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Accelerated Aging of Paper: Can It Really Foretell the Permanence of Paper

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Introduction

After more than a century of scientific investigation into the permanence, or lack thereof, of paper (Kantrowitz, 1940; Fellers, 1989), this field of inquiry is more fertile than ever. We, and our predecessors before us, have done our utmost, and we still wonder about exactly how paper ages. This is a tribute to the complexity of paper and the everchanging, constantly advancing technology of its manufacture.

Many issues remain to be resolved, but few merit as much concern as the development of a consensus around a sound and practical accelerated aging methodology. Such a development would in itself serve to resolve many other long-standing issues.

The present NISO, as well as ISO standards for permanent paper are heavily weighted towards specifying the composition of paper, rather than its performance (NISO, 1992; ISO, 1994). While the recent revision of ISO 9706 improved upon the NISO permanent paper standard by incorporating an accelerated aging test, it did not do so to replace any of the composition related requirements.

Composition-based standards leave much to be desired. From a fundamental perspective, the consumer, whether a librarian, an archivist or a conservator, is ill-equipped to tell the paper maker how to make paper. However, the consumer does know the end qualities and performance he/she would like to see in the product. The real problem in the case of permanent paper standards is that the consumer does not have a credible set of tests for quality control on which he/she can depend. That is why the consumer is forced to depend on the composition of the product, for which presumably adequate test controls are available. However, a composition based set of requirements is always less efficient and more vulnerable than a performance-based standard.

Consider, for example, the NISO standard, which mainly requires (1) a minimum alkaline reserve of 2% calcium carbonate equivalents, (2) a maximum limit of 1% on lignin content and (3) minimal tear resistance, which is adequate enough to allow the printing of paper. A paper product can meet all of these requirements, and still not be permanent if it were contaminated with significant concentrations of copper, iron or other oxidative catalysts, free radical chain initiators or oxidants. Any of these contaminants would have the potential to accelerate the degradation of paper in spite of the presence of the specified alkaline reserve. Even if other testing requirements were to be introduced to cover this gaping hole in the standard, the consumer would still be none the wiser

regarding the effects of any number of paper chemicals and additives that could have been introduced in its manufacture, or in the processing of the recycled content.

Not only does this composition-based standard allow the possibility of an unstable paper being passed off as permanent, but more importantly, it excludes truly permanent papers, such as a good rag paper, which may last indefinitely even if it lacked an adequate alkaline reserve. Let me hasten to add that the NISO Committee that revised the standard recently, was well aware of these problems, but had little choice. Therefore, the only real solution that would be in the best interests of the consumer and the manufacturer alike, is to develop an accelerated aging test, or a set of such tests, that will distinguish between permanent and impermanent paper in a reliable, reproducible and efficient manner. But that seems like a tall order today.

Protestations against accelerated aging are not hard to come by in the literature on paper permanence and in personal communication among scientists and conservators. A few of the more recent, as well as eloquent works are cited here.

Bansa and Hofer subjected a naturally aged paper to further aging at temperatures ranging from 50 to 95°C (Bansa, 1989). Their work convinced them that there was no correlation between natural and accelerated aging. They suggest that if at all we must depend on artificial aging, it must be at 80°C and 65% RH.

In a more recent work, Bansa expresses further frustration with accelerated aging techniques (Bansa, 1992). With the aid of extensive accelerated aging data, he has attempted vainly to find reproducible degradation patterns with different papers. He is convinced that accelerated aging experiments can lead to conclusions that may not only be doubtful, but can even be "deceitful." He points to several findings from his data that perplex him: These include the lack of any predictability in the observed decay patterns (some are curved upwards, others downwards, while some remain flat), the unexplained stability of a paper sample with a pH of about 5, and also the stability exhibited by alkaline groundwood papers.

Stroefer-Hua (1990) has presented a thought-provoking examination of basic assumptions on which accelerated aging experiments are generally based, and questions their validity. He believes that Arrhenius plots for the aging of paper are not straight lines, and therefore the mechanism by which paper degrades at higher temperatures is probably quite different from that which prevails under ambient conditions. For these reasons, accelerated aging of paper seems nothing more than a futile exercise to him, and he finds that even relative inferences drawn from such tests may not have any merit. He presents a thesis that each paper has a unique history that determines how that paper will age in the future, and accelerated aging experiments cannot predict that pattern.

Regardless of rampant skepticism in accelerated aging techniques, there is no substitute for the process in the research laboratory. Scientists engaged in research on the permanence of paper have no choice but to employ one or more of the artificial aging techniques presently available, and to temper their observations from such tests with

caution. It behooves us therefore, to look a little deeper into one of our best tools and hone it as best as we can. Any efforts in this direction are bound to be richly rewarding.

