Comparing Mass Drying and Sterilization Protocols for Water-Damaged Books

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Overview
Defining the most effective way to dry water-damaged books *en masse* is a problem endemic to all libraries, with the field of book conservation tracing its own origins directly back to the Florence flood. Yet, despite dozens of large-scale disasters and literally thousands of smaller events that have occurred in libraries worldwide since 1966, “best practices” remain unclear, especially for large-scale recovery efforts, because of the limited amount of research conducted on the long-term consequences of various drying and sterilization methods.¹

Recovery specialists need to have a clear sense of the pros and cons of existing treatment options before they can respond effectively. This information is critical to making event-specific decisions so that collection permanence is optimized and distortion minimized within fiscal and operational constraints. Questions to be addressed in reaching those event-specific decisions include: What is the optimal approach to drying water-damaged books given the amount of material affected? What constraints are imposed by the availability and capacity of freezers, electricity, heating, ventilating and cooling (HVAC) systems, labor (trained and untrained), equipment (e.g., book presses, sorbants, fans) and vendors? What regional industrial resources can be called into service (e.g., freeze driers, flash freezers, sub-zero warehouses)? What percentage of the damaged material is rare and which technical options are preferable for material with significant cultural or monetary value? How will a chosen treatment affect paper permanence or the physical cockling of damaged books? When is sterilization justified and what is a responsible treatment option? And if the collection is insured, what constitutes “restoration to usability”?

These judgments must be predicated upon an understanding of the comparative benefits, contraindications, and expenses of the proposed alternatives. The grim reality of flood situations where entire collections are submerged is that all choices are less than optimal, and a recovery can potentially be complicated by sewage-borne contaminants, pathogens, pollutants, and exponentially accelerated microbial growth.

Working with a sense of urgency to address these questions for the population of books most frequently affected by floods, a research project was forged in 2004 that included stakeholders (British Library, National Library of the Czech Republic, and University of Utah Marriott Library), research scientists (British Library, National Library of the Czech Republic, Huntsman Cancer Institute, and Applied Paper Technology, Inc.), and vendors of emergency drying services (Belfor USA, and Artifex Equipment, Inc.). Support for the research was generously provided by the National Center for Preservation Technology & Training.²

The goal of this research project was to define which of five drying and two sterilization techniques caused the least mechanical damage to eighteenth-twentieth century handmade and
machine-made book papers. The enquiry hoped to determine reasons recovery specialists should choose one drying or sterilization technique over another given 1) the age and historical value of the collection; 2) predominant paper types comprising the damaged material; and, 3) institutional or insurer-imposed fiscal constraints. Importantly, the performance of bindings and binding materials was not addressed in this study because books exposed to major flooding typically require rebinding.

**Experimental Design**
The sample set for this study was culled from books published 1767-1979. Multi-volume sets were used in the expectation that the paper would be sufficiently similar from volume-to-volume within each set to provide a reasonable basis for comparison of the methods. While it was recognized at the outset that this choice eliminated the possibility of precisely replicating the study, the experimental design erred on the side of simulating real life situations so the results would correlate in a meaningful way with real library disasters.

A total of 171 volumes (39 three, five, and seven volume sets) were used as samples. Of these, nine sets were published in the eighteenth or early nineteenth century and printed on handmade (cotton/linen) paper, while the remaining 25 sets were published in the nineteenth and twentieth centuries and printed on machine-made (wood fiber) paper. The ratio of handmade to machine-made paper roughly approximated the distribution that might be expected in a mid-sized research library collection, except that sets printed on clay-coated paper were unavailable for destructive testing.

One volume from each test group was retained undamaged as a control, while 22 volumes were wetted and subsequently dried by one of five contemporary drying techniques (air drying, vacuum freeze drying, thermal drying, vacuum packing, and Vacme press drying with Zorbix), or were sterilized after freeze drying with one of two commercially available options (ethylene oxide or gamma irradiation). The experimental design produced 528 data points for each of the seven protocols tested.

