

# **EVALUATION AND COMPARISON OF COMMERCIAL MASS-DEACIDIFICATION PROCESSES: PART 1- PROJECT PLANNING AND SELECTION OF MATERIALS**

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## **I INTRODUCTION**

The degradation and embrittlement of paper by acid is a serious problem for most libraries and archives. An important part of dealing with these chemically unstable materials is the neutralization of the acid and the deposition of a buffer reserve to protect the fibres from the introduction of future acid. Any deacidification technique chosen must be able to treat large numbers of items, safely, quickly and cheaply. However, institutions across North America, have found it difficult to make decisions concerning which processes should be applied to their collections. The need to preserve millions of books at the cost of tens of millions of dollars makes it critical that the correct decisions be made. Feasibility studies such as that undertaken by the Lord Cultural Resources in Toronto<sup>1</sup> have concluded that there is insufficient technical information available at this time, to allow institutions to make these decisions. What is required is accurate experimental data on how the various processes compare in terms of their effectiveness; what degree and type of screening must be performed for the different technologies; and what materials are best treated by what methods.

In 1990, the Canadian Conservation Institute (CCI), agreed to carry out an evaluation of commercial mass-deacidification processes for the National Library of Canada. Coincidentally, the Metro Toronto Chairman's Committee for Preserving Documentary Heritage (CCPDH) was planning to do an independent technical evaluation of mass-deacidification technologies. After discussions with CCI, the National Library and the CCPDH agreed to coordinate research efforts. The result was that later that same year, the project was redefined to include a broadened scope of topics and became part of the technical plan by the CCPDH to collect and interpret information concerning the selection of a suitable mass-deacidification method for Canadian collections. The CCI/CCPDH project is financed by contributions from a large number of institutions in Canada and the United States. A list of financial contributors is given in Appendix A.

The testing programme involves the assessment of the three most promising commercial mass-deacidification processes: the Wei T'o, Diethylzinc, and FMC's MG3 (Lithco) technologies. The Wei T'o treatments will be carried out at the National Archives of Canada; diethylzinc at the AKZO plant in Texas, and the MG3 process at the FMC facility in North Carolina. The Wei T'o and FMC processes are solvent based

and involve the deposition of an alkaline buffer reserve. The solvents used and the chemicals deposited (magnesium carbonate in the case of Wei T'o; butoxytriglycol/magnesium carbonate (MG3) in the case of FMC) are quite different and there are strong technical reasons to predict that they will not act in an identical manner on book materials. The diethylzinc method is gas phase and results in the deposition of a near neutral pH buffer reserve (zinc oxide). The FMC process is the only one claiming to strengthen paper directly. However, the companies marketing all three processes claim that paper stabilized through the application of their technologies, will result in paper which in years to come, will be stronger than similar paper which has not been deacidified.

## **II PROJECT OUTLINE**

### **2.1 Effect of Mass Deacidification on Paper**

The project was divided into four parts. The first and most important phase is an evaluation of the effect of the various deacidification processes on the paper which forms the book block. Attempts have been made to cover as wide a range of materials as is possible within the limitation of time and resources that the project must operate.

#### **2.1.1 PAPER TYPES TO BE SELECTED**

The paper which will be examined falls into three groups: (1) naturally-aged paper, (2) paper aged through artificial means, and (3) new paper. The three groups are important for different reasons. Much of the chemical data available on deacidification has been collected through the testing of new papers. This means that relatively little is known about how already degraded paper will react under treatment conditions. As naturally-aged and degraded paper forms a very significant portion of most North American collections, it is an important area to investigate.

However, naturally-aged paper often is not homogeneous. This creates problems with interpretation of data. Therefore, the decision was made to produce a second group of damaged papers by taking homogeneous new paper and subjecting it to artificial ageing. The ageing is being carried out in a humid oven under the conditions of 80°C and 50% relative humidity. The presence of moisture during ageing is critical for paper. The most important mechanism for the degradation of paper is acid-catalysed hydrolysis which is strongly dependent on the amount of water available in the paper substrate.

A third group of papers is comprised of the new papers chosen for group two. They will act as a type of control for group two as well as provide valuable information about how these new papers are affected by mass deacidification. The investigation of the new papers is important for two reasons:

1. Many institutions place priority on the treatment of new paper as acidic new fibres have more to gain from deacidification than already degraded paper.

2. As yet, there are no published chemical data that allow a direct comparison of the three mass-deacidification methods.<sup>a</sup> Information is available on individual processes but there have not been any published studies in which the same paper was used for testing of all three of the processes. The best data from a purely statistical point of view will come from the experiments carried out on new papers.

