No. 5 Format

National Institute of Advanced Industrial Science and Technology No.092-7614

March 8, 2018

KISTEC

Test Measurements Results Report

SBMplus Co., Ltd.

705-1 Shimoimaizumi, Ebina, Kanagawa Prefecture 243-0435

Regional Independent Administrative Institution

President, National Institute of Advanced Industrial Science and Technology, Kanagawa Prefecture

The results of the test measurements that were requested on March 2, 2018 are as follows.

Test measurement type (name): NO2 Removal Test Gas bag method

Item name as listed on the application form: eco FRESH (LIQUID)

eco FRESH (GEL)

Continued on next page

(Note) The descriptions in this certificate are the test results for the test items brought by the user, and do not guarantee the performance, efficacy, etc. of the entire product (material, parts, products etc.).

The product name, rating, specifications, etc. of the test measurement items are those stated based on the application of the applicant.

When using the name of our laboratory in catalogs and the Internet based on the results of test measurements, it is necessary to obtain approval for the use of the name in advance for each use.

Regional Independent Administrative Institution
National Institute of Advanced Industrial Science and Technology, Kanagawa Prefecture

1. Contents of Application

Conduct NO_2 Gas Removal Test

2. Method

Introduce prepared NO_2 gas to tedlar bag containing specimen, and evaluate the change in concentration.

. Contents of Specimen

Name (1) ORGALOS-ZERO/eco FRESH (LIQUID)

Sponge sheet 10 cm x 15 cm containing specimen

(2) ORGALOS GEL/eco FRESH (GEL)

Gel in 190 ml container

4. Test Method

4.1.1 Equipment and chemicals used

Humidity-generating equipment	SRG-1R-1L (DaiichiKagaku)
Gas collection equipment	GSP-300FT-2 (Gastec)
Gas bag	Material PVF, Size 5L (GL Science)
Detector tube (Gastec)	Nitrogen oxide detector tube 11HA
Sulfuric acid	Special grade reagent (Wako Pure Chemical)
Sodium nitrite	Special grade reagent (Wako Pure Chemical)

4.1.2 Test Method

1) A few drops of an aqueous solution of sodium nitrite were added to 71% sulfuric acid to generate NO_2 gas. This gas was collected with a syringe and introduced into a tedlar bag such that the nitrogen oxide concentration was about 600 ppm while flowing air (3L) conditioned to 50% relative humidity. Gas was introduced from this bag to the bag containing the sample.

2) The concentration of nitrogen oxide was measured immediately after the gas introduction, and at 2 h, 4 h and 24 h with a detector tube.

4.1.3 Test Date

Continued on next page

5. Results

The results of the test are shown in Table 1.

Table 1 Change in concentration of nitrogen oxide (ppm) over time for each specimen

	Supplied concentration	Immediately after gas introduction	2 h	4 h	24 h
(1) ORGALOS-ZERO/eco FRESH (LIQUID)	600	330	100	30	0
(2) ORGALOS GEL/eco FRESH (GEL)	600	570	400	350	280

END

Japan

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No. 104062007-001

Skin Primary Irritation Test Using Rabbits

Summary

Using Grafton LNF as a specimen, a skin primary irritation test using rabbits was conducted according to OECD Guidelines for the Testing of Chemicals 404 (1992).

Specimens were applied to 3 rabbits on intact and abraded skin for 4 hours by closed patch. As a result, extremely mild erythema was observed in all cases one hour after removal, but disappeared in 24 hours.

The primary irritation index (PII) obtained in accordance with Federal Register (1972) was 0.3, and in the skin primary irritation test using rabbits, the specimen was evaluated as falling within the category of "nonirritating."

Requested By

Test Item

Grafton LNF

Test Period

July 5, 2004 to July 23, 2004

Test Location

Japan Food Research Laboratories Tama Laboratory

11-10 Nagayama 6-chome, Tama-shi, Tokyo

Person Responsible for Testing

Japan Food Research Laboratories Tama Laboratory

Safety Test Department Safety Test Section

Shinichi Katsuta

No. 104062007-001

Test Implemented By

Tomoko Shimazaki, Takeshi Nagai, Jun Fukai, Yasuharu Kawamoto

1 Test Objective

To examine primary irritation of the skin in rabbits for the specimens according to OECD Guidelines for the Testing of Chemicals 404 (1992).

2 Test Item

Grafton LNF

Properties: Slightly yellowish transparent liquid

3 Test Animals

Japanese white rabbits were purchased from Kitayama Labes Co., Ltd. and after preliminary raising for over 1 week, and confirming that there was no abnormality in their general condition, 3 rabbits were used for the test. The test animals were individually housed in FRP cages and kept in a breeding room set at room temperature of 22° C +2° C and illumination time of 12 hours/day. Feed was restricted to rabbit / guinea pig solid feed (LRC4, Oriental Yeast Co., Ltd.), and tap water was allowed as drinking water ad libitum.