Status of Accelerated Aging of Paper

At this time, there is practically universal agreement that the presence of moisture during the aging process is essential. Substantial evidence exists today to demonstrate conclusively that different reaction mechanisms dominate under dry and humid aging conditions. Most recently, David Erhardt has used GC-MS analysis to study the accelerated aging of paper. He has compared degradation products extracted from Whatman filter paper aged under humid and dry conditions (Erhardt, 1987). Glucose and xylose, products of hydrolytic degradation of cellulose and xylan, were the dominant reaction products for papers aged at 90°C and 100% RH. Dry oven aging at 90°C and above gave product mixtures with very little glucose, demonstrating that hydrolysis of cellulose was not the dominant reaction under relatively dry aging. Even under dry conditions, aging at 150°C and above produced a different degradation product distribution than that observed at lower temperatures.

Three different sets of humid aging conditions have been favored by most workers: 80°C - 65% RH, 90°C -50% RH and 90°C -25% RH. To the best of my knowledge, the ISO 9706 Standard Committee did not consider the 90°C -50% RH condition, but compared the other two humid aging conditions and decided in favor of the 80°C -65% RH aging condition for incorporation in the ISO standard. This was a bold, ground-breaking decision by the ISO Committee to place their confidence in an accelerated aging test despite the mounting resistance and criticism of such testing over the past several years. However, the practical significance of this specification has yet to be demonstrated. One wonders whether vendors would be agreeable enough to withdraw a product if the failure of the product is determined several weeks after delivery. Now that an accelerated aging requirement has been introduced in an international standard, it can always be improved upon if necessary. Such a need may catalyze further research efforts.

Research in Accelerated Aging of Paper

Research on accelerated aging is one of the primary interests at the Library of Congress. Several investigations have been in progress at the Swedish Institute of Pulp and Paper Research (Frojd, 1992). Tommy Iversen is comparing natural and artificial aging processes in order to assess the relevance of artificial aging. Petter Kolseth is engaged in a critical review and evaluation of accelerated aging methods. He has compared aging of paper under several temperature and relative humidity condition combinations, including 80°C/65% RH, 90°C/50% RH and 90°C/65% RH.

Several other workers have an active research interest in the investigation of artificial aging, including David Erhardt at the Conservation Analytical Laboratory of the Smithsonian Institution, Helen Burgess and Klaus Hendriks at the Canadian Conservation Institute and Paul Whitmore at the Carnegie Mellon Institute remain actively interested in

this area. This is certainly not an exhaustive list of all the laboratories with an active interest in this field of investigation. Thus, much is happening in this area, and we may yet see significant advances over the next few years.

Comparison of Accelerated Aging of Paper in Stacks and Sheets

Several years ago at the Library of Congress, we undertook an investigation aimed at studying the effect of cycling relative humidity and temperature conditions on the accelerated aging of paper (Shahani, 1989). For these experiments it was obvious that a paper in a bulk, such as in a book, would absorb and desorb moisture more slowly than a single sheet of paper. Therefore, we compared the effect of cycling relative humidity conditions on the aging of the same paper samples as single sheets, as well as in 100-sheet thick stacks.

In this study two different wood pulp papers, one an unsized, unadulterated waterleaf, and the other, an alum-rosin sized 15-year old paper, were aged at 90°C and constant relative humidities of 40, 50 and 60 percent and also a relative humidity that cycled constantly between 40 and 60 percent. The result of these experiments, which is not really relevant to the present subject was that the cycling humidity conditions did not exert a measurable effect on the aging of the paper in a stack, while the same paper in single sheets deteriorated significantly faster due to the fluctuating relative humidity. But from our present perspective, a much more interesting result of these experiments was that paper within a stack aged faster than the same paper aged as single sheets that were free to interact with the environment.

These data supported an observation from a condition survey of pre-1840 paper records at the Philadelphia Center of the National Archives, that paper within bound volumes was invariably weaker than loose rolled up sheets inside ventilated cardboard boxes (Shahani, 1980). Extending our earlier work on the comparison of aging of single sheets with paper within stacks, we undertook the aging of a few selected papers over a wide range of temperatures (60 through 90°C). Some of these data obtained with Springhill™ offset, an alum-rosin sized bleached Kraft book paper, are shown Figure 1.

All of the experimental data presented in Figure 1 and elsewhere in this work were obtained with Springhill™ offset paper, which was manufactured by International Paper. This paper was obtained as a 6-foot wide roll. A 3-inch width was discarded from both ends, and the rest sectioned into 1 1-inch wide rolls. Eight-inch long sheets were cut from the same 1" wide roll for use in each set of these experiments. All papers were preconditioned according to TAPPI T402, then conditioned at 73°F and 50% RH for at least 24 hours for single sheets, or for at least 4 weeks in the case of stacks of paper, before being sealed inside an enclosure or before introducing them into temperature and humidity controlled aging chambers.