**Wetting Protocol**
Each book was submerged completely for 24-hours in distilled water in a flat bottomed sink. While not realistic, distilled water was used because of the difficulty of providing uniformly contaminated flood water at five sites in Europe and the U.S. When buoyancy posed a problem the text was weighted slightly to maximize its submersion and the text’s subsequent wetting. The wet books were then drained under a polyethylene sheet to maintain high relative humidity (approximately 95%) at ambient room temperature for 24 hours (22 C/72°F), simulating a flooded library prior to pack-out. Books to be vacuum freeze dried were frozen (-18 C/0°F) using a commercial freezer facility, while each of the other techniques proceeded directly to drying.

Books prepared for sterilization followed the same wetting procedure but after draining for 24-hours were individually bagged in polyethylene freezer bags and left to mold at ambient room temperature for seven days (168 hours).
Drying Protocols

Following wetting, five drying protocols were tested in five different locations.3

Air Drying with Intermittent Pressing

In a low relative humidity environment (30% RH) at the University of Utah (Salt Lake City, Utah, USA), wet books were stood on end on counter-high tables and fanned open to stimulate evaporation. None of the books were printed on coated stock paper so interleaving with silicon release paper (or wax paper) was unnecessary. Supports to prevent books from falling over included plastic VeloBind4 combs inserted at the head of the text to act as a cross brace, as well as five-pound weights placed as necessary at the base of the boards. Six large electric fans were positioned around the table and turned on the maximum setting to circulate air thoroughly throughout the drying process, accelerating evaporation and discouraging microbial growth. As the still-wet books approached dryness (after approximately three days) they were pressed for approximately 12 hours overnight between boards in a bookbinder’s standing press, and returned to the tables each morning for further fanning and air drying until all were thoroughly dry (taking five to seven days). Dried books were pressed between boards for an additional seven days following drying.

Vacuum Freeze Drying

At BELFOR USA (Fort Worth, Texas, USA) frozen books were placed on rolling wire racks inside a commercial vacuum freeze drying chamber. The books were firmly packed together on the racks, spine down, to help maintain their shape during drying. Vapor pressure within the chamber was reduced below the triple point of water (4.57 torr/0.6092833 kilopascals). A slight amount of heat (40.5 C/105°F),5 was introduced intermittently to the chamber to stimulate sublimation (direct conversion from solid to vapor). Ice from the frozen books sublimed and was captured as ice on the unit’s evaporator coils outside the chamber. The chamber contents were checked daily after the fourth day and thoroughly dry books removed until all books were finished (approximately seven days). Although the vendor provides more expensive services in which books are freeze dried while physically compressed to yield flatter text blocks, this option was not part of this study to reflect financial constraints typical of most post-disaster recoveries.

Thermal Drying

In a commercial wood drying kiln outside Prague (Czech Republic), staff of the National Library of the Czech Republic placed wet books on elevated wire racks, and interleaved each book with sheets of absorbent paper (printed newsprint) every 10-15 pages. In addition to facilitating egress of water from the text block through wicking, these sheets were replaced daily to provide bulk removal of water. Books where then stacked vertically between pairs of unglazed ceramic tiles, and sheets of Holytex6 were placed between the unglazed tile surface and the book cover to promote diffusion of moisture while preventing wet bindings from sticking to the tiles due to adhesive migration or thermoplastic adhesion. Each stack of books was weighted on top (3.2 kg/7.0 lb) to produce constant pressure that reduced text block distortion during drying.
Air was circulated within the closed kiln and the temperature raised to 60°C/140°F with the relative humidity set at 70%. After two days the relative humidity was reduced to 40%-50% while the temperature remained constant. Complete drying time took between seven and twenty days depending on the size and physical characteristics of each text block and cover. Books with plasticized covers took much longer to dry as moisture was eliminated only through the edges of the text. Interleaving was replaced daily during routine inspection for dampness. When the drying cycle was completed, the kiln was slowly returned to ambient conditions to allow books to equilibrate while still under mechanical restraint.