Both of these points form a persuasive argument for including new papers in the CCI experiments.

Each group of papers includes three types of paper: 100% rag, lignin-free processed wood pulp, and ligneous wood pulp. These papers span the full range of the fibre types which are common in North American collections. It was considered important to include all these types of paper because it is likely that mass deacidification will affect each one in a different manner. It is also likely that the type and amount of size and fillers could have an important impact. However, time and resources are limited and there are no immediate plans to carry out a comprehensive study in which the type and amount of size and/or filler are principal experimental variables. Instead, CCI chose papers which were sized with a material which is representative of the type used most often with that particular paper. For example, the rag paper is sized with gelatin, the processed wood pulp is sized with alum/rosin and the ligneous wood pulp is unsized. In the past, ligneous wood pulp paper has been sized with alum/rosin, but just as frequently was left unsized. Today, this type of paper is almost never sized by the manufacturer. The papers chosen for experimentation are either unfilled or filled with relatively small amounts of material.

Even though the mass-deacidification study does not include a full examination of the effect of type of size on process effectiveness, this problem is being examined in another part of CCI's current research programme, i.e., an investigation of aqueous deacidification methods. This study is financially supported by the Conservation Committee of the Canadian Council of Archives. It is anticipated that information gained from the project will be useful in the formulation of recommendations for the non-aqueous mass methods.

A last point concerning the papers being tested in both the aqueous and the non-aqueous deacidification studies is that they are all uncoated. Research concerning coated papers involves a large number of experimental parameters and would considerably lengthen the time and effort required to come to any conclusions.

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<sup>a</sup> The raw data from the Library of Congress' RFP became available in October, 1991 but as yet, no interpretation of results or conclusions have been formulated and published.

## 2.1.2 ANALYSIS

Samples of all of the papers in the various groups are being analysed before deacidification, after deacidification, and after accelerated thermal ageing. Four types of samples are involved: unaged & aged undeacidified material and unaged & aged deacidified material. The conditions for the artificial ageing are 80°C and 50% relative humidity, the same as that used to pre-age the group of new papers. The time of ageing will vary depending upon the fibre content of the paper and its degree of degradation.

The physical and chemical changes in the samples are being monitored by a number of analytical procedures. The chosen techniques include the following:

### 1. *Quality Control*

(a) Surface and cold extracted pH- using standard procedures developed by the Technical Association of the Pulp and Paper Industry (TAPPI).<sup>2,3</sup>

(b) Alkaline reserve- a titrimetric method that involves analysis of the amount of buffering present in the paper. CCI uses a modification of the TAPPI Standard<sup>4</sup> in which a back-titration method is used and the end-point is determined with a pH meter and combination electrode. The procedure is more time consuming but the results are more accurate than that obtained with the unmodified TAPPI method. The ASTM standard<sup>5</sup> is even less accurate than the unmodified TAPPI standard as it relies on the use of a methyl red colour indicator that changes from a pink to a yellow colour over a wide pH range of 4.2 to 6.2.

### 2. *Physical Testing Methods*

(a) Colour change- three types of data are being collected: (a) brightness based on reflectance at 457 nm; (b) Hunter L\*a\*b values which describe the type and amount of colour shifts which occur; and (c) delta E values (calculated from L\*a\*b) which give a good over-all estimation of colour change.

(b) Zero-span tensile strength measurements are being carried out. In a limited number of cases, Instron tensile testing will also be performed. The tensile method is being used in preference to fold endurance for a number of reasons:

- (i) zero span is less dependent on small environmental changes and so less data scatter is observed
- (ii) zero span allows one to analyse papers which are weaker than those which can be tested with fold endurance
- (iii) zero span is a measure of the intrinsic strength of fibres and hence is better related to degree of chemical degradation; in the strictest sense of the definition, fold endurance is not a strength test, but instead is a measure of other physical properties such as brittleness

- (iv) zero span requires less sample material than most other physical testing methods.

### **3. Chemical Methods of Analysis**

(a) Degree of polymerization- analysis measures the average length of the cellulose molecules which are the principal component of paper. Consequently, monitoring changes in degree of polymerization gives an extremely accurate idea of the amount of chemical degradation that a particular paper undergoes during treatment and/or accelerated ageing. The method chosen by CCI involves the use of the solvent cadoxen and is based on viscosity. As the paper fibres degrade, they tend to result in a less thick solution when dissolved. The degree of thickness or viscosity of sample solutions is determined and can be accurately converted to a number representative of the average length of the molecules that make up the paper sample.