Turning our attention back to Figure 1, the data at 60°C should have evidently been pursued for a longer time interval. For the rest of the data obtained at 70, 80 and 90°C, it is clear that the paper inside a stack ages appreciably faster than a single sheet hung on a rack. Unlike the single sheets, the paper inside the stack is not in immediate contact with the air and moisture flowing through the humid aging chamber. The only explanation that suggests itself is that degradation products formed in the aging process accumulated inside the stack of paper accelerate its degradation.

It is remarkable that significant concentrations of the degradation products are retained within the paper mass in spite of the high vapor pressures that must prevail at 90°C, even though these stacks of paper were not sealed or enclosed in any way. The top sheets were kept in place by a 1/8-inch thick Plexiglas™ sheet. The high affinity of the degradation products for paper suggests a high degree of polarity. Also, the acidity within the stacked paper was about a half pH unit lower than that of the single sheets. These observations, along with the fact that the stacked paper aged faster, suggest that at least some of these accumulated degradation products must be acidic.

It was also noticed that the paper aged within stacks had an odor somewhat reminiscent of old books. While one generally notices an odor in humid aging chambers where even single sheet samples are being aged, especially at the times that the chamber door is opened to remove samples periodically, we cannot recollect any persistent odor in single sheet samples, whether aged in a dry or humid oven.

An important feature of the data in Figure 1 is that while the single sheets lose strength at a constant rate, the rate of degradation of the paper within stacks increases constantly, due in all probability to the increasing acid concentration within paper.

Aging of Paper Sealed within Polyester Film

Further evidence of the significance of acidic degradation products for the aging process came from a parallel study from our laboratories that investigated the effects of "encapsulation" of paper within two sheets of clear polyester film. Such an encapsulation treatment is frequently used in paper conservation as a protective measure to shield precious and fragile documents from physical stress, as well as from dust, etc. Some of the findings of this work are shown in Figure 2. These data were also obtained with Springhill™ Offset paper at 90°C and 50% RH for the following paper samples:

1. Sheets encapsulated inside sheets of 4-mil thick polyethylene terephthalate (PET) film by sealing off all four edges completely,
2. sheets sealed inside half-open PET envelopes, sealed along two adjacent edges,
3. sheets encapsulated along with an alkaline paper,
4. sheets deacidified with aqueous magnesium bicarbonate and then encapsulated by sealing along all four edges, and
5. control sheets which were not treated in any way or encapsulated.

Paper encapsulated without deacidification became brittle by the tenth day of aging, as compared with the control which took twice as long to lose most of its strength. Even half-sealed paper aged just as rapidly as paper sealed completely. This is a clear indication of the affinity of the degradation products. They are probably hydrogen-bonded to the cellulose matrix.

Another possible explanation for the strong tendency of paper to retain these degradation products could be that as the paper ages and its morphology changes, the degradation products get trapped inside folding chains as the degree of crystallinity increases. However, other experiments not described here have clearly demonstrated to us the tendency of paper within books to retain polar compounds such as ammonia almost indefinitely at room temperature. To some of you this will suggest immediately an opportunity for affordable mass deacidification of paper, which is indeed another direction in which we are pursuing research in our laboratories.

Returning to our discussion of the data in Figure 2, the retention of much of its physical strength by deacidified paper and the stabilization of paper gained through mere contact with alkaline paper within the PET envelope, add further credence to the assumption that the degradation products are indeed acidic in nature. The autocatalytic effect observed in Figure 1, for paper in stacks is again evident for encapsulated sheets of paper.

Only one logical scenario can explain all of the observed facts in Figures 1 and 2: Acidic compounds produced as a result of the aging of acidic paper are retained by a paper matrix that is isolated from free interaction with the environment. As the concentration of these acidic species increases with aging time, the rate of acid hydrolysis of cellulose accelerates. This process is autocatalytic, that is, it feeds upon itself. An alkaline buffer incorporated in paper, or even in contact with an alkaline paper stabilizes acidic paper by neutralizing the acidic degradation products.

The fact that acidic degradation products are produced in the accelerated aging of acidic paper in a humid environment, was clearly established at NBS in the seventies (Parks, 1971-1972). However, these data did not point to the accumulation of these acids within a paper mass, such as a book, and the subsequent autocatalytic nature of the degradative process.

Indeed, it was not too long ago that an expert panel assembled by the National Academy of Science for the National Archives and Records Administration suggested that the rate of aging of acidic paper decreases with time (National Academy of Science, 1986). The inference was that acidic paper, even if it is in a brittle state, will last indefinitely if it is not misused or mishandled. This view enjoys wide support among the library and archival community simply because it makes intuitive sense. After all, few of us have ever observed a book self-destruct by crumbling to dust.

However, this view that the rate of degradation of paper decreases constantly and reaches a plateau is based not on any specific observation or experiment, but rather on the general shape of accelerated aging curves obtained from accelerated aging of single sheets,

particularly those showing the decreasing slope of fold endurance loss with progression of time.

