J Wood Sci (2007) 53:121–126
DOI 10.1007/s10086-006-0830-3

ORIGINAL ARTICLE

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Modified atmosphere and humidity packages for conservation of paper antiques

Received: January 4, 2006 / Accepted: July 19, 2006 / Published online: October 4, 2006

Abstract Modified atmosphere and humidity (MAH) agents were developed to manipulate the gas composition and humidity for controlling the foxing of paper materials during storage. Sodium ascorbate, sodium carbonate decahydrate, ferrous sulfate heptahydrate, and silica gel were selected as the basic ingredients to formulate the MAH agents that could effectively remove oxygen, release carbon dioxide, and control the relative humidity (RH). With all the MAH agents developed in our study, RH was reduced and maintained without decreasing the MAH efficiency. To evaluate the inhibition of microorganisms on antique paper by MAH agents, the properties of Wikstroemia papers were measured after accelerated aging and inoculation with *Aspergillus flavus* and *Penicillium citrinum*. Under high (90%) or low (50%) RH conditions, as long as MAH agents were used, even after 80 days, the color difference value of Wikstroemia paper was kept below 1.5 and foxing was not found. Paper packaged without MAH agents and under RH as high as 90% showed a substantial color difference in 60 days. Snowflake-like foxing was also found by ultraviolet light inspection. Wikstroemia paper inoculated with *P. citrinum* without an MAH agent showed a significant color difference in 80 days.

Key words Modified atmosphere and humidity (MAH) package · Foxing · Color difference · Conservation · Paper antiques

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Introduction

The deterioration of paper constituting books and archival materials because of the degradation of its cellulose component has been recognized since the turn of the twentieth century.[1–3] The principal changes in the structure of cellulose involve biodegradation, photodegradation, acid hydrolysis, and oxidation.[4–6] In general, the degradation of paper antiques is caused by a combination of the effects of air (especially O_2), relative humidity (RH), light, insects, and microorganisms.[7–11] To determine the effectiveness of conserving paper antiques, chemical insecticides and fungicides have been used in some museums to obtain an instant improvement in the storage environment and to prevent molding. However, the chemicals in insecticides and fungicides are likely to damage paper antiques, and are not environmentally friendly.[12]

In recent years, there has been widespread investigation into the use of low-O_2 methods to control insects and microorganisms,[13,14] microwave methods[15] for eradicating microorganisms, and methods that alter the air composition in a closed chamber. For methods involving a low O_2 concentration, the use of vacuum, O_2 absorbers, and N_2 and CO_2 gas infusion can be applied separately or combined for the packaging of paper antiques.[16]

For microwave methods, the longer the treatment time for paper antiques, the greater was the effect of microorganism eradication; however, the properties of the paper were degraded simultaneously.[15] Moreover, there is risk of burning paper antiques during microwave treatment. The use of a replacement gas (e.g. N_2) or vacuum packaging can reduce the problem of the oxidation of paper antiques. However, 2%–3% O_2 still remains after mechanical infusion of N_2 in the package, which will still cause oxidation, foxing, molding, and color changes in paper antiques,[17] thus degrading the quality of paper antiques and shortening their storage life. Brittle paper antiques are likely to be damaged by vacuum packaging methods. Once the new technology involving modified atmosphere (MA) agents is fully developed,[18] the problem of the remaining O_2 is able to be solved.

Antiques of different materials require different storage humidities. However, if a humidity controller is added to decrease the RH, the efficiency of MA agents may also be affected.[18]

The purpose of this study was to develop modified atmosphere and humidity (MAH) agents with better gas composition manipulation and humidity control for controlling foxing of paper materials during their storage. Wikstroemia paper was selected for our study and was inoculated with *Aspergillus flavus* or *Penicillium citrinum* after accelerated aging. The effect of microorganism inhibition using MAH agents in the packaging storage of paper antiques was investigated.