Vacuum Packing
At the British Library (London, United Kingdom) wet books were interleaved with sheets of printed newsprint every 10-15 pages. If the wet binding felt slippery it was wrapped with Bondina to prevent the covering material from adhering to the polyester vacuum pouch (Archipress). Placing the book inside the pouch, the edge was sealed with an Archipress Vacuum Packing Machine and a vacuum pulled. After several days the pouch was opened and the interleaving exchanged for dry blotting paper to remove bulk water, and a new pouch used to re-seal the book. Drying took up to twenty exchanges of bag and blotter over sixty days.

Vacme Press with Zorbix
At Artifex Equipment, Inc. (Penngrove, California, USA) wet books were interleaved with sheets of Zorbix every 10-15 pages. The covers were wrapped with Holytex to prevent binding materials from adhering to the inside of a proprietary, re-sealable vinyl bag integrated with a vacuum hose fitting (Artifex). The vinyl bag’s re-sealable opening was rendered airtight with a Teflon folder while it was evacuated with a vacuum pump (Artifex). Saturated Zorbix interleaving was replaced at 48 hour intervals. Drying required approximately six exchanges of Zorbix over fourteen days.

Sterilization
Following wetting, molding, and subsequent vacuum freeze drying, books were sterilized by licensed practitioners using either ethylene oxide (EtO) or gamma radiation. It should be noted that both sterilization methods required books to be free of liquid water before treatment as any moisture not chemically bound to the cellulose will otherwise react with the sterilizing agent.

Ethylene Oxide
In the Czech National Archives in the Prague (Czech Republic), vacuum freeze dried moldy books were placed in a 6.4 m³ vacuum sterilization chamber (Matachana, type 3.100 LGE-2). The chamber was preheated to 30°C/86°F, air was evacuated to 0.069 bar and a calculated amount of water injected. The water was evaporated at 0.09 bar and the air evacuated a second time to reach 0.054 bar. At 1,125 torr (150 kilopascals) the temperature was held at 30°C/86°F and 80% relative humidity. Books were exposed to a 10% ethylene oxide/90% carbon dioxide mixture (trade name, Etoxen) for 6 hours at 1.5-2.5 bar. The chamber was then aerated, exhausted, and refilled thirty times with the EtO gas incinerated in a plasma flame. At the end of the process books were transferred to a ventilation tunnel where they offgassed for six days.
Gamma Radiation
At Sterigenics, a licensed, commercial gamma irradiation facility in Fort Worth, Texas (USA), vacuum freeze dried moldy books were passed through an irradiation chamber were they received a calculated dosage in the range 12.6-18.8 kilogram (kJ). No pretreatment dehydration or post-treatment equilibration was required.

Analytical Testing
To evaluate treatment-associated loss of mechanical integrity, 24 leaves were removed at equal intervals from each dried or sterilized book and sent to one of two analytical labs. Each leaf was subjected to four internationally standardized mechanical tests: tensile strength, stretch-to-break, tearing resistance; and MIT fold endurance.\textsuperscript{10}

Discussion
Four drying methods (air drying, vacuum freeze drying, vacuum packing, and drying in a Vacme press with Zorbix), and one sterilization technique (ethylene oxide) retained essentially all of the pre-treatment mechanical integrity of the book papers tested and were deemed non-damaging. By contrast, samples from the thermally dried and gamma-irradiated books lost 19% and 24%, respectively, of their mechanical integrity (Fig. 1), and were determined to be inappropriate for treating water-damaged books of permanent retention value.

Physical cockling in treated material was most successfully minimized when book paper was pressed as the moisture was removed. Vacuum packing and Vacme press drying best achieved this ideal but at different levels of fiscal investment. The initial cost of a vacuum packing machine may prove prohibitive for many collecting institutions in addition to which numerous, disposable polyester vacuum pouches were required for each book dried.\textsuperscript{11} The Vacme press, conversely, is so inexpensive that even relatively poor libraries might consider acquiring one as a precautionary measure in the event a limited numbers of rare books ever require drying. Used in conjunction with Zorbix the Vacme press can significantly reduce drying time, but in trials conducted outside of this study the Vacme press also proved effective when readily available newsprint (printed or unprinted) was used as interleaving.