What is often overlooked in such plots is that the fold endurance value cannot fall below zero, even though the paper keeps degrading in every other manner that is measurable (as shown later in Figure 4). However, laboratory data presented here suggest that the decline in physical properties of acidic paper does not slow down with increasing deterioration; it does not even continue to decline constantly at the same rate as for single sheets in Figure 1, but instead it continually gathers momentum as acidic degradation products continue to accumulate. Like a rock rolling down a slope, the decline in physical, as well as chemical properties of acidic paper within books or other isolated micro-environments, continues to accelerate until the paper becomes unusable.

Several other workers have stressed the important role that acidic degradation products play in the aging of paper. Work reported by Klaus Hendriks recently also showed a faster rate of aging of paper within paper stacks as compared with aging of single sheets (Hendriks, 1994). Marianne Bjorklund Jansson at the Swedish Pulp and Paper Research Institute is engaged in the identification of degradation products formed in the aging of paper and their comparison in naturally and artificially aged paper. She has already identified a few low molecular weight acids, namely, acetic, propionic and levulinic acids.

It is reported that the correlation between these and other cellulose and hemicellulose degradation products and paper strength parameters is being tested using multivariate data analyses (Frojd, 1992). In the US, the National Institute for Standards and Technology completed a preliminary study for the identification of degradation products in the presence and absence of sulfur dioxide (Parks, 1991).

Inadequacy of Single Sheet Accelerated Aging Methods

We believe we have presented enough evidence here from our laboratories and from other workers to show that accelerated aging methods which facilitate the volatilization of degradation products by exposing single sheets to a constant current of air and moisture may be missing an important element of the manner in which paper ages within a mass of paper that is bound into a book, or housed in a closed container. When we open an old book, we would be surprised if it did not have a characteristic smell. Yet the accelerated aging methods we have practiced thus far never develop a noticeable odor. Our accelerated aging methods have been missing an essential element of the aging process. Therefore, it is not surprising that we find none of them really satisfactory.

Accelerated Aging within Sealed Enclosures

A more realistic accelerated aging of paper can be achieved by equilibrating it to a characteristic moisture content under standard conditions, and then enclosing it in an air-tight container before subjecting it to an elevated temperature.

Aging within sealed enclosures has been undertaken previously by several workers (Richter, 1954; Czepiel, 1960; Browning, 1968, MacClaren, 1980). However, until now the intent of all such experiments has been to retain the moisture content of paper. Also, since these studies mainly compared the aging of sealed humidified paper against open sheets in a dry environment, they attributed the observed increase in the aging of acidic paper inside sealed environments to the presence of moisture and missed the added effect due to the accumulation of acidic degradation products.

At the Library of Congress, we have undertaken extensive experimental work for the aging of paper within sealed enclosures. These experiments have included aging of paper within aluminized PET bags and also within glass tubes. In Figure 3, we present some of this work, which compares the loss of fold endurance for different amounts of paper sealed within aluminized PET bags and aged at 100°C. Also shown in the same figure data for paper within stacks aging at 90°C and 50% RH to provide a frame of reference.

Although the temperature for aging of the paper in a stack is ten degrees lower, yet the enhancement of the rate of degradation achieved within an air-tight enclosure is evident. An increasing mass of paper within the same enclosure leads to a faster rate of degradation. This observation is easily explained. A higher amount of moisture within nearly the same space may understandably raise the level of relative humidity within the enclosure and thereby, the rate of degradation of paper. However, in this respect this work presents data contrary to the observations reported by Richter (Richter, 1954).

Comparison of Accelerated Aging Methods

The aging of Springhill™ offset paper, a typical alum-rosin-sized book paper from a few years ago, was undertaken under several different aging conditions. Experimental data on the deterioration of physical, optical and chemical data were obtained. However, for the sake of brevity, only the data on loss of fold endurance are summarized in Table 1. For each aging condition, a *relative lifetime* " ***T*** ", which equals three half-lives for the MIT fold number, is calculated as a convenient measure of the rate of degradation.

As might be expected, single sheets aged at 80°C /65% RH age just a little more slowly than those aged at 90°C /25% RH. Stepping up the relative humidity from 25 to 50% and maintaining the temperature at 90° doubles the rate of degradation. At 90°C and 50% RH, paper in stacks ages twice as fast as single sheets. Sheets sealed inside PET or polypropylene, which is less permeable to moisture, aged at about the same rate as the sheets within paper stacks when both, the stacks and the encapsulated sheets aged at the same rate. However, when the same encapsulated sheets were aged in a dry oven at 90°C, the rate of deterioration of paper slowed by as much as six times. Evidently, the porosity of the plastic films permits the moisture to escape, reducing the moisture content of paper. Finally, sheets sealed within aluminized PET and aged at 100°C are shown to have a relative lifetime of only 6 hours when 10 sheets are enclosed inside the sealed bag, as compared with 15 hours for a single sheet inside the same enclosure. These relative lifetimes compare with 12 days for the same paper when aged conventionally as single sheets at 80°C and 65% RH.