Materials and methods

Antique paper

In a practical sense, any kind of suitably fibrous material can be made into paper. Since the eleventh century, Chinese artists have been fond of using hand-made paper made from phloem fibers, especially in Hsuan county, and thus the traditional name of Hsuan paper emerged. One of the most famous Hsuan papers is made from *Wikstroemia canescens*, which typically possesses slender fibers about 4.0 mm long and 10 μm wide with a low average lignin content of about 10%; these properties contribute to its extraordinary characteristics in papermaking. Wikstroemia paper was selected as the antique paper for this study because of its popularity for use in traditional Chinese paintings and calligraphy.

Microorganisms

Aspergillus flavus Link (CCRC no. 32113) and *Penicillium citrinum* Thom (CCRC no. 32382) are very common fungi[19–21] and they were selected for biodeterioration experiments on Wikstroemia paper.

Chemicals

For the preparation of MAH agents, the following chemicals were used: sodium ascorbate (SA) ($NaC_6H_7O_6$, Sigma); ferrous sulfate heptahydrate (FS) ($FeSO_4$ $7H_2O$, Riedel-de Haen); sodium carbonate decahydrate (SC) (Na_2CO_3 $10H_2O$, Riedel-de Haen); and silica gel (Riedel-de Haen).

Preparation of MAH agents

MA agents with two different modes of action were prepared. One agent to control low O_2 and high CO_2 concentrations was adopted from the food science field,[18] with a composition of 1.3 g of SA, 2.6 g of SC, and 2.2 g of FS in a 285-ml flask. Another type of agent, used to control low O_2 concentrations, was tested in a preliminary study and was finally prepared with a composition of 0.8 g of SA and 3.0 g of SC. The gas composition in each test was analyzed for both systems by gas chromatography (GC) (GC-320, Gasukuro Kogyo) after 5 days.

In addition to the two major types of MA agent, silica gel with a particle diameter of 2–3 mm instead of 1–3 mm[18] was added as a humidity controller in a gas-proved low-density polyethylene (LDPE) package measuring 85 × 60 mm. The concentrations of O_2 and CO_2 for each different type of packaging were monitored using Gaspace 2-V3.3 software (Systech Instruments, Thame, Oxfordshire, UK) during 2, 4, and 7 days of storage, with air calibration.

SC was first ground and prepared in powder form (60–100 mesh). FS was also in powder form (140–270 mesh) at 5°C. Silica gel was sieved to 2–3 mm and was kept dried. SC and FS were mixed evenly in a fully N_2-infused LDPE package (85 × 60 mm) at 25°C and placed into a 285-ml flask with 1–4 g of silica gel. The gas composition and RH were analyzed every 12 h using GC and a hygrometer (Hygro Estern M-4), respectively.

Effects of MAH packages on foxing

To determine the microorganism inhibition efficiency of MAH agents, Wikstroemia paper was inoculated with *A. flavus* or *P. citrinum* after accelerated aging under conditions of 85°C and 65% RH for 264 h.[22]

Aspergillus flavus and *P. citrinum* were cultured on potato dextrose agar (PDA) at 25°C. When the spores had spread over 90% of the plate, they were washed off and collected using sterilized water, and were used to prepare a suspension of 10^6–10^7 spores/ml, which was checked using a hemocytometer. Then 1 ml of suspension was inoculated onto the center of each aged paper sample for test. Specimens were placed in darkness at 90% or 50% RH for 1 day for preconditioning. Then the inoculated Wikstroemia paper samples were stored in darkness with MA or MAH agents at 25°C. Samples were collected at 20-day intervals for measurement of color difference (ΔE^*); foxing appearance was checked under ultraviolet (UV) light.