Air drying, which remains the most commonly applied book drying technique due to its low setup cost, produced far better results in terms of physical flatness when semi-dried books were pressed overnight, followed by further air drying the next day. Mold did not form inside the books given the limited duration of this damp pressing, but intermittent exposure to freely circulating air is requisite and, of course, this unrestrained period promotes further page cockling.\textsuperscript{12} A final long pressing as the book reaches its dry state helps reduce this recurring distortion.

Air drying, vacuum packing, and drying in a Vacme press with Zorbix are all labor intensive methods best monitored by trained technicians handling relatively small batches of books (e.g., <100 volumes at a time). Given sufficient freezer capacity to forestall microbial growth, these
three techniques can be applied to several hundred books by simply thawing manageable batches prior to treatment.

Vacuum freeze drying remains the most efficient method for drying large quantities of books (e.g., >500 volumes), especially for books printed on coated stock paper. Unrestrained vacuum freeze drying, however, produced the greatest amount of cockling in the book papers tested. While not tested as part of this study, examples of vacuum freeze dried books pressed during sublimation were examined at the British Library and were shown to produce excellent results. The British Library’s small vacuum freeze drying chamber has been modified with a mechanical jack inside the chamber that is used to manually squeeze books between thin steel plates. The chamber must be opened every two days and the jack’s pressure increased to compensate for decreasing book thickness caused by sublimating ice crystals. While approximately doubling the drying time and significantly increasing labors costs, this approach to vacuum freeze drying produces far better results than books are dried without constraint. Consequently, it is recommended that consumers insist commercial vendors flatten permanent retention collections during the sublimation process and that they be prepared to compensate them for the added time and effort involved.

During the past three decades sterilization of cultural property has largely been downplayed as a technical option. Instead, conservators have learned to rely on environmental stabilization to return mold to a dormant state before removing the desiccated spores with a small vacuum aspirator or vacuum cleaner equipped with a HEPA filter. The literature does a poor job, however, of identifying appropriate technical options when sterilization must seriously be considered. As noted by Fausta Gallo in 1978, sterilization should be reserved for “cases in which arresting infection and infestations is an unarguable necessity.” This can occur, for example, when significant delays in a recovery cause wet collections to mold excessively, or when floodwaters contain contamination such as sewage or other biological hazards. Ignoring sterilization in such circumstances, even when dealing with irreplaceable collections, can pose potential health risks to future users and result in long-term liability issues for recovery specialists and collecting institutions.

Gamma irradiation continues to suggest a promising alternative to ethylene oxide sterilization, but as reported by Butterfield in 1987 and confirmed in the present study, this approach damages book paper to an unacceptable degree. The mechanism by which ionizing gamma radiation kills microbes and renders spores nonviable simultaneously cleaves the cellulosic (and other polysaccharidic) chains from which paper derives its mechanical integrity. Lower levels of damage have been reported in experimental settings where paper was treated with low levels of gamma radiation, but it remains to be demonstrated that significant cellulose degradation can be avoided at doses that yield effective sterilization, or that this approach is commercially viable. Consequently, the authors cannot recommend gamma irradiation over ethylene oxide for sterilizing books of enduring cultural significance under any circumstances we can envision.

The use of ethylene oxide remains controversial in the U.S. Its detractors concede, however, that
loss of lignocellulosic mechanical integrity as a result of ethylene oxide fumigation is not the issue. Objections focus instead on potential latent effects to book components other than paper such as plastics, adhesives, skin-derived materials, and media, as well as possible health risks associated with ethylene oxide off-gassing. The first concern has limited relevance in the context of a flooding incident whose scope and severity are so extreme that sterilization would need to be considered. Bindings in the recovery phase of such an event are quite often discarded, and the authors are unaware of any well-grounded studies reporting replicable findings of significant ethylene oxide-induced alteration of media likely to be encountered in books that would be considered for mass sterilization. The present study corroborates the research of Flieder (1999) and Gallo that found that book paper thoroughly sterilized by a commercial dosage of ethylene oxide remains mechanically undamaged.