(b) Carbonyl analysis- quantifies the amount of a particular chemical grouping present in the paper sample (i.e. aldehyde and ketone groups). These groups, called carbonyls, are formed when oxidation occurs. Therefore, this procedure will be carried out for those samples in which it will be useful to have a measure of the amount of oxidative damage which has occurred. CCI uses a method involving the derivatization of the paper fibres with a phenyl-hydrazine compound. It gives more accurate data than other commonly used methods for estimating degree of oxidation. (Note: An increase in carbonyl content can be attributed mainly to oxidation; acid hydrolysis contributes relatively small amounts of aldehyde functional groups.

(c) Magnesium or zinc content of the paper- together with the alkaline reserve, this gives an excellent description of the amount of buffer reserve plus how much acid has already been neutralized in the paper. The principal quantitative method being used for estimation of magnesium or zinc content is atomic absorption techniques.

Scanning electron microscopy (SEM) combined with microprobe elemental analysis also is being used to obtain some semi-quantitative data indicating what materials can be found on the surface of the paper. Analysis of a cross-section (i.e. through the "z" direction of the sheet) of the treated papers should give some information about degree of penetration of the paper by the deacidification compounds.

A limited number of analyses (surface pH & magnesium or zinc content by atomic absorption) are being used to map the distribution of buffering salts over selected sheets of paper. Data should give valuable information concerning the evenness and completeness of the deacidification of the various paper types under study.

The results for the first phase of the project will give data that will allow for an excellent differentiation of the three processes, based on their effect on the chemical stability of paper. However, the papers chosen will be either new or of an average

degree of degradation for their particular age and fibre content. Many of the papers in library or archive collections are especially degraded for one reason or another. Therefore, a second phase was developed in which CCI will investigate what effect mass deacidification has on papers which are degraded in some pre-determined manner.

## **2.2 Effect of Mass Deacidification on Degraded Paper**

Four papers will be included in the second phase of the study. Two of them will be naturally-aged rag paper, one of high to medium degree of polymerization, and one of medium to low degree of polymerization. The third and fourth papers studied will be ligneous, one naturally aged, and one new.

The four papers will be degraded by two different processes. The first will be extensive exposure to sulphur dioxide and nitrogen dioxide polluted air. The concentrations of the air pollutants will be only slightly above what can occur in a heavily polluted urban area. At this time, it is planned to model the exposure after a similar set of experiments which was sponsored by the Getty Conservation Institute in Los Angeles.

The second degradative treatment will be immersion of the papers in an aqueous oxidative bleaching bath. Alkaline hypochlorite will probably be used as it has been employed, historically, in both the manufacture of paper as well as in restoration treatments. The accelerated ageing and analytical evaluation will be similar to that used for Phase I.

It is recognized that people in charge of the preservation of book collections will need to know more than just how mass deacidification affects the book block. Therefore, a third phase of the project was designed in order to evaluate media and other materials found associated with books.

## **2.3 Effect of Mass Deacidification on Media, Bindings and Special Paper Types**

The variation of materials found in book and paper collections is tremendous and it will be impossible to carry out a full scientific study of such a wide range of materials. Therefore, the initial analysis will rely mainly on a careful visual evaluation of the materials before and after mass deacidification. This will be followed by identification of problematic material. Samples of these materials will be processed again but with some simple scientific testing being done before and after treatment. This phase will also include a few special types of paper such as the coated stocks referred to earlier, which could not be included in the first two phases. The bulk of this work is being carried out by a conservator, Sherry Guild, who has been seconded from the Paper Lab at CCI for the duration of this phase of the project.

The materials to be tested are being taken from a list compiled through suggestions from participating institutions. This information is coming from a

questionnaire which was distributed in June, 1991 to over a hundred institutions across North America, Europe, and Australia. In the questionnaire, institutions were asked to identify the contents of their collections, as well as give information concerning their preservation needs. In addition to identifying which materials CCI should test, this questionnaire also indicates which institutions are able to donate material for testing. CCI does not have its own collection and so this cooperation on the part of other institutions is vital to the success of the project. The conservator will also be compiling a list of materials derived from discussions with commercial printers and binders. An important part of this third phase of the project will be an open house at CCI in which all contributors to the questionnaire or project will be invited to come to CCI and view the treated materials.

#### **2.4 Effect of Mass Deacidification on Protein Materials**

The last phase of the research project involves the evaluation of the effect of mass deacidification on protein materials. Protein breaks down easily under alkaline conditions and so there is much concern over the deposition of an alkaline reserve into book components which contain proteins. Leather or parchment bindings, glue adhesives, protein sizes, and the gelatin or albumen layer associated with photographs are the most common sources of protein materials in books. For this part of the study, CCI will evaluate changes in pH, colour, polymer length, and physical strength, before and after accelerated ageing.