Color difference, ΔE^*

Color measurements were conducted using a color difference meter (Dr Lange, LMG082). Tristumulus values X, Y, and Z for all specimens were obtained directly from the colorimeter. The recommended Commission International de l'Eclairage (CIE) color parameters L^* (lightness), a^* (along the X-axis, red–green), and b^* (along the Y axis, yellow–blue) were then computed to calculate the color difference (ΔE^*) based on the following formulae: $\Delta L^* = L_i^* - L_0^*$; $\Delta a^* = a_i^* - a_0^*$; $\Delta b^* = b_i^* - b_0^*$; $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$; where the subscripts 0 and i indicate the values obtained from inoculated paper packaged without MAH (control group) and with MAH agents, respectively.

The yellowness index (YI) and brightness retention rate (BR%) of the specimens were calculated with the following formulae: $YI = 100(1.28X - 1.06Z)/Y$; $BR\% = Y_i/Y_0$; where Y_i and Y_0 are the yellowness index of inoculated paper

packaged with MAH agents and without MAH (control group), respectively.

Foxing inspection of Wikstroemia paper

A*spergillus flavus* and *P. citrinum* are known as paper-foxing microorganisms, which cause a change in color and damage to paper materials. Foxing on Wikstroemia paper was investigated both with the naked eye and under UV light of 365 nm (Spectroline ENF-2400). A fluorescent response indicated the existence of foxing caused by microorganisms.

Results and discussion

Efficiency of MA agents

MA agents used to control O_2 to low concentration and CO_2 to high concentration were developed to give a final CO_2 release ratio of 21.56% (w/w) and O_2 release ratio of 0.72%, with RH of 88.1% ± 1.9%. The MA agent for control of low O_2 concentrations was developed to decrease the O_2 level to 1.39%, with RH of 84.3% ± 2.0% over a period of 4 days.

Effect of silica gel on efficiency of the MA agent

MAH agents combined with 0.8 g SA and 3.0 g SC as an O_2 absorber and silica gel of 2–3 mm diameter (1, 2, 3, or 4 g) were used for humidity control. As shown in Fig. 1, when silica gel was added to a low-O_2 MA, the O_2 concentration decreased from 21% to less than 3.5% in the first 48 h; after 84 h, the O_2 concentration was controlled at 1.0% ± 0.5%.

As silica gel (1–4 g) was added, the O_2 consumption rate was affected only in the early period. It was reasonable to assume that silica gel competed in the early stage with SA to react with the $10H_2O$ in SC, which delayed the dissociation of SA into Na^+ and ascorbate ions and thus delayed O_2 consumption. After the H_2O of SC was released and an alkaline environment developed after 48 h, SA oxidation was then accelerated to reach a stable O_2 concentration in 96 h. For reaction time longer than 96 h, the use of silica gel considerably enhanced the decrease in O_2 level (<1.0% ± 0.5%).

The agent composed of 1.3 g SA, 2.6 g SC, 2.2 g FS, and 0–4 g silica gel (2–3 mm) was used to reduce O_2 and release CO_2 with RH control. The CO_2 release rate is plotted in Fig. 2, showing hindrance of CO_2 release over 24 h with increasing amounts of silica gel. However, the amount of silica gel added is irrelevant to the CO_2 release rate of the MA agent developed after 48 h. After adding 1–4 g of silica gel (2–3 mm), the MA agent consumed O_2 and decreased its concentration from 21% to less than 1.0% ± 0.5% after 48 h (Fig. 3). The seven binding water molecules of FS and ten binding water molecules of SC, which provided the H_2O sources to react with SA functioning as a low-O_2/high-CO_2 agent, were used in this study. FS dissociates into Fe^{2+} and SO_4^{2-} in water and releases H^+ in the oxidation process, forming CO_2 gas after reacting with SC.

