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In collaboration with:
Development of Automatic Fumigation System for Archive's Stack Rooms, and Development of Customized Natural Fumigation Agent

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* End user: National Archives of Korea (NAK)
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* Research period: 2008. 5. 1 - 2008. 11. 30

Summary
- Title of Project: Development of Automatic Fumigation System for Archives Stack Rooms and development of customized natural fumigation agent
- Keywords: Preservation archives stack rooms, Records preservation, Archives stack rooms fumigation, Unmanned fumigation systems, Biological environment, natural herb based fumigation agent

I. Purpose of Research
- Development of customized natural fumigation agent
- Safe preservation of the archives by suppressing the growth and the propagation of the Microorganism
- Maintenance of the best condition to prevent the biological damages to the important Archives
- Development of the customized automatic unmanned sterilization system which doesn't require the visit to the library
- Construction of the guidelines for the biological environment management to preserve the important archives

II. Results of Research
- Selection for the customized natural fumigation agent (ArchiPerplus) for the biological protection of the archives
- Confirmation of the safety of ArchiPerplus to the archives
- Confirmed that the natural fumigation agent makes no effect to the carbon filter of air-conditioner
- Confirmed that there is no O₃ generation from the use of ArchiPerplus because the propellant of mass sterilization system is the compressed air.
- Confirmation of the safety of ArchiPerplus for the metal corrosion by corrosion tests for the 7 kinds of metals
- Confirmed that the fire detector didn't react to the injection more than 400 times of normal usage of the disinfectant.
- Biological damage can be prevented if the density of TVOC is kept more than 800ppb for 30 minutes in case of one time injection per day
III. Expected Impacts
- Construction of the best biological condition for the archives and the cost reduction for the sterilization
- Ensuring the security by the unnecessary of the outsider's visit for the sterilization
- Applying to the mass and unmanned sterilization for the important archives

I. Purpose of Research

1. Final target of the research
- Development of customized natural fumigation agent
- Safe preservation of the archives by suppressing the growth and the propagation of the Microorganism
- Maintenance of the best condition to prevent the biological damages to the important Archives
- Development of the customized automatic unmanned sterilization system which doesn't require the visit to the library
- Construction of the guidelines for the biological environment management to preserve the important archives

2. Application of research results
- Construction of optimum biological environment of archives stack room, and save fumigation disinfection cost
- Acquire security from not allowing human entering into the stack room for Fumigation work.
- Apply for mass & unmanned fumigation for important archives’ stack room

II. Method of Study and Research Results

1. Biological environment monitoring at the Nara-Repository of NAK
Among the micro-organisms, mostly, fungi cause biological deterioration of stack room, archives. Fungi (100,000 over kinds are known) take nutrient from organic materials such as paper, dust, adhesives, leather, fiber, starch etc. Fungi reproduce with spore which attach to insects or animals or via air to move to another places. Therefore, fungi can exist at everywhere in our environment. Under suitable temperature and humidity, spore germinate to start a life of fungus.

Mostly Archives have been disinfected with chemical method until 1980s, and Methyl bromide (MB) & Ethylene oxide(EO) are commonly applied. MB is known that has Insecticidal effect but has not disinfection effect, make nasty smell after fumigation, weaken adhesives of documents. MB is now limitedly used for fumigation because of strong toxicity.

This study is to resolve the problems of existing MB/EO, and develop new optimum natural fumigation agent.
2. Analysis of Biological environment monitoring at the Nara-Repository of NAK

a. Place for monitoring
   (1) Place: Nara-Repository of NAK
   (2) Date: 2008. July 30
   (3) Stack Room: NB(106), NB(108), NF(306), NF(308), NF(706), NF(709)

b. Survey equipment and method
   (1) Equipment: Media (PDA, NA), Air Sampler (MAS-100, Germany)
   (2) Sampling airborne microorganisms in stack rooms

Take air samples using ready prepared media (PDA, NA) and Air sampler (MAS 100, Germany). Air filtering speed is 100L/min, absorbing speed (touching speed of micro-organism to media) is 11m/sec for 10 minute sampling. Media (PDA, NA) is cultivated under 27°C, 72 hours. For bacteria cultivation, 37°C, 24 hours in incubator, and find the status of micro-organism colony (CFU), and analyze morphological characteristics of harmful micro-organism. (Fig. 1).