#### **2.5 Project Schedule**

We estimate that the four phases of the project will require around forty person months of laboratory work. In addition, there has been an initial period of six months which covered the selection of the papers to be tested in Phase I. This has been an extremely important part of the study. The correct papers must be chosen if the data collected is to be interpreted and applied in a meaningful way to actual collections.

The experimental phase of the project began in January, of 1991, with the hiring of a contract scientist, Elzbieta Kaminska, who is working at CCI under the direction of Helen Burgess. A second contract scientist, Aranka Boronyak-Szaplanczay began work on the project in October, 1991.

### **III SELECTION OF PAPERS**

This section summarizes the work carried out in the first eight months of Elzbieta Kaminska's contract. It describes the selection of the paper to be used in Phase I of the project, i.e. four naturally-aged papers and four new papers.

### 3.1 Screening Methods

The criteria used in the selection of the papers were the following:

- a) availability of sufficient quantities of homogeneous paper to carry out analyses
- b) source and type of processing of the fibre
- c) degree of degradation (as measured by degree of polymerization)
- d) soluble cellulose content
- e) type of sizes and fillers.

The suitability of specific papers for the experiments was determined through the use of a number of analytical procedures. They were as follows:

1. visual examination for homogeneity
2. phloroglucinol test for lignin
3. Liebermann-Storch test for rosin
4. iodine test for starch
5. biuret test for protein
6. aluminon test for aluminium
7. surface pH
8. cold water extraction pH
9. moisture content
10. viscometric average degree of polymerization
11. determination of soluble cellulose content (lignin content)
12. colour and brightness measurements
13. SEM-microprobe analysis for general information on inorganic content
14. FTIR analysis for presence of protein
15. light microscopy analysis to distinguish wood-pulp fibres from those of rag fibre origin.

The first six procedures<sup>6</sup> were useful for sorting papers into broad categories, e.g. lignin and lignin-free papers, sized and unsized papers, etc. The determination of average degree of polymerization and soluble cellulose<sup>b</sup> (insoluble fraction is lignin content) and the colour measurements proved to be the most useful for final selection of papers within those broad groups.

Sixty-four papers were analyzed during this screening process. The "old" papers were obtained from second-hand book stores and from materials sent by various institutions contributing to the project. The new papers were obtained as samples from several paper mills and paper distributors. The results of the screening tests are given in Tables 1 & 2.

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<sup>b</sup> The insoluble fraction contains complexes which are made of a high percentage of lignins.



### 3.2 Homogeneity of Naturally-Aged Paper

The chemical and physical homogeneity of the chosen paper is essential in ensuring that one is able to draw direct comparisons among the various treated and untreated samples. New paper will be extremely homogeneous as long as it is machine made and all obtained from the same roll. Hand-made new paper can or cannot be homogeneous depending upon the skill and intent of the paper-maker as well as the type of fibres used. There are significant difficulties in obtaining homogeneous, naturally-aged material in quantities suitable for carrying out experiments of this type<sup>c</sup>. Therefore, in order to locate suitable experimental materials, it was necessary to analyze many more naturally-aged samples than new papers.

Initially, it was thought that the most likely source of suitable material would be from books available in sets (e.g. encyclopaedia). A number of prospective sets were obtained based on the evenness of their visual appearance and apparent brittleness (by manual single-fold test). The chemical homogeneity of papers was determined by estimation of degree of polymerization (DP). Preliminary measurements were carried out on samples randomly taken from several locations and different volumes from three individual sets of books (i.e. *Chums*, *Exposition of Holy Scripture*, and *Report of the Department of Public Records and Archives of Ontario*; see Table 1).



Figure 1. Degree of polymerization (DP) of various volumes of *Chums* (designated by Year of Publication).

*and Report of the Department of Public Records and Archives of Ontario*; see Table 1).

The results showed substantial differences in degree of polymerization of the samples taken from the same sets (see Table 1 and Figure 1). These results strongly suggested that it would be difficult to select four complete books having similarly degraded paper. In view of this conclusion, it was decided to select one book showing a relatively high and homogeneous average degree of polymerization and divide it into four portions (one part for a control and three parts for the three mass-deacidification processes). The volumes of *Chums* published in 1920 and 1927 were

<sup>c</sup> Our first estimates were that we would require 4 or 5 books of each type of naturally-aged paper chosen.

selected for this purpose as they had the highest values of degree of polymerization according to the preliminary determination (see Table 1 and Figure 1). Nine samples, in addition to those previously analyzed, were randomly taken from the 1927 volume and their degrees of polymerization were determined. The results are summarized in Table 3.