4 Test Method

The trunk back coat of each test animal was shaved approximately 24 hours before the test. For each test animal, 4 points with an area of about 6 cm^2 were set, then using an 18-gauge injection needle, a well-like scratch was made in the cornified layer, not reaching the dermis, in 2 points (abraded skin), and the other 2 points were kept intact (intact skin).

0.5 mL of the specimen was uniformly applied to a gauze patch cut to about 2 cm × 3 cm, and affixed to 1 intact and abraded skin point, then fixed with a plaster (PMDA). In addition, the plasters were held by Blenderm Surgical Tape [3M Healthcare Co., Ltd.] so that the patches came in contact with the skin. The remaining intact and abraded skin points were used as controls.

The exposure time was 4 hours, after which the patches were removed, and the exposed surface cleaned with pure water. Observations were made at 1, 24, 48 and 72 hours after removal, and the stimulation reaction was scored according to Table 1. Also, in accordance with Federal Register (1972), the scoring values for 1, 24 and 48 hours after patch removal were totaled, divided by 6, and the mean of each test animal was calculated to obtain the primary irritation index (PII), and the irritation of the specimen was evaluated based on the standard of ISO 10993-10 shown in Table 2.

The body weight of the test animals was measured at the start of the test and at the end of the test.

5 Test Results (Tables 3 and 4)

Very mild erythema (score 1) was seen at all applied sites at 1 hour after removal but disappeared at 24 hours, and thereafter no irritation reaction was observed.

The PII calculated from the scoring result was 0.3.

6 Evaluation

A primary skin irritation test for the specimen using rabbits was conducted according to OECD Guidelines for the Testing of Chemicals 404 (1992).

As a result, extremely mild erythema was observed in all cases 1 hour after removal but disappeared in 24 hours.

Primary irritation index (PII) obtained in accordance with Federal Register (1972) was 0.3, and in the primary skin irritation test using rabbits, the specimen was evaluated as falling within the category of "nonirritating."

7 References

・ Federal Register (§ 191, December, 1972).

・ ISO 10993-10 Biological Evaluation of Medical Devices-Part 10: Tests for irritation and delayed-type hypersensitivity 6.3 Animal skin irritation test (2002).

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Table 1 Evaluation of skin reaction

Erythema and crust formation

No erythema 0

Very mild erythema (barely discernable) 1

Clear erythema 2

Moderate to high erythema 3

From high erythema to formation of slightly crusted skin (deep damage) 4*

[Highest score 4]

* Scores for bleeding, ulcers and necrosis are classified as deep damage.

Formation of edema

No edema 0

Very mild edema (barely discernable) 1

Mild edema (clear distinct edge due to distinct bulge can be identified) 2

Moderate edema (bulging about 1 mm) 3

High edema (bulge of 1 mm or more and spread beyond the exposure range) 4

[Highest score 4]

Reaction category	P.I.I.
Non-irritancy	0~0.4
Weak irritancy	0.5~1.9
Moderate irritancy	2~4.9
Strong irritancy	5~8

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Table 2 Primary stimuli reaction categories in rabbits

Table 3 Test animal body weight (kg)

Test animal	At start of testing	At end of testing
(1)	3.43	3.42
(2)	2.58	2.66
(3)	3.16	3.15

Table 4 Scoring results of skin reaction

Observation time (hours)	Test animal (1)		Test animal (2)		Test animal (3)	
	Intact	Abraded	Intact	Abraded	Intact	Abraded
1	1/0	1/0	1/0	1/0	1/0	1/0
24	0/0	0/0	0/0	0/0	0/0	0/0
48	0/0	0/0	0/0	0/0	0/0	0/0
72	0/0	0/0	0/0	0/0	0/0	0/0

Results are shown in order of erythema / crust formation / edema.

END

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No. 103113958-001

Acute Oral Toxicity Test Using Female Mice

Summary

An acute oral toxicity test (limit test) using female mice was performed using Grafton LNF as a specimen. Samples at a dose of 20 mL/kg were orally administered to female mice at a dose of 20 mL/kg in the test group and water for injection was orally administered to the control group for 14 days. As a result, no abnormalities and deaths were observed during the observation period. From this, it was considered that the LD50 value by a single oral administration in the specimen mice was 20 mL/kg or more in females.

Requested By

Test Item

Grafton LNF

Test Period

December 11, 2003 - January 6, 2004

Test Location

Japan Food Research Laboratories Tama Laboratory

11-10 Nagayama 6-chome, Tama-shi, Tokyo

Person Responsible for Testing

Japan Food Research Laboratories Tama Laboratory

Safety Test Department Safety Test Section

Shinichi Katsuta

Test Implemented By

Japan
Food
Research
Laboratories

No. 103113958-001

Tomoko Shimazaki, Takeshi Nagai, Jun Fukai

1 Test Objective

To examine acute oral toxicity in female mice for the specimen.