There is no virtue in a mere acceleration of the rate of degradation. However, it is a big plus when it is accompanied by a more realistic aging method and also does not require specialized and expensive equipment such as a humid aging chamber. Humid aging experiments have also been carried out in closed containers over salt solutions. However, having learned the role that degradation products play in the aging of paper, one might question the use of such experimental setups since all solutions would tend to absorb these compounds.

Further work in progress in our laboratories is aimed at the study of a variety of variables in sealed enclosure aging. These include the effect of moisture content and air volume inside the enclosure, and a comparison of activation energies among different accelerated aging methods. If the Arrhenius relationship does not hold at higher temperatures, the rate of this aging technique is fast enough to allow us to go to appreciably lower temperatures. In other experiments the degradation products from different accelerated aging methods will be compared with those extracted from naturally aged books. The greater the number of commonalities we can find with natural aging, the more acceptable an accelerated aging method will become.

We need to introduce a word of caution about the use of aluminized PET bags. Within a few days of the aging process, degradation of PET reaches such an advanced state that the whole bag just crumbles. We believe that acid hydrolytic degradation of polyester induced by the products of degradation of cellulose is the cause. An empty bag without any paper in it does not degrade noticeably even after weeks of aging. Therefore, we have discontinued the use of such bags and are now using capped glass tubes, which are much more convenient too.

Accelerated Aging under Light

It would be a serious omission to limit this presentation on accelerated aging of paper exclusively to aging in the dark. Traditionally, most accelerated aging experiments have excluded the effect of light, probably because most library materials are not expected to see a significant exposure to light. However, with the possibility of inclusion of appreciable lignin content in permanent paper of the future, accelerated aging in the presence of light will undoubtedly be practiced more widely. For those of us who need to undertake light-aging experiments, a useful handbook/guide has been published by Robert Feller (Feller, 1990). This work includes an extensive methodology for such aging experiments, as well as the underlying theory and principles.

Measurement of Rates of Degradation

The experimental setup for accelerated aging, which we have discussed extensively so far, provides only part of the methodology that needs to be standardized. We also need a broad consensus on what properties of paper we need to quantify the degradation of paper as it ages. Ideally, we should measure selected properties among each of the three dimensions that paper presents to us, namely, physical, chemical and optical.

Most research laboratories come to develop a preference for a selected set of tests that they may have practiced over long stretches of their history, just as there are tests that are liked, there are also those that are not liked. However, few tests for physical testing of paper arouse as much passion as the fold endurance test. It has been said that this test is not functional, it is too time-intensive, and that it fluctuates too wildly to be of any practical use. The NISO committee on permanent paper saw it fit to drop this test from its requirements and place its confidence entirely on a tear resistance requirement.

At the Library of Congress, we have recently completed a lengthy study on a comparison of a host of physical and chemical properties. In this study, three very different papers were subjected to accelerated aging at 90°C and 50% RH for a period of 90 days. The decline in several physical, chemical and optical properties was measured periodically. Selected data for some physical properties are shown in Figure 4. The percent loss in MIT fold endurance, tensile energy absorption, tear and burst is presented as a function of aging time for a single test paper. Clearly, the fold endurance test is much more sensitive than the other three tests. While percent loss in tear resistance is only about 35 percent and that in tensile energy absorption is almost 40 percent, fold endurance has lost 90 percent of its initial value. The sensitivity of this test more than makes up for its high standard deviation.

In fact, its high standard deviation is mainly a result of its high sensitivity to the homogeneous structure of paper. Besides, the high standard deviation can easily be lowered by an appropriate statistical treatment if desired. However, for measuring the physical properties of weak paper, fold endurance is at a decided disadvantage, unless it were to be modified in some way. This is not intended to be an exhaustive discussion of the physical properties of paper, for it would not be complete without an honorable mention of the zero span tensile test.

Conclusion

We have not yet answered the question we posed at the outset: Can accelerated testing really foretell the permanence of paper?

We may never be able to duplicate the natural aging of paper in the laboratory. It is also doubtful if we can confidently predict the exact lifetime of a given sample of paper. However, it may not be beyond our capabilities to capture the essential elements of the natural aging process of paper, and accentuate them in a laboratory system so that we may obtain a reliable measure of the relative permanence of paper samples. What is needed most is a test that can differentiate between good, long-lasting paper samples and relatively less stable paper, in a reproducible manner and with a high degree of confidence.