The results clearly showed that the paper in this volume is not homogeneous enough to be used in the project. Similar inhomogeneity of the paper was found within several sections of the 1920 volume, as shown in Table 4.

At this point, attempts were made to replace the viscometric method of determination of paper deterioration (a very sensitive but costly and time-consuming analysis) with a simpler, non-destructive measurement which might be correlated with the degree of polymerization (DP). For this purpose, paper colour (i.e. L\*a\*b values) was measured for nine papers selected from the 1927 volume of *Chums* and plotted against the previously determined values of DP (see Figure 2). This relationship showed a correlation coefficient of 0.76 ( $r^2 = 0.58$ ) which was judged unsatisfactory, even for screening purposes.

The problem of paper inhomogeneity was further studied through the analysis of the book *Jesu de Haresi Janseniana* by S. Dechamps. This book contains pages with a wide range of thickness, colour and degree of deterioration (for example, the degree of polymerization measured for six randomly selected pages ranges from 360 to 1030, the latter being associated with a clean, unprinted page at the end of the book). For the same book, the inhomogeneity of the degree of polymerization

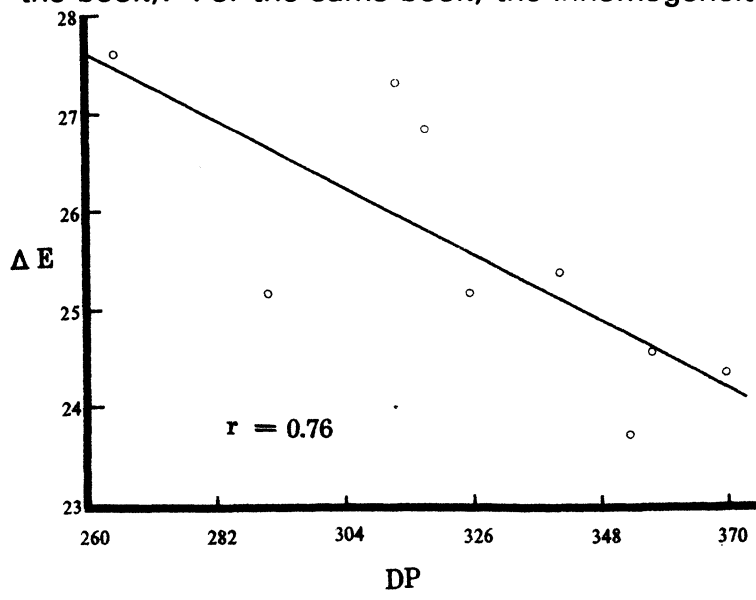


Figure 2. Change in colour of paper (measured by delta E) as a function of degree of degree of polymerization (DP).

within a single page (measuring 26 cm x 40 cm) was studied. The results are summarized in Figure 3. The variation in DP values within the page (about 6%) is comparable with that normally observed in an entire book of naturally-aged, machine-made paper.

A similar problem of inhomogeneity of single pages was encountered in connection with naturally-aged books having pages with wide margins. The differences in degree of polymerization between the printed (central point) and the unprinted (margin) areas of a single page were about 10%, as illustrated

by Table 5.

All of these experiments clearly show that there can be great difficulties in locating homogeneous naturally-aged papers. In particular, there are serious problems in obtaining suitable ligneous machine-made and rag hand-made papers.

### 3.3 Sample Preparation

#### 3.3.1 NATURALLY-AGED PAPERS

The studies of homogeneity of available, naturally-aged paper showed that no four complete books having similarly deteriorated paper would easily be available for research purposes. Under these circumstances, a different approach was followed. The decision was made to take "homogeneous" paper from a single book and rebind it in such a manner that four books are created. The goal was to use paper (for each set of 4 volumes) that was uniform in colour and did not vary more than 5 to 6% in DP. Four distinct paper types were selected:

1. *Jesu de Haeresi Janseniana...*; printed book; circa 1728; linen fibre; very small lignin content; positive gelatin; DP of approximately 650.
2. Ledger Paper; watermarked *Whatman Turkey Mill 1856*; circa 1856; linen fibre; positive gelatin; DP of approximately 660.

Spot	TOP OF PAGE			
	A	B	C	D
1	647			
2	698	732	714	703
3	648	687	689	681
4				
5	657	683	697	720
6	729			

3. *Expositions of Holy Scripture: Saint Mark*; printed book; circa 1906; processed wood pulp; negative lignin, aluminum, starch and rosin; DP of approximately 700.

4. *Summa Theologiae: Volume II*; printed book; circa 1948; ligneous wood pulp; positive lignin; negative starch, aluminum and rosin; DP of approximately 240 (50% soluble cellulose).