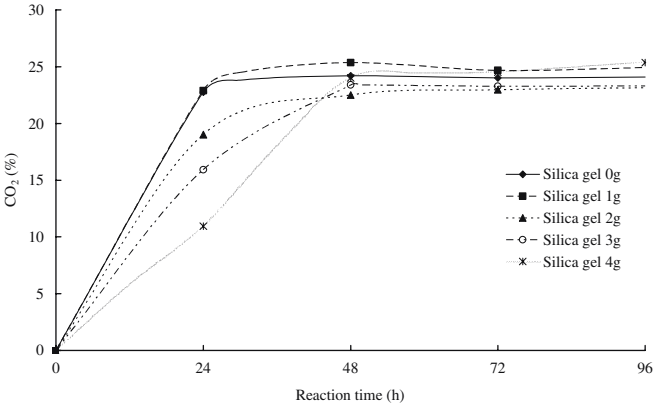

Fig. 2. Effect of silica gel on the CO_2 release rate of the MA agent in a 285-ml flask at 25°C [SA, 1.3 g; SC, 2.6 g; ferrous sulfate heptahydrate (FS), 2.2 g]

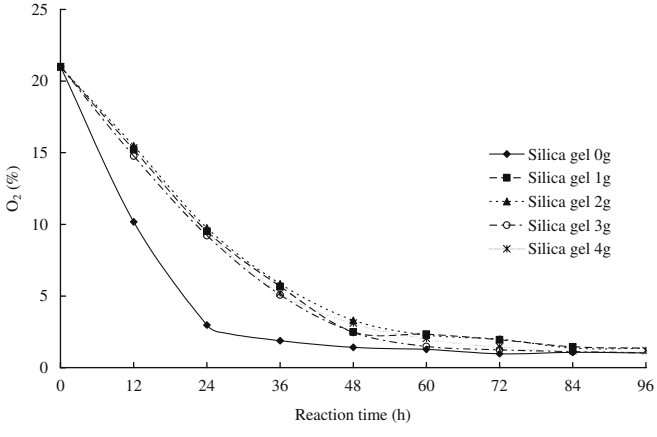

Fig. 1. Effect of silica gel on the O_2 consumption rate of the modified atmosphere (MA) agent in a 285-ml flask at 25°C [sodium ascorbate (SA), 0.8 g; sodium carbonate decahydrate (SC), 3.0 g]

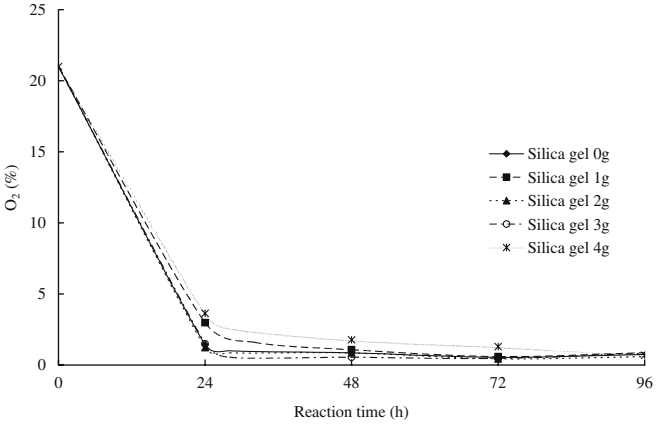

Fig. 3. Effect of silica gel on the O_2 consumption rate of the MA agent in a 285-ml flask at 25°C (SA, 1.3 g; SC, 2.6 g; FS, 2.2 g)

The effects of silica gel (0–4 g) on RH control in a 285-ml flask after 5 days with MA agents are shown in Fig. 4. The RH decreased with increasing amounts of silica gel in packages with either a low-O_2 or low-O_2/high-CO_2 agent. Figures 1–3 show that the O_2 consumption rate decreased in the early stage when silica gel was used; the agents producing low O_2 and high CO_2 were also affected in the CO_2 release in the initial stage. However, as shown in Fig. 4, when silica gel was added, the aim of decreasing the RH was achieved in 5 days. After 48 h, the formation of CO_2 was still higher than 20% (Fig. 2), with the O_2 level below 1.0% ± 0.5% (Fig. 3).