An influential study performed at the Library of Congress by Hengemihle, Weberg and Shahani (1995) determined that following sterilization, “off-gassing of ethylene oxide by library materials is a reality.” This finding is often misconstrued to suggest that ethylene oxide is inappropriate for use in conservation, but in fact, the study simply concludes that “fumigated materials should be added to the collections only after the ethylene oxide concentration is decisively under 1 ppm.” This conclusion mirrors U.S. workplace safety standards implemented nearly a decade before (in 1986) that mandate a 24-hour off-gassing period following sterilization to permit ethylene oxide concentrations to fall below 1 ppm. This legal standard is adhered to by licensed U.S. contractors and is readily achieved because EtO is a volatile and reactive gas that dissipates quickly, reacting with atmospheric water molecules to yield more benign species.

Both gamma irradiated and ethylene oxide sterilized books contained a residual and, by consensus, objectionable odor. It is speculated this smell related to decomposing mold spores within the books. Whether this could be mitigated by surface vacuuming, followed aeration or exposure to absorbent media (e.g., activated carbon, potassium permanganate, or baking soda) in a confined space is outside the scope of the present study. Clearly, neither method is ideal. Further research into alternative mass sterilization techniques is desperately needed, including but not limited to an investigation into plasma fumigation.

Conclusion
Relying on surrogates to stand in for complex, real-world book collections, this study compared five drying and two sterilization techniques to determine the long-term affects of these recovery options on the permanence of handmade and machine-made book papers.

Mechanical testing revealed that air drying, vacuum freeze drying, vacuum packing, and drying in a Vacme press with Zorbix had essentially no deleterious affect on handmade and machine-made book papers. Thermal drying, however, was shown to reduce paper’s mechanical strength by 15 percent. Similarly, sterilization with ethylene oxide caused no mechanical damage to moldy, water-damaged book paper while gamma radiation weakened comparable book paper by 25 percent. These findings indicate that thermal drying and gamma irradiation should be avoided when drying or sterilizing water-damaged books of permanent retention value.
Visual observation revealed that handmade papers dry with less distortion than machine-made papers treated by the same method. Books dried and pressed simultaneously (vacuum packed, Vacme press dried with Zorbix, and thermal dried under weight) produce flatter results than books dried without constraint. Thermal drying is therefore deceptive, producing visually flat books that are molecularly damaged. Air dried books can be rendered reasonably flat if they are pressed overnight followed by further air drying the next day. Vacuum freeze drying produced the least flat books in this study but the technique can be modified so that books are pressed during sublimation.

Cost factors for drying can vary considerably depending upon the availability and price of labor, or the initial outlay required for equipment such as a vacuum packing machine. Of the non-damaging techniques tested, air drying and drying with a Vacme press proved the least expensive, while vacuum freeze drying remains the most cost effective approach to drying large numbers of books. Multiple approaches can also be applied to the same recovery so it is reasonable to consider Vacme press drying, careful air drying, and vacuum packing books of enduring cultural significance while less valuable parts of the collection were vacuum freeze dried. As mentioned above, vacuum freeze dried can be modified to press books during sublimation when drying books of permanent retention value. Lastly, freezing wet books remains essential for delaying mold formation and thereby improving the quality of the recovery by allowing the books to be dried in manageable batches.

It is hoped this study’s findings help clarify for disaster responders the implications for permanence of specifying one drying or sterilization technique over another when treating water-damaged books.

Endnotes

2. Investigators for this project included: conservation administrators – Deborah Novotny (Head of Preservation, Collection Care, The British Library); Dr. Jiri Polisensky (Director of Preservation Division, National Library of the Czech Republic), and principal investigator Randy Silverman (Preservation Librarian, Marriott Library, University of Utah); research scientists – Barry Knight (Head of Conservation Research, British Library), Dr. Jiří Neuvirt (Chemist, National Library of the Czech Republic), Hal Erickson (Researcher, University of Utah Health Sciences Center), and Miranda Bliss (Lab Manager, Applied Paper Technology, Inc.); microbiologists – Dr. Jan Francl (Chemist, National Library of the Czech Republic) and Niki Fidopiastis (Market Development Manager, Sterigenics); disaster drying specialists – Kirk Lively (Director of Technical Services, Belfor USA); and, Nicholas Yeager, (President, Artifex Equipment, Inc.), and bookseller Tony Weller (Owner, Sam Weller's Zion Bookstore). Special thanks for creative input go to Olivia Primanis (Senior Conservator, Harry Ransom Humanities Research Center, University of Texas at Austin).