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Table 1.
Accelerated Aging of Springhill Offset™ Paper Under Different Conditions

Aging Conditions	Temperature (˚C)	Relative Humidity (%)	Relative Lifetime ***T****
Single sheets	80	65	12
Single sheets	90	25	10
Single sheets	90	50	4.8
Stacks of 100 sheets	90	50	2.5
3 sheets sealed inside PET/PP	90	50	3.0
3 sheets sealed inside PP	90	<10	17
Single sheets sealed Inside A1 + PET	100	<5	0.63
10 sheets sealed Inside A1 + PET	100	<5	0.25

**T* = Time to three half-lives

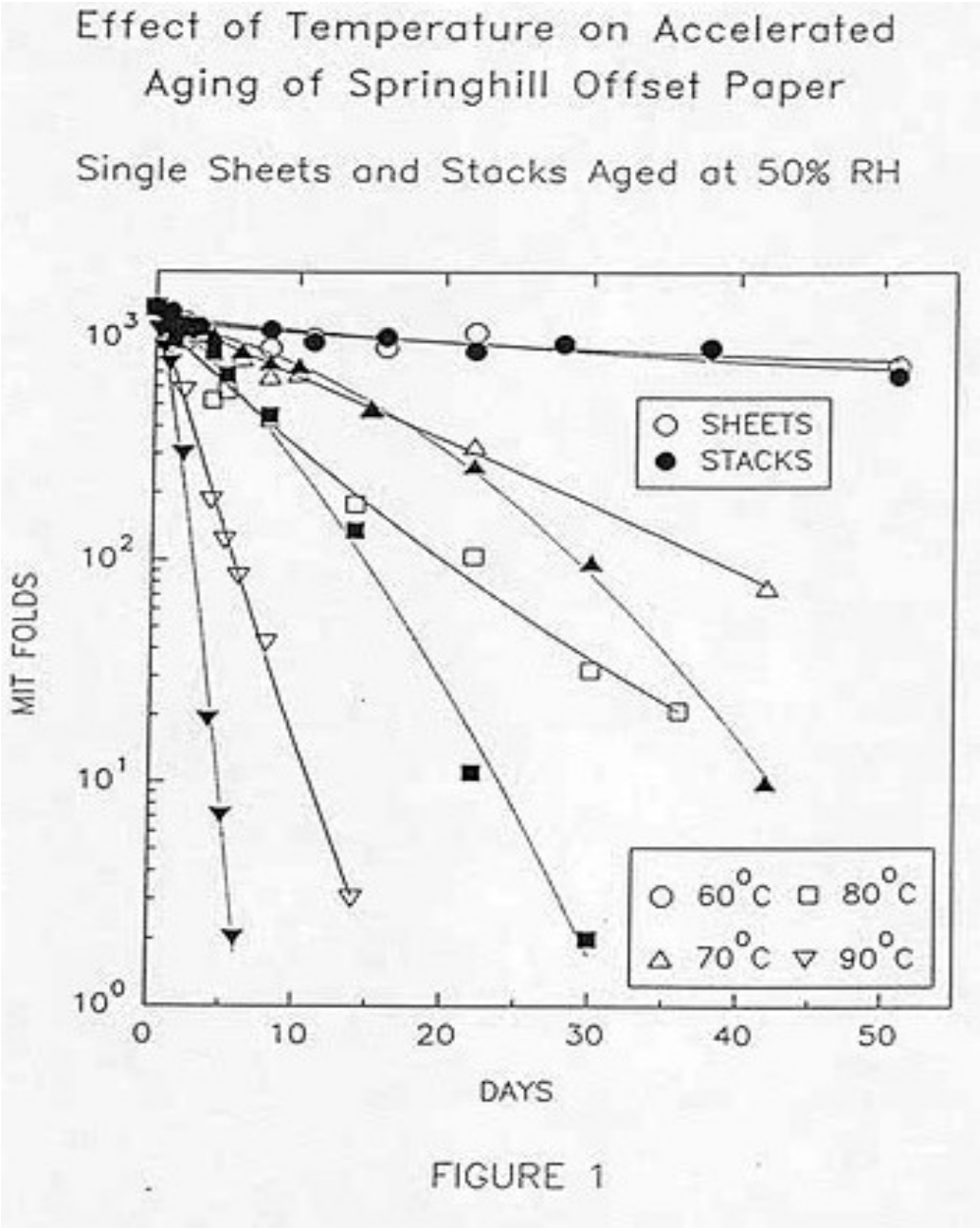
Effect of Temperature on Accelerated Aging of Springhill Offset Paper
Single Sheets and Stacks Aged at 50% RH
MIT FOLDS
10^3
10^2
10^1
10^0
0
10
20
30
40
50
DAYS
SHEETS
STACKS
60°C
80°C
70°C
90°C