![Sampling of airborne microorganisms in stack rooms](image)

Fig.1. Sampling of airborne microorganisms in stack rooms

c. Analysis result of airborne microorganism distribution in stack rooms

After sampling and cultivation, apply the cultivated micro-organism to PDA, and cultivate for 7 days under 25±1°C, and examine with light microscope (AX10, Carl Zeiss) and contact microscope (IcamScope, somtech). Prepare cultivated microorganism fragment at 5mm×5mm size, put into 2.5% glutaraldehyde liquid for 2 hours, and wash with 5.5M cacodylate buffer, and put into 1% osmium tetroxide for 1 hour. After this procedure, dewatered with alcohol series, and dry with Critical point dryer HCP-2, Hitachi, and examine with Scanning electron microscope, S-3000N, Hitachi at 15kV.
(1) NB (106) Stack Room

Fungi (14cfu/m³)  Bacteria (73cfu/m³)

(2) NB (108) Stack Room

Fungi (37cfu/m³)  Bacteria (66cfu/m³)

(3) NF (306) Stack Room

Fungi (13cfu/m³)  Bacteria (31cfu/m³)
(4) NF (308) Stack Room

Fungi (16cfu/m³)  Bacteria (34cfu/m³)

(5) NF (706) Stack Room

Fungi (30cfu/m³)  Bacteria (6cfu/m³)

(6) NF (709) Stack Room

Fungi (3cfu/m³)  Bacteria (72cfu/m³)
d. Separated micro-organism and behavior in stack rooms

The analysis result of sampled airborne microorganism confirms 15 kinds of fungi and 9 kinds of bacteries. Separated micro-organism are:
Aspergillus niger, Aspergillus sp., Alternaria sp., Bacillus sp., E. coli, Fusarium sp., Penicillium sp., Rhizopus sp., Staphylococcus sp., Tricoderma sp., etc.

and Aspergillus, Penicillium series is major (Fig. 8).

Fig. 8 Morphological characteristics of the fungi isolated from stack rooms.

3. Develop natural fumigation agent to protect archives and stack rooms from biological damages

a. Examine natural agents and selection

1). Testing materials

Take existing natural agent and customized natural agent, and perform antibacterial activity test against microorganisms from stack rooms at PDA(Potatoes, Infusion from: 200g, Dextrose: 20g, agar: 15g/ L pH 4.5), LA(Tryptone: 10g, Yeast Extract 5g, Sodium chloride: 10g, agar: 15g/ L pH 7.0).

2). Measure antibacterial effect against bacteria, and measure Antifungal activity against fungi

b. Select optimum natural fumigation agent

1). Compare antibacterial/ Antifungal effect of existing natural fumigation agent and customized natural fumigation agent.

Test with separated microorganism from stack room [Aspergillus niger, Aspergillus sp., Alternaria sp., Bacillus sp., E. coli, Fusarium sp., Penicillium sp., Rhizopus sp., Staphylococcus sp., Tricoderma sp., etc.], and measure antibacterial activities. The results is as <Table 2>. ArchiPer plus is selected as best antibacterial activity agent.
Table 2 Comparing antimicrobial activity of isolated microorganisms

<table>
<thead>
<tr>
<th>Antimicrobials Test microorganisms</th>
<th>Inhibition zone diameter (㎜)*</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Customized Agent</td>
</tr>
<tr>
<td>Aspergillus niger</td>
<td>42</td>
</tr>
<tr>
<td>Aspergillus sp.</td>
<td>40</td>
</tr>
<tr>
<td>Alternaria sp.</td>
<td>46</td>
</tr>
<tr>
<td>Fusarium sp.</td>
<td>40</td>
</tr>
<tr>
<td>Penicillium sp.</td>
<td>38</td>
</tr>
<tr>
<td>Rhizopus sp.</td>
<td>48</td>
</tr>
<tr>
<td>Tricoderma sp.</td>
<td>50</td>
</tr>
<tr>
<td>Bacillus sp.</td>
<td>68</td>
</tr>
<tr>
<td>E. coli</td>
<td>70</td>
</tr>
<tr>
<td>Staphylococcus sp.</td>
<td>66</td>
</tr>
</tbody>
</table>