3 While vacuum freeze drying and thermal drying were part of this study, these techniques must not be confused with similarly-named with thermal vacuum drying (TVD), in which bulk liquid water is boiled away at the intermediately-elevated temperatures permitted by a vacuum chamber, or thermal vacuum freeze drying, (TVFD) in which a proprietary method of thermally-aided flattening is applied to vacuum freeze dried materials. TVD and TVFD were not considered for testing due to significant limitations but have been summarized in the work by Hilary A. Kaplan and Kathleen A. Ludwig, cited above.

4. VeloBind combs, developed by the General Binding Corporation (now merged with ACCO) are inserted into perforated leaf edges in a proprietary quick binding method. The combs – various called “spines” or “hot knife strips” – are available from office supply companies specializing in quick binding equipment.

5. Temperature in vacuum freeze drying is critical. Heat is required to provide the energy for sublimation, but increasing the drying temperature causes cellulose damage. Some vendors refer to their product as “vacuum freeze drying,” but introduce an excess of heat (TVD), e.g., 54°C (130°F), to accelerate the sublimation. While faster turnaround of treatment batches potentially increases profit for the service provider, it results in permanent damage to paper.
6. 100% polyester nonwoven polyolefin sheet.

7. Bondina is a proprietary non-woven polyester material with a smooth surface that facilitates release.


9. The Vacme Press and Zorbix are available exclusively from: Artifex Equipment, Inc., 9595 Main St., #1, P.O. Box 319, Penngrove, CA 94951 USA; cel. (707) 331-0237; artifex@pipeline.com; http://www.artifexequipment.com. Designed by Artifex Equipment, Inc. and the United States Department of Agriculture’s National Agricultural Library, Zorbix is a super absorbent polymer embedded in a sheet of blotting paper able to absorb 50 times its weight in water.

10. Paper samples were tested by or under the supervision of two of the authors (Miranda Bliss and Barry Knight) at their respective institutions. Tests included: tensile strength and stretch-to-break (ISO 1924-2) (machine direction and cross-machine direction: maximum load, strain %, and tensile energy absorption); tearing resistance (ISO 1974) (machine direction and cross-machine direction, mN/ply); and MIT folding endurance (ISO 5626) (machine direction and cross-machine direction; note that # of double-folds (#DF) is deprecated in favor of $\log_{10}$#DF).

11. Conservation By Design Limited is the sole manufacturer of the Archipress Vacuum Packing Machine and Polyester Vacuum Pouches. Three models available sell for £2652.00 ($5,331.83); £4604.00 ($9,256.32); and £6528.00 ($13,124.51); retrieved from the World Wide Web 6 July 2007: http://www.conservation-by-design.co.uk/equipment/archipress4.html. Dissatisfaction has been reported by some who used a vacuum packing machine following the 2004 floods in the Czech Republic because of damage caused to fragile books by the significant pressure applied by the pouch, and mold that formed inside of incompletely sealed pouches.

12. Pressing can only be applied to semi-dry books printed on uncoated paper. Books wholly or partially printed on coated stock must have every coated leaf separated with silicon release or waxed paper interleaving and allowed to thoroughly dry prior to pressing. Cockling is therefore maximized by air drying books of this type and alternative drying methods are suggested.


23 Plasma fumigation relies on radio waves. Wet, moldy books are placed inside a vacuum chamber, a vacuum is pulled, and the chamber is backfilled with an inert gas. Radio waves directed within the chamber convert the inert gas to argon, nitrogen and helium and draw hydrogen molecules from all water within the vacuum. Creating plasma energy in this way damages the DNA of mold, destroying is viability. More on the application of plasma fumigation to sterilization of library material can be found at Midwest Freeze Dry Ltd. (7326 N. Central Park, Skokie, IL. 60076) http://www.midwestfreezedry.com/pestinfestation.html