FIGURE 1

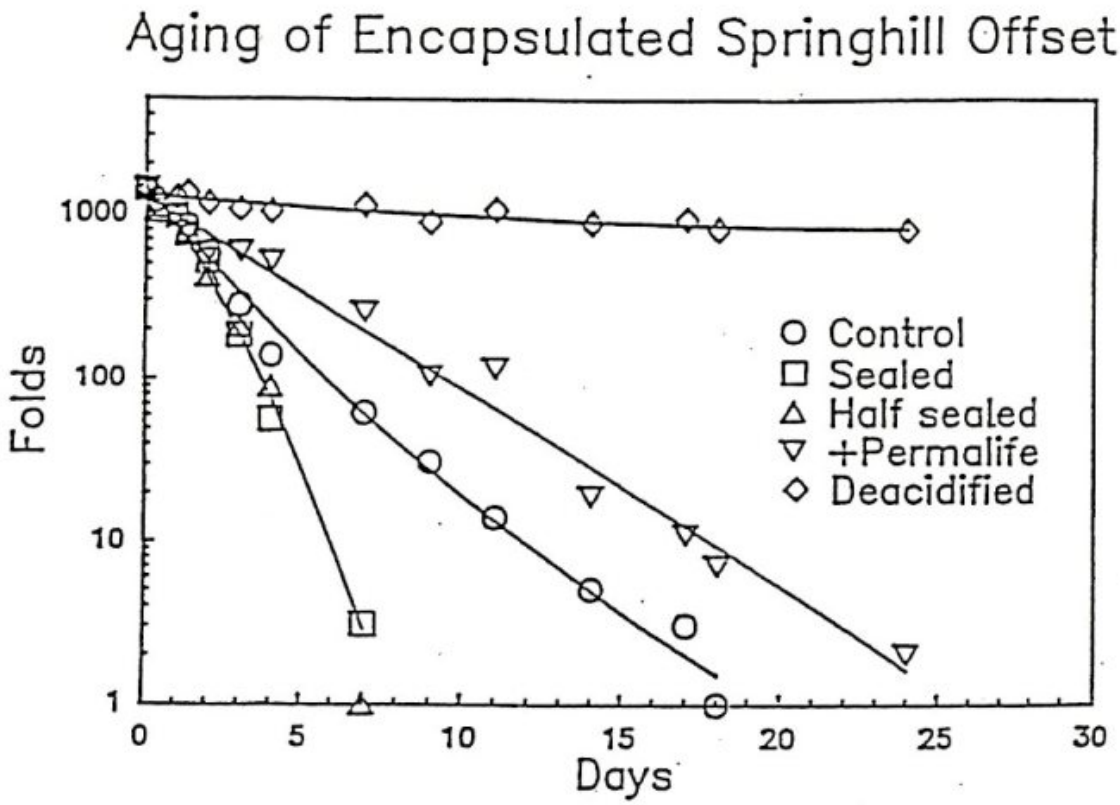
Aging of Encapsulated Springhill Offset
Folds
1000
100
10
1
0
5
10
15
20
25
30
Days
Control
Sealed
Half sealed
+Permalife
Deacidified