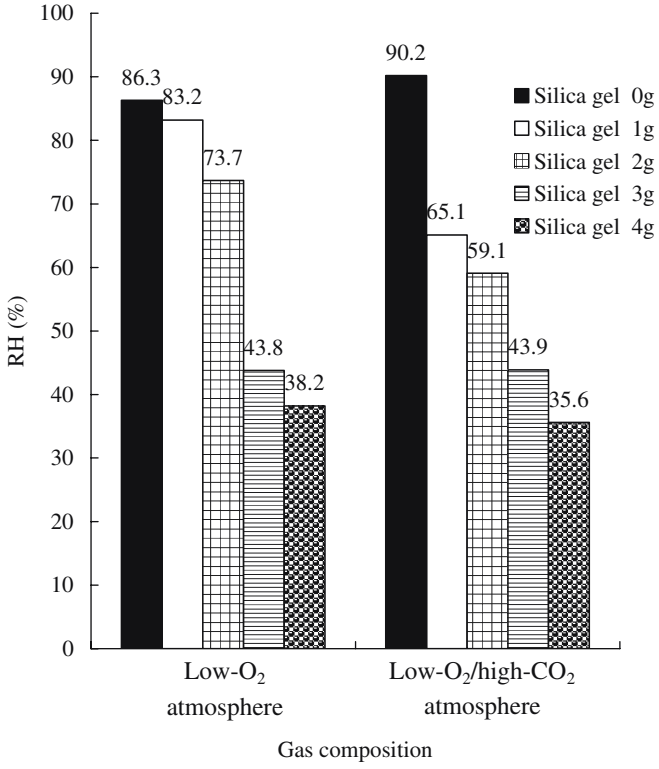

Fig. 4. Effect of silica gel on relative humidity (*RH*) with MA agent in a 285-ml flask after 5 days

From the point of view of O_2 consumption, Figs. 1 and 3 show that the rate for the low-O_2/high-CO_2 MA agent was higher than that for the low-O_2 MA agent. Combined with silica gel, the low-O_2/high-CO_2 MA agent showed a better effect than that of the low-O_2 MA agent when the amount of silica gel used was 1–2 g. When this was increased to 3 g, both MA agents gave promising RH control, at 44% RH, which is a very good RH environment for paper antiques.[23]

Effects of MAH agents on prevention of foxing

Changes in surface color

The L^* value represents the lightness of test material, and the ΔL^* value is the difference in L^* before and after accelerated aging. When the ΔL^* value is positive (+), this indicates higher lightness after accelerated aging; when the ΔL^* value is negative (–), this indicates lower lightness after accelerated aging. As shown in Table 1, the ΔL^* value of Wikstroemia paper inoculated with *Penicillium citrinum* decreased under storage at 25°C and 90% RH under normal atmospheric conditions after 60 days. After 80 days, the ΔL^* value was even lower. On the other hand, paper inoculated with *Aspergillus flavus* and stored at 25°C and 50% RH showed no significant difference in the ΔL^* value between day 60 and day 80.

Under normal atmospheric conditions, paper inoculated with either fungus stored at 90% RH gave significantly darker results, i.e. ΔL^* values below –1.0. With MA agents, the decrease in lightness was greater for inoculated Wikstroemia paper in a low-O_2 atmosphere than for that in a low-O_2/high-CO_2 atmosphere at 90% or 50% RH. The ΔL^* values of inoculated paper under reagent-controlled conditions exhibited only very slight color differences.

The a^* parameter represents color difference on the red–green axis. The Δa^* value is the difference in a^* before and after aging. A positive (+) Δa^* value means that the color of the paper has shifted toward red and a negative (–) Δa^* value means the color has shifted to green. Comparison of the

Table 1. Effect of different gas compositions and accelerated aging conditions on ΔL^*, Δa^*, and Δb^* for Wikstroemia paper inoculated with *Penicillium citrinum* or *Aspergillus flavus*