2 Test Item

Grafton LNF

Properties: Slightly yellowish transparent liquid

3 Test Animals

Five-week-old female ICR mice were purchased from Japan SLC, Inc., preliminarily raised for about one week to confirm that there was no abnormality in general condition, and then used for the test. The test animals were housed in polycarbonate cages holding 5 animals each and kept in a breeding room set at room temperature of 23° C +2° C and illumination time of 12 hours/day. Feed (solid feed for mice/rats; laboratory MR stock, Nosan Corporation] and drinking water (tap water) was allowed as drinking water ad libitum.

4 Test Method

Five animals were used in the test group and the control group, respectively. The test animals were fasted for about 4 hours before administration. After body weight was measured, a single forced oral administration of the specimen was given to the test group, and of water for injection to the control group at a dose of 20 mL/kg using a stomach tube.

The observation period was 14 days, and frequent observations were made on the administration day, followed by daily observations from the next day. Body weights were measured 7 and 14 days after administration and comparisons between the groups were made at a significance level of 5% by t-test. At the end of the observation period all animals were necropsied.

5 Test Results

(1) Fatal cases

No deaths were observed in any of the administration groups during the observation period.

2) General condition

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No abnormality was observed during the observation period in any of the administration groups.

3) Weight change (Table 1)

In the body weight measurement on days 7 and 14 after administration, there was no difference in body weight value in the test group compared to the control group.

4) Necropsy findings

At necropsy at the end of the observation period, no abnormalities were observed in any of the test animals.

6 Discussion

For the specimens, an acute oral toxicity test (limit test) using female mice was performed. As a result of a single oral administration of the specimen at a dose of 20 mL/kg, no abnormalities or deaths were observed during the observation period. Therefore, the LD50 value by a single oral administration of the specimen for mouse was considered to be 20 mL / kg or more in females.

7 References

・ OECD Guidelines for the Testing of Chemicals 420(2001).