Two types of rag paper were included because surveys of 18th and 19th century book collections revealed two broad categories of rag paper: (a) a fully processed linen fibre that contains no lignin and is usually sized with

**Figure 3.** Variation of degree of polymerization (DP) within one page.

gelatin; and (b) an incompletely processed linen fibre that contains discrete particles of lignin and which is usually lightly sized with gelatin and is fairly absorbent. Previous experiments concerned with the effect of aqueous alkalization on paper showed that these two types of paper are affected in different ways by deacidification treatments.<sup>7</sup>

The sample books were made up as follows:

1. The 1728 book (*Jesu de Haeresi Janseniana* by S. Dechamps) was known to be inhomogeneous from the preliminary homogeneity studies. After a careful visual examination of the book combined with determination of DP of several additional samples, a group of pages showing DP > 550 was collected. These pages tended to be thicker and whiter in colour than the average. One of these pages was arbitrarily chosen as a "standard" and used for selecting pages closely matching its colour characteristics. Of the total of about 1000 pages, 18 pages met the imposed requirements and were used for preparation of the four required sample books. The paper (experimental pages) was cut into 36 sheets with dimensions of 13 x 20 cm (corresponding to 9 sheets per volume) which were put aside for binding.
2. The 1856 ledger paper was extremely homogeneous and no special selection procedures were necessary. The paper was cut into 20 sheets with dimensions of 21 x 26 cm (corresponding to 5 sheets per volume) which were put aside for binding.
3. The 1906 and 1948 books were fairly homogeneous. Eight sections of eight pages were taken from each book (64 pages); they were then separated into 4 equal parts of 16 pages each (experimental pages).

The individual piles of experimental pages were prepared for binding by first placing them between two stacks of "filler" paper. The total thickness of the assembled "book block" was about one inch. The choice of "filler paper" was based on the likelihood of it absorbing deacidification chemical in a manner similar to the "experimental" sheets.

The "filler" paper for the four paper types used is described below:

1. 1726 book- new bond paper, 25% rag content, acidic pH
2. 1856 ledger- new bond paper, 25% rag, acidic pH
3. 1906 processed wood pulp- paper from other volumes of same set
4. 1948 ligneous wood pulp- paper from the 1914 & 1917 volumes of *Chums*

In all cases except for the 1906 volume, the experimental pages were separated from the "filler" by a sheet of *Whatman No. 1* chromatography paper. Each "sandwich" was then bound into an individual book (see **Figure 3**) by a commercial binder. A standard buckram library binding was used. The pages of the 1906 and 1948 books were glued into place; the 1726 and 1856 volumes were sewn.

### **3.3.2 NEW PAPERS**

The criteria used in selecting the new papers was similar to the naturally-aged material. The main difference with this group of new papers is that the DP must be relatively high (i.e. more than 1000). As described earlier, the original plan was to study three papers: rag, processed wood pulp with alum/rosin sizing, and unsized ligneous wood pulp. This list was expanded to include an unbuffered, alkaline-processed wood-pulp paper. The testing of this last paper should be interesting as it will provide valuable conclusions about the extent to which this type of paper will benefit from mass deacidification.

In choosing the new papers, six companies in Canada and the United States were contacted. After ascertaining which companies manufacture the various types of paper required, requests were made for samples of these materials. Once again, the most difficult paper to locate was a suitable ligneous material.

After suitable analyses as described above, four new papers were chosen for experimentation:

1. rag water-colour paper; 100% cotton; hand made; 90 lb wt; hot press; gelatin sized; manufactured by Arches; negative lignin, rosin & aluminum; near neutral pH
2. processed wood pulp off-set paper; machine made; alum/rosin sizing; manufactured by Domtar under name of *Winsor*; negative lignin; positive aluminum & rosin; acidic pH
3. alkaline processed wood pulp off-set paper; machine made; neutral sizing; manufactured by Mohawk; negative lignin, rosin & aluminum; near neutral pH
4. ligneous wood pulp paper; unsized; manufactured by Canadian Pacific Forest Products; positive lignin; negative rosin & aluminum.

Each of the four types of paper was cut to an approximate uniform size (20 cm x 30 cm), and bound by a commercial binder in a standard buckram library binding (only one type of "experimental" paper in each book; no "filler" paper used). The pages were glued rather than sewn into place. Four books of each paper type will be used without pre-ageing (i.e. one control & three for the three mass-deacidification treatments). Four sets of four books will also be artificially aged and then treated.