Gas composition	Accelerated aging conditions	ΔL^*		Δa^*		Δb^*	
		P. citrinum	*A. flavus*	*P. citrinum*	*A. flavus*	*P. citrinum*	*A. flavus*
Low-O_2 atmosphere	I	–0.7	0.3	–0.6	–0.5	0.5	0.0
	II	–0.6	0.0	–0.6	0.2	0.2	–0.2
	III	–0.6	0.1	–0.8	–0.4	0.4	0.0
	IV	–0.7	–0.1	–1.0	–0.1	0.5	–0.6
Low-O_2/high-CO_2 atmosphere	I	–0.2	0.2	–0.8	–0.7	0.6	0.4
	II	–0.2	0.9	–1.1	–0.4	0.5	0.2
	III	0.2	0.5	–0.9	–0.9	0.9	0.2
	IV	0.0	0.0	–1.1	–0.7	0.8	0.1
Normal atmosphere	I	–1.6	–1.3	–1.3	0.2	0.4	0.3
	II	–3.5	–1.6	0.6	0.6	0.8	1.4
	III	–1.2	–0.5	–0.6	0.2	0.1	–0.4
	IV	–0.7	0.2	–0.2	–0.6	0.2	–0.4

I, 90% relative humidity (RH) 60 days; II, 90% RH 80 days; III, 50% RH 60 days; IV, 50% RH 80 days

Table 2. Effect of different gas compositions and accelerated aging conditions on yellowness index (YI), brightness retention (BR), and ΔE^* for Wikstroemia paper inoculated with *Penicillium citrinum* and *Aspergillus flavus*

Gas composition	Accelerated aging conditions	YI		BR (%)		ΔE^*	
		P. citrinum	*A. flavus*	*P. citrinum*	*A. flavus*	*P. citrinum*	*A. flavus*
Control[a]	–	30.4	32.4	100.0	100.0	–	–
Low-O_2 atmosphere	I	31.9	32.0	98.0	100.7	1.5	0.8
	II	31.3	32.2	98.2	100.1	1.1	0.7
	III	31.5	32.0	98.2	100.3	1.2	0.7
	IV	31.4	31.2	97.9	99.8	1.5	1.0
Low-O_2/ high-CO_2 atmosphere	I	31.7	32.5	99.3	100.5	1.2	0.9
	II	31.4	32.2	99.4	102.8	1.3	1.1
	III	32.0	31.8	100.6	101.6	1.5	1.1
	IV	31.7	31.9	100.0	100.0	1.5	1.1
Normal atmosphere	I	31.3	33.4	95.5	96.2	2.2	1.6
	II	33.9	35.7	90.0	95.5	3.9	2.7
	III	31.3	32.0	96.7	98.6	0.9	0.8
	IV	31.6	31.2	98.0	100.7	1.5	1.8

[a] The control was measured directly after accelerated aging, with 0 days of storage

effects of gas compositions on Δa^* value for Wikstroemia paper inoculated with either *P. citrinum* or *A. flavus* and stored under reagent-controlled conditions showed a slightly greater tendency to shift to green (Table 1).

The b^* parameter represents color difference on the yellow–blue axis. Δb^* is the difference in b^* before and after aging. A positive (+) Δb^* value means that the color of the paper has shifted toward yellow and a negative (–) Δb^* value means the color has shifted to blue. As shown in Table 1, paper inoculated with *P. citrinum* exhibited a greater tendency to shift toward yellow than paper inoculated with *A. flavus*. Paper stored under normal atmospheric conditions without reagents exhibited a significant difference between high RH conditions and low RH conditions inoculated with either *P. citrinum* or *A. flavus*. Inoculated paper preserved with reagent showed variation from the above tendency.

YI represents the yellowness index. The higher the YI value, the yellower the paper. Table 2 demonstrates that inoculated paper stored at 90% RH showed substantially increased YI values after 80 days, with the YI value of paper inoculated with *A. flavus* reaching 35.7 and that with *P. citrinum* reaching 33.9. YI values for inoculated paper stored in a reagent-controlled environment or at low RH without agent (i.e., 50% RH only) showed only slight differences between 60 and 80 days.

BR% represents the brightness retention. For Wikstroemia paper inoculated with *P. citrinum* stored at 90% RH without MA or MAH agents, BR% decreased to 95.5% after 60 days and to 90.0% after 80 days. For paper inoculated with *A. flavus* and stored under the same conditions, BR% decreased to 96.2% and to 95.5%, respectively. *Aspergillus flavus* showed a less harmful effect than *P. citrinum* in terms of BR% with or without reagent control in this study. Paper preserved with an MA package and stored at 90% or 50% RH for 60 or 80 days showed only a slight decrease or no decrease in BR%.

A ΔE^* value of 1.5–3.0 is defined as noticeable, and 3.0–6.0 is defined as an appreciable difference.[24] Paper inoculated with *P. citrinum* and stored at 90% RH without an MA package showed a noticeable ΔE^* value (2.2) after 60 days and an appreciable ΔE^* value (3.9) after 80 days. Paper inoculated with *A. flavus* and stored under the same conditions showed a noticeable ΔE^* value (1.6) after 60 days and a noticeable ΔE^* value (2.7) after 80 days.

For paper preserved with an MA package and stored either in a low-O_2 or low-O_2/high-CO_2 atmosphere at either 90% or 50% RH for 60 or 80 days, ΔE^* values were all 1.5 or less. According to statistical analysis, significant differences ($P < 0.05$) did not exist for the color difference between paper specimens preserved with an MA package.

Appearance of foxing

Arai[25] indicated that there is a close relationship between foxing formation and fungi. *Aspergillus flavus* and *P. citrinum* have been reported to cause foxing and changes in color on papers.[24] Cain and Miller[26] have observed the nature of foxing by photographic and chromatographic techniques. Our results show that there were no obvious foxing spots observed with the naked eye. However, foxing fluorescence spots appeared on paper under a normal atmosphere at 90% RH.

Paper preserved with an MA agent and stored under either a low-O_2 or low-O_2/high-CO_2 atmosphere showed no obvious foxing fluorescence for either 90% or 50% RH. With an MAH agent at 50% RH, even under a normal atmosphere, no presence of foxing on paper was found by the naked eye or under UV light.

Conclusions

Based on the experimental work conducted in this study, the following conclusions can be drawn. The MAH agent composed of 0.8 g of SA, 3.0 g of SC, and 0–4 g of silica gel was effective for reducing O_2 levels and controlling RH to

86.3%–38.2%. With this reagent combination, the O_2 concentration was reduced and controlled to a stable 1.0% ± 0.5% and the CO_2 concentration was constantly below 0.5%. On the other hand, the MAH agent composed of 1.3 g of SA, 2.6 g of SB, 2.2 g of FS, and 0–4 g of silica gel was effective in reducing O_2 and releasing CO_2, with RH controlled in the range 90.2%–35.6%. With all the MAH agents developed in our study, the RH was reduced and maintained without reducing the efficiency of the MAH agents.

To evaluate the inhibition of microorganisms on antique paper by MAH agents, Wikstroemia papers were inoculated with *Aspergillus flavus* and *Penicillium citrinum* after accelerated aging. The visual appearance of the papers was then measured after conservation with MAH agents over time. As long as MAH agents were used, under conditions of 90% or 50% RH and after 60 or 80 days, ΔE^* values for Wikstroemia paper were kept below 1.5. Wikstroemia paper packaged without MAH agents and at 90% RH showed a substantial color difference in 60 days. Wikstroemia paper inoculated with *P. citrinum* showed a significant color difference in 80 days. Under UV light inspection, snowflake-like foxing was found on Wikstroemia paper packaged without MAH agents and at 90% RH.

Acknowledgments The authors thank Professor Dr. Shang-Tzen Chang, School of Forestry and Resource Conservation, National Taiwan University, for his assistance with experiments.

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The publication of this article was made possible by an Emachu Research Fund. The authors are grateful for the fund.