**c. Safety test of customized natural fumigation agent (ArchiPer®) for paper materials**

1). **Tested material , testing method**

(a) **Tested material**

Treat traditional paper and medium quality paper with natural fumigation agent, and check the physical, chemical changes of papers during long keeping to find degradation stability. Dry artificial degradation test is adopted to analyze safety of treated papers for long keeping in terms of changes of pH, folding endurance, brightness, breaking length etc. .

○ Characteristics of traditional paper
  Basis weight(g/m²): 45g/m², paper fiber : White Broussonetia kazinoki : 100%
  Cooking method (NaOH): ash, Bleaching : sunlight ,
  Pulping : Broussonetia kazinoki Bat , Paper making: single mat,
  Drying : heat plate

○ medium quality paper : Basis weight 60g/m² , contains 40 ~ 70% of Bleached chemical pulp

(b) **Degradation properties of treated papers**

Damages of paper materials are shown as physical & chemical changes, and biological degradation by microorganism , insects.
Paper is consist of Cellulose, Hemicellulose, lignin, additives, gelatin, starch which are polymer, and can be good foods for microorganism. Mostly Fungi love acidic status, Carbohydrate, so that easily grow at paper materials. Bacteria can easily grow at alkali environment, protein. Nutrient, temperature, moisture are important for microorganism to survive.

(c) Customized natural fumigation agent (ArchiPer\textsuperscript{plus})
This agent is harmless to human body, environment friendly vegetable natural fumigation agent, carefully selected to protect archives, stack rooms from biological damages

(d) Tested sample papers is put into the temperature & humidity chamber (0.1 m\textsuperscript{3}).
Spray ArchiPer\textsuperscript{plus} with following conditions for 10 days, and examine the safety of papers.

- ArchiPer\textsuperscript{plus} spray conditions
  - condition 1: not treated sample
  - condition 2: normal (173.25 m\textsuperscript{3}/30 mg/7.5min)
  - condition 3: 10 times of normal
  - condition 4: 100 times of normal
- Duration of spray : 10days

(e) Dry artificial degradation method
Treated paper samples (traditional paper, medium quality paper) is tested at Circulation dry oven for long term safety & preservation quality under temperature : 105±2°C (Tappi T453Pm-85) method.
Traditional paper is tested for 20 days, 40 days, 60 days, Medium quality paper is tested for 7 days, 14 days, 21 days as degradation condition.

(f) Degradation safety test of paper materials after treatment with ArchiPer\textsuperscript{plus}
Sample preparation for long term safety test after ArchiPer\textsuperscript{plus} treatment is done under Tappi Standard T220 om-83 with temperature 20±1°C, relative humidity 65±2%\textsuperscript{o}. Physical & chemical property measurement for degradation test is done with folding endurance (Tappi Standard : T511om-83), Tensile strength (Tappi Standard : T494om-81), Brightness (Tappi Standard : T412om-83), pH(Tappi Standard : T509om-83) for 10 times each.

(g) pH measurement and analysis
Sample treatment at 105±2°C, 0% Relative Humidity for artificial degradation measurement, and examine the change of each sample. pH is measured with pH Meter on the sample surface as; traditional paper is at 20 days, 40 days, 60 days, and medium quality paper is at 7 days, 14 days, 21 days, measuring 10 times each with pH Meter.

(h) Folding endurance is tested for samples of artificial degradation with FOLDING ENDURANCE TESTER under TAPPI T 512 om-83 (MIT Schopper type).
(i) Brightness Test
Under artificial degradation condition of sample at temperature 105±2°C, 0% RH, color is measured with Color instrument under TAPPI T 412om-83.

(j) Breaking length test
Dry artificial degraded paper samples as traditional paper for 20 days, 40 days, 60 days, as medium quality paper for 7 days, 14 days, 21 days, measure breaking length with L&W TENSILE TESTER under TAPPI T 494 om-88.

2) Safety Test results of treated paper samples with ArchiPerplus: customized natural fumigation agent

(a) Endurance property of paper material under artificial degradation
Folding endurance is the key index to show degradation level of paper (Wilson). Artificial degradation under 100°C, 72 hrs is equivalent with 24 years natural degradation. Fig 9, Fig 19 show folding endurance changes of traditional paper and medium quality paper at non treatment, normal treatment of ArchiPerplus, 10 times of normal usage, 100 times of normal usage.

The test result shows there is no quality changes after ArchiPerplus treatment at normal, 10 times, 100 times compare with not-treated sample.

![Graph](image)

*Fig. 9 Effect of folding endurance by accelerated aging (traditional paper)*
Fig. 10 Effect of folding endurance by accelerated aging (medium quality paper)

Fig. 11-12 show breaking length by accelerated aging. Property of paper upon increasing of ArchiPer⁺ usage is not changed compared with not-treated sample.

Fig. 11 Effect of breaking length by accelerated aging (traditional paper)
Fig. 12 Effect of breaking length by accelerated aging (Medium quality paper)

(b) Brightness and PH changes by artificial degradation (accelerated aging)

Severe change of brightness shows chemical change of the paper, and the change of brightness decrease the value of documents, show degree of damage.

Fig. 13 Effect of brightness variation by accelerated aging (traditional paper)
Fig. 14 Effect of brightness variation by accelerated aging (Medium quality paper)

Brightness after artificial degradation (accelerated aging) is not changed compare with not-treated sample.

Fig. 15 Effect of pH by accelerated aging (Traditional paper)
d. Conclusion
Biological degradation of paper materials by microorganism shows surface contamination, color change, decoloring, hardening of material. Natural agent proves safety for the paper material, and antimicrobial activity.

4. Develop fully automatic fumigation system with customized natural fumigation agent (Archiper plus)

a. Natural degradation test of customized natural fumigation agent Archiperplus

1) Apply fumigation system for test
Instead of fumigation to stack room (50 M2), prepare test chamber of 1/400 size of stack room (450 liter) with air circulation unit. Auto spray system of Archiperplus for the chamber is also installed at the chamber to measure the change of TVOC value by GRAY WOLF, Toxic Gas TG-502 Probe, USA. (Total Volatile Organic Compound)
○ Spray condition
- Periodic spray into the chamber at 30 mg / every 7 minute
- Examine natural degradation time of the agent after 30 mg/ each spray

b. Examine the generation of ozone from the natural degradation of agent

1) Testing method &. Testing equipment

* Portable O₃ Analyzer
- UV absorption type
- Data logging
- Continuous measurement

This study is to measure ozone consistency which may be generated during natural degradation of natural agent ArchiPer⁺. Test Chamber is prepared with continuous ozone monitoring unit, auto spray system of natural agent. Ozone is measured upon different spray condition. (Fig 2)

< Table 1 > shows different spray condition of natural agent.

<table>
<thead>
<tr>
<th>No.</th>
<th>Spray condition</th>
<th>Fluorescent lamp</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Normal spray condition</td>
<td>use</td>
</tr>
<tr>
<td>2</td>
<td>Normal spray condition</td>
<td>use</td>
</tr>
<tr>
<td>3</td>
<td>Normal spray condition</td>
<td>use</td>
</tr>
<tr>
<td>4</td>
<td>Normal spray condition</td>
<td>use</td>
</tr>
<tr>
<td>5</td>
<td>LPG spray condition</td>
<td>use</td>
</tr>
</tbody>
</table>

Fig. 2 Measuring of ozone after spraying ArchiPer⁺ in chamber

2) Testing result of ozone generation from natural degradation of agent
Measured value of TVOC upon continuous spray as 30 mg/7min shows
Constant value TVOC value is not increase, but constant value).
This shows natural agent is naturally degraded to low molecular based material,
And there is not problems of ozone generation.

c. Analyze absorption degree to activated carbon filter for air-conditioning system application.