## **IV SUMMARY**

The acid in library and archive materials is causing a serious problem with the deterioration of important collections. One possible method of dealing with this issue is through use of commercial mass-deacidification processes. However, there is insufficient technical information to allow decisions to be made regarding choice of

technologies. This scientific information is being obtained through a research project being carried out in the Conservation Processes Research Division of the Canadian Conservation Institute. The work is supported financially by a large number of institutions (see Appendix A) under the coordination of the Technical Committee of the Metro Toronto Chairman's Committee for Preserving Documentary Heritage.

The purpose of the investigation is to give information that will be helpful in deciding what parts of a collection can be deacidified as well as what process will be most suitable for what material. It is possible that recommendations will involve the suggestion that different processes be applied to different types of material. Furthermore, it is not likely that it will be feasible to institute any mass-deacidification process without at least some degree of selection and screening. The extent and type of the required selection process will become more clear when the investigation is complete.

After careful consideration of available information, it was decided that the investigation would concern three commercial mass-deacidification technologies: Wei T'o, diethylzinc (AKZO), and MG3 (FMC). It was also decided that the project would cover a variety of paper types and binding materials.

The experimental phase of the project began in early 1991 with the task of selecting appropriate papers for study. Analytical evaluation of over sixty naturally-aged and new book papers showed that it was not possible to select the required number of books that contained paper that was homogeneous enough to allow for a valid comparison of the three mass-deacidification processes. Consequently, one book was selected for each of four different types of naturally-aged paper and small portions of the selected book were bound into four individual books, together with additional, non-experimental sheets of a comparable type of paper. A standard buckram library-binding process was used. Similar books were prepared from four types of new papers.

The eight papers chosen were as follows:

1. an unbleached linen paper, 18th century
2. a bleached and very strong linen ledger paper, mid-19th century
3. lignin-free wood pulp paper, early 20th century
4. ligneous wood pulp paper, mid 20th century
5. new gelatin-sized cotton paper
6. new alum-rosin sized lignin-free wood-pulp paper (off-set)
7. new alkaline-processed, neutral-sized paper (off-set)
8. new ligneous wood-pulp, unsized paper.

The naturally-aged material has been sent to the three mass-deacidification facilities for treatment and subsequently has been returned to CCI. The books were cut into two, one half to be analysed without ageing, the other half to be analysed after artificial ageing. All artificial ageing of the naturally-aged material is complete

and analysis of the samples is underway. The first data from the deacidification of the naturally-aged material should be obtained by early 1992.

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## **APPENDIX A: Financial Contributors to Mass-Deacidification Project\***

AKZO Chemicals Inc.	Municipality of Metropolitan Toronto Archives
Archives of Ontario	National Library of Canada
Atlantic School of Theology Library	North York Public Library
Bibliothèque nationale du Québec	Ontario Legislative Library
City of Toronto Archives	Robarts Library
Committee on Institutional Cooperation	Toronto Public Library
Concordia University	University of Alberta
Conférence des recteurs et des principaux des universités du Québec	University of British Columbia
FMC Corporation	University of Manitoba
Harvard University	University of Toronto Library
Library of Parliament	University of Victoria
McGill University, McLennan Library	University of Waterloo
Metro Toronto Reference Library	Vancouver Public Library
Ministry of Culture and Communications	York Public Library
	York University

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\* List is based on financial contributions made before November 1, 1991.

**Table 1. Test Results from Screening of Naturally-Aged Papers**

Book or Paper Title	Year	DP	Other Characteristics
1. Rag paper	1823	375	---
2. Chums	1914	174	
3. Chums	1917	158	surface pH 3.9
4. Chums	1918	236	
5. Chums	1919	239	
6. Chums	1920	387	surface pH 4.36
7. Chums	1921	304	
8. Chums	1923	278	lignin + +, Al <sup>+3</sup> (-), starch (-)
9. Chums	1924	293	
10. Chums	1925	312	
11. Chums	1926	287	
12. Chums	1927	331	
13. Chums	1928	295	
14. Chums	1929	300	
15. Chums	1930	208	
16. Chums	1931	233	
17. Chums	1933	273	
18. Chums	1935	110	rosin (-)
19. Rag paper	1801	683	
20. Processed wood pulp	1900	307	
21. Sainte Beuve	1952	160	starch (-), lignin + +, rosin (+), Al <sup>+3</sup> (+)
22. Autour du catholicisme social	1908	255	starch (+), lignin + +, rosin(+), Al <sup>+3</sup> (+)
23. Raw Flax	modern	4891	
24. Raw Flax III (1)	modern	5464	
25. Raw Flax III (6)	modern	3471	
26. A Disquisition on Government and a Discourse on the Constitution by J.C.Colhoun	1854	478	lignin δ+, gelatin (-)



