increased in acidity after 27 years. However, the alkaline papers increased in alkalinity over the same period. This latter unusual, and unexpected, result may be an artefact of the cold extraction method of determining pH.

FTIR spectroscopic analysis can be utilised to detect changes in the cellulose component of paper that are indicative of the deterioration path of the paper sample.

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Notes

1. This paper was originally classed as 'Archival' for the 1980 testing, but testing results suggested otherwise.

2. Factorial analysis of variance (ANOVA) is a statistical model used to study the effects of two or more treatment variables. For this analysis; $p < 0.0001$.

AUTHOR BIOGRAPHIES

Ian Batterham was in the first intake of students for the (then) Canberra College of Advanced Education (CCAE) Materials Conservation course in 1978. On graduation he went to work for the National Archives of Australia (NAA) where he remains to this day. He achieved his Masters Degree in Conservation and the University of Canberra (UC) in 2000. Over the years, at different times, he has worked as a lecturer in paper conservation at UC. He has had numerous papers published including one in the journal *Restaurator* on conservation work he carried out on the Walter Burley Griffin Canberra Designs. His first book *The Office Copying Revolution* is to be launched at the AICCM Book and Paper Symposium in July 2008. He is currently in the position of Assistant Director, Operations Development at the NAA where he writes preservation policy and carries out Preservation-related research.

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Rajani Rai obtained her Masters Degree in Chemistry (Organic) from the Tribhuvan University in her native country Nepal. She worked as an assistant lecturer in the Department of Chemistry at the Engineering College in Nepal for three years. In 1995, she came to Australia to pursue a Masters Degree in Applied Science at the University of Canberra. She then worked there as a research assistant for two years. In 2002, she joined the National Archives of Australia as a Conservation Chemist in the NATA accredited paper testing facility.

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