FIGURE 2

Accelerated Aging of Springhill Offset Paper within Aluminized PET Bags

Effect of Number of Paper Sheets

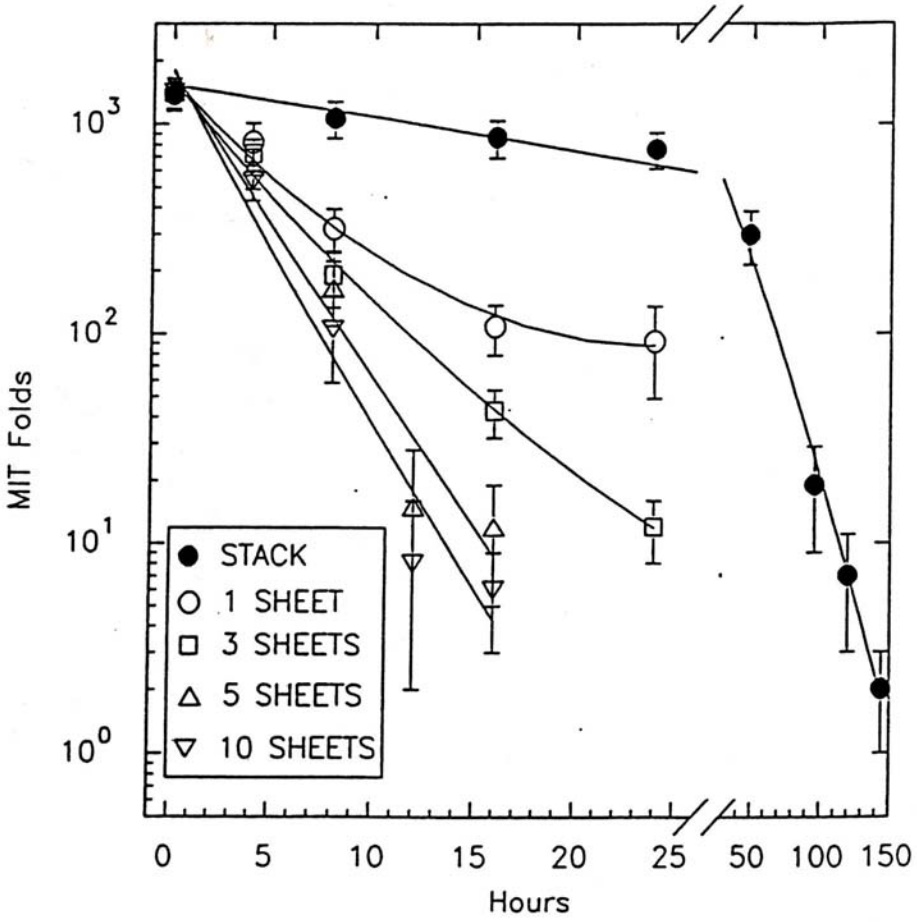

FIGURE 3

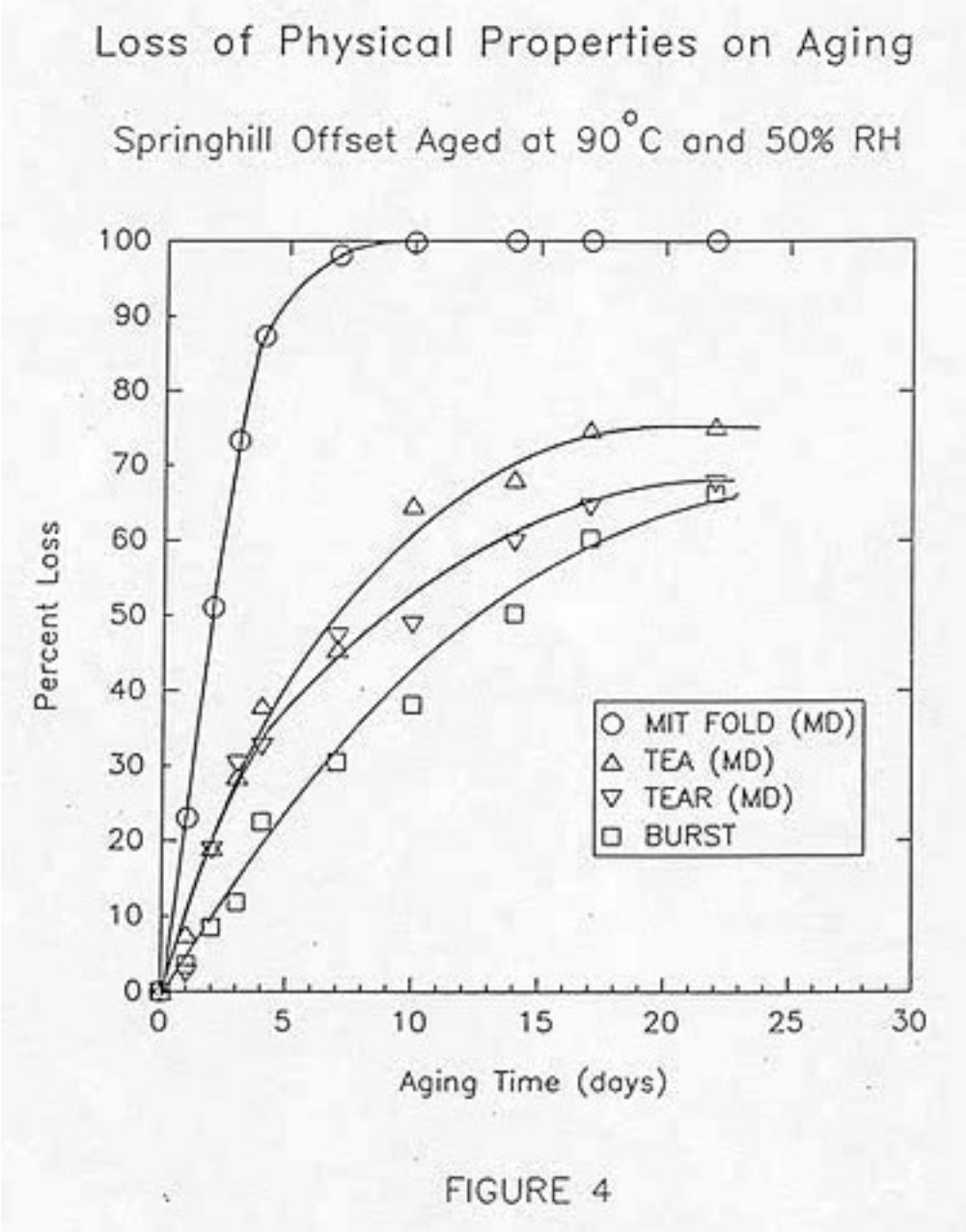
Loss of Physical Properties on Aging
Springhill Offset Aged at 90°C and 50% RH
Percent Loss
Aging Time (days)
MIT FOLD (MD)
TEA (MD)
TEAR (MD)
BURST
0
10
20
30
40
50
60
70
80
90
100
0
5
10
15
20
25
30

FIGURE 4