**Table 1 (continued)**

Book or Paper Title	Year	DP	Other Characteristics
27. Life of Jeremy Taylor	1839	241	lignin $\delta$ +
28. Le Bouclier Canadien	1928	199	67.8% sol. cellulose, rosin (-), lignin + +, starch (+), Al <sup>+3</sup> ?
29. Manuel illustré d'histoire de la littérature française	1947	149	lignin + +, rosin (-), Al <sup>+3</sup> (-), 41% sol. cellulose
30. Hommes et Dieux	1925	204	lignin + +, rosin (+), Al <sup>+3</sup> (+), starch (-), 34.5% sol. cellulose
31. Le Cahier by M. Proust	1928	200	lignin + +, Al <sup>+3</sup> (+), 32.7% sol. cellulose
32. Jesu de Hæresi Janseniana...	1728	360 to 1030	surface pH 4.8
33. Summa Theologiae I	1948	241	58% sol. cellulose, lignin + +
34. Summa Theologiae II	1948	210	52% sol. cellulose, lignin + +, cold extraction pH 4.3, surface pH 4.2
35. Summa Theologiae III	1948	209	
36. Report of the Department of Public Records and Archives of Ontario (grey cover)	1903	322	lignin + +
37. --- (orange cover)	1903	344	lignin + +
38. ---	1915	233	lignin $\delta$ +
39. ---	1920	156	lignin $\delta$ +

**Table I (continued)**

Book or Paper Title	Year	DP	Other Characteristics
40. Report of the Department of Public Records and Archives of Ontario (orange cover)	1929	276	lignin $\delta$ +
41. ---	1930	185	lignin $\delta$ +
42. ---	1931	275	lignin $\delta$ +
43. ---	1932	171	lignin $\delta$ + +
Expositions of Holy Scripture			
44. Isaiah vol. I	1907	218	surface pH 4.4
45. Isaiah vol. II	n.d.	705	lignin (-), Al <sup>+3</sup> (-), starch (-), rosin (-)
Isaiah vol. II	n.d.	---	
46. Genesis	n.d.	288	
47. Ezekiel	1908	166	
48. St Mathew	1905	173	
49. The Book of Esther, Job, Proverbs...	n.d.	346	
50. The Epistles to the Ephesians	1909	290	
51. St. Mark	1906	723	cold extraction pH 5.0
52. The Epistles General	1910	369	
53. Ancient and Modern History by Voltaire	1901	334	surface pH 5.2
54. Ledger Paper	1856	663	gelatin (+)

**Table 2. Test Results from Screening of New Papers**

Book or Paper Title	Year	DP	Other Characteristics
1. Citizen newsprint	1989	181	lignin ( + + + ), Al <sup>+3</sup> (-), starch (-), rosin (-)
2. Domtar processed wood pulp	modern	1009	starch ( + ), rosin ( + ), lignin (-)
3. Dalhousie Newsprint	1991	167	
4. Gatineau Newsprint	1991	165	
5. Trois Rivieres (batch #1)	1991	693	no longer produced
6. Trois Rivieres #5	1991	471	lignin ( + + + )
7. Trois Rivieres #7	1991	506	lignin ( + + + ) 26% cellulose
8. Trois Rivieres #8	1991	497	lignin ( + + + ) 33% cellulose
9. Chromatography Paper- Whatman #1	1990	1934	
10. Mohawk off-set	1990	846	gelatin ( + )
11. Arches	1991	> 1500	gelatin ( + + )

**Table 3. Estimation of Degree of Polymerization (DP) for Various Sheets of Paper Selected Randomly from 1927 Volume of *Chums***

Page #	DP
7	318
81	325
133	264
199	291
487	357
561	370
633	313
763	353
815	341

Average  $\bar{x} = 326 \pm 33.8$

**Table 4. Estimation of Degree of Polymerization (DP) for Various Sheets of Paper Selected From Three Sections of the 1920 Volume of *Chums***

Section I	page	17	DP	359	$\bar{x} = 341 \pm 16.4$
		21		329	
		25		333	
Section II	page	170		265	$\bar{x} = 335 \pm 15.1$
		173		349	
		177		336	
Section III	page	311		208	$\bar{x} = 208 \pm 6.5$
		317		214	
		320		194	

**Table 5. Degree of Polymerization (DP) of Naturally-Aged Paper With and Without Applied Printing Ink**

Title of Book & Date	DP	
	Printed	Non-printed
Ancient and Modern History; circa 1901	334	355
A Disquisition on Government and a Discourse on...; circa 1854	478	542

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