1) Testing materials and method

Soak 5g of activated carbon in natural agent for 1 hour, clean natural agent on the surface of
activated carbon with tissue wiper, and install at activated carbon cartridge of the chamber, measure
TVOC value under air circulation.
When TVOC value decrease up to initial value, spray natural agent periodically to check TVOC
value to check activated carbon function. (Fig. 3).

Fig. 3 Soaking carbon into ArchiPer\textsuperscript{\textregistered} plus

2) Check the influence of activated carbon filter when natural agent spray
system is installed at air conditioning system.

In case activated carbon system is adopted at air-conditioning system,
This test is to examine whether operation life of activated carbon filter is shorten
by absorption of natural agent or not.
3) Analyze of absorption result of activated carbon filter of air conditioning system. The test shows that activated carbon filter absorb natural agent, but natural degradation of natural agent, vaporization of natural agent make no harm to activated carbon filter of air conditioning system. In case activated carbon filter is adopted to improve air quality, natural agent spray system can be operated together.

d. Corrosion test of metal pipe, duct by natural agent spray via air conditioning system

1) Testing material and method

(a) Testing materials
3 kinds of metal sample (akino, color steel, galvanized steel) which are commonly adopted for air conditioning system, 4 kinds of general metal samples [Al, STS316, Cu, CM(normal metal)] with 32×32(mm) size.(Fig. 5).

(b) Testing method
Soak each sample at air, distilled water (comparison), natural agent at 20°C, 24 hours, and measure the weight, examine the surface condition with stereo microscope (ZEISS, Stemi 2000C), IMAGE ANALYZER(SIS) to check corrosion status.

(c) Measure change of metal weight to check corrosion level
Measure weight difference to check corrosion of metal after reaction of metal and liquids.

(d) Examine corrosion status by micro scope
Examine metal surface with stereo micro scope (ZEISS, Stemi 2000C), IMAGE ANALYZER(SIS).

2) Examination result of corrosion test for metal sample.
Corrosion phenomenon is found at distilled water soaking sample, and not found from the soaking samples with air or natural agent. Natural agent not make corrosion to metal.
e. Examine whether natural agent affect to smoke detectors at stack room.

1) Testing materials and method

Prepare 3 types of smoke detectors such as beam analog type which is installed at Nara Repository, ion type, non linear type, 3 sets each. And test whether natural agent is detected by smoke detectors or not

(a) Examine any influence to smoke detector in the test chamber

This is to test whether sprayed natural agent can react smoke detectors or not. The test is performed with 400 times of spray than normal spray condition at stack room. (Fig. 6).

2) Examination result in the chamber

Under 400 times spray, smoke detector not react the spray, (Fig. 7).

Fig. 7 Effect to the smoke sensor by amount of spraying ArchiPer$^{\text{plus}}$ at Pilot Plant
f. Analyze disinfection effect after apply natural agent via air conditioning system

1) Testing materials and method

Measure effective consistency of natural agent with TVOC tester (GRAY WOLF, Toxic Gas TG-502 Probe, USA). When Natural agent is sprayed into the air, some of agent is naturally decomposed, and should maintain effective consistency of natural agent for effective fumigation. To get standard figure, measure TVOC value upon microorganism growth level. Test condition of stack room (chamber) is at 27°C for fast test. (Normal operation condition of stack room is temperature (20±2°C), Humidity (45±5%)).

2) Examine test result

(a) Disinfection effect with effective consistency of natural agent

Put prepared media (PDA, NA) in incubator. Measure consistency of natural agent inside of incubator with TVOC tester continuously. (GRAY WOLF, Toxic Gas TG-502 Probe, USA)

Examine the growth of microorganism on media according to the consistency of natural agent.

3) Examination result to test effective consistency of natural agent

Test results show that effective disinfection (block microorganism growth, and insecticide effect) under following cases:
- Maintain TVOC at 800ppb for 30 minute at 1 time spray per day
- Maintain 10 ppb
- Maintain TVOC at 200ppb for 30 minute at 1 time spray per day, Shows effectiveness from 7th day (Fig. 32).
4) Application Plan of study

Customized natural agent which is harmless, and safe can be adopted for automatic mass disinfection system, enable to provide safer, unmanned system.
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