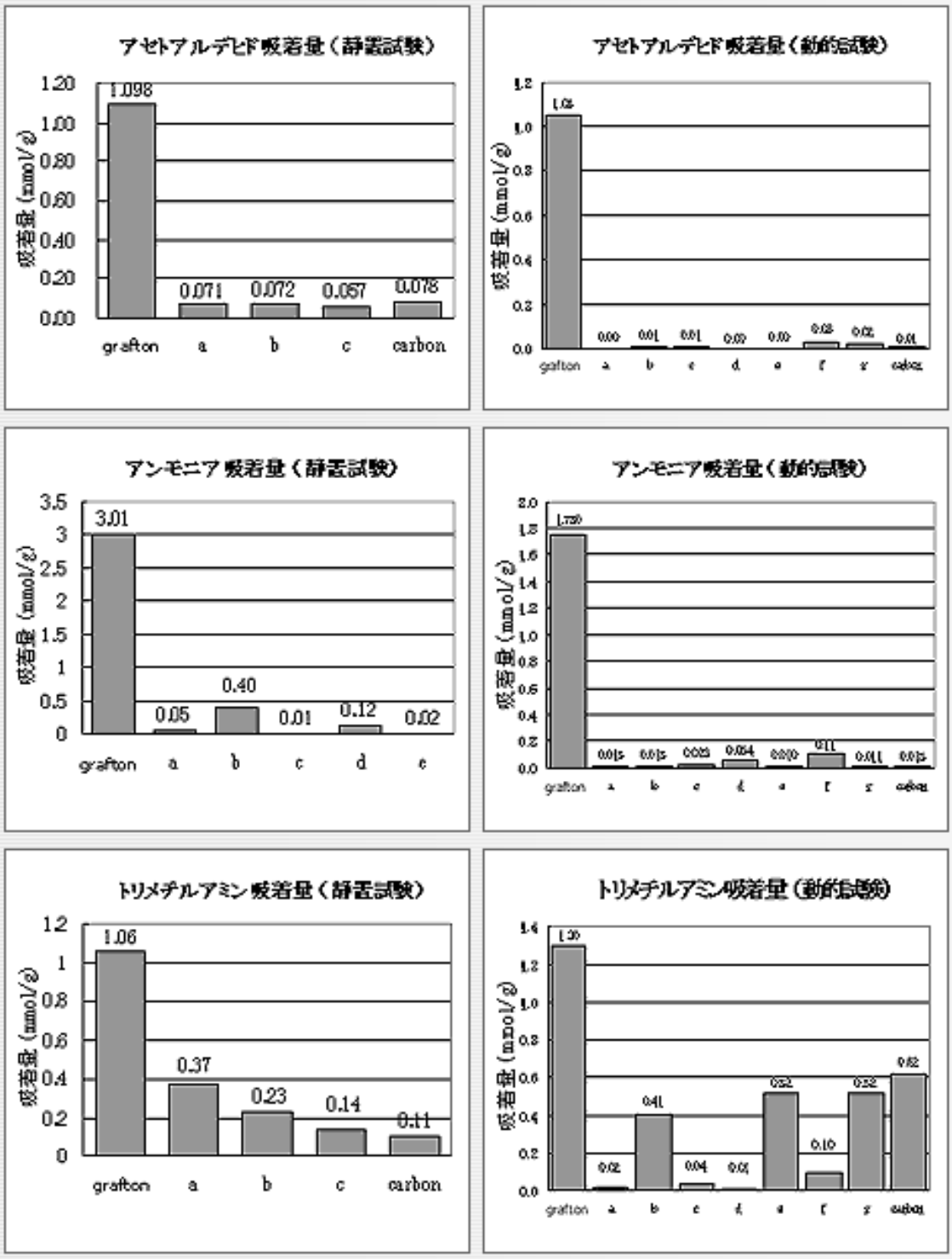
Table 1 Weight change

Administration group	Before administration	After administration (days)	
		7	14
Test group	28.0+0.9(5)	30.3+1.6 (5)	33.4+1.7 (5)
Control group	27.7+0.8 (5)	31.0+1.5 (5)	33.8+2.2 (5)

Body weight is expressed as mean + standard deviation (unit: g). The numbers of animals are shown in parentheses.

END

グラフト重合剤による各種試験データ



商品名：ECO FRESH

試験方法

5ガス体、とくに悪臭に対する試験方法は未だ公的に規定されたものはない。したがって環境技術研究協会発行の環境技術別刷に掲載された東大工学部の測定方法に準拠して行った。

検体の重量は80g。動的試験の各社の製品はそのまま使用し、積算吸着容量は試料重量で換算した。

IMPROVEMENT OF IAQ BY COATING OF ADSORPTIVE POLYMER: THE DEVELOPMENT OF MATERIAL, EVALUATION AND ITS APPLICATION

M Hori[1*], T Ohkawara[2], S Handa[2] and T Wakui[1]

[1]Yokohama National University; Yokohama, Japan
[2]Grafton Inc., Tokyo, Japan

ABSTRACT
A coating material of adsorptive polymer by graft polymerization was developed for the purpose of improving IAQ to decrease formaldehyde and VOC concentrations. The material was evaluated by a simple method with a small glass chamber of a circulation system. The ability of sealing of both of formaldehyde and VOC and adsorption, respectively, was performed by emission rate test and adsorption test. The polymer with three kinds of function group of sulfonic, amino and carboxyl was relatively effective to sealing and adsorption. The material was superior to the other materials on market in Japan. The material was applied to improve IAQ in two kinds of new multi-family house, and was evaluated by concentrations of indoor air and emission rate from individual parts. It was shown that formaldehyde concentration and TVOC, respectively, decrease to one fifth or one sixth and a half in the room treated with the coating material.

INDEX TERMS
VOC, Formaldehyde, Adsorptive polymer, Improvement of IAQ, Coating

INTRODUCTION
Improvement of IAQ by coatings is required when VOCs and formaldehyde concentrations can not be decreased to use selected materials. The required conditions of coating material are follows: Strength of coating membrane; Adsorbent of both of VOC and formaldehyde; To be coated easily with brash, roller or spray; Water soluble; Non-color; Economical. A coating material with Graft polymerization was developed by Ohkawara in 1996. In this paper it was studied to apply the material with graft polymerization to coating material for chemicals emission control. Sealing effect test for emission control and adsorption test for removal from indoor air was performed to evaluate the coating materials with a simple method of circulation system, and in addition to the material test field test was performed.

MATERIAL AND METHODS
Material: Coating material was prepared to add some kinds of functional groups to base of carbohydrade such as glucose in 6 h exposure of radiation in the graft polymerization. Acrylate of carboxyl group forms coating film, and urea of amino group, styrene sulfonic acid and ethanol as a polymerization adjustor was also mixed. The functional groups were also added on a seat such as cell rose with graft polymerization.

Evaluation of the ability to seal formaldehyde and VOCs in a board: Emission test was performed in a small glass chamber of 6.5L with a circulation system shown in Figure 1(1).

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One or 3 pieces of sample veneer boards and control of 20cm ×10cm was set, and volatile compounds emitted for 3h to 24h were collected with a DNPH cartridge or an active carbon tube (Figure 1). Formaldehyde and VOCs in the sample respectively were determined with HPLC and GC method. The chamber was set in an experimental room controlled at 23 to 25°C and on the other hand the humidity was not controlled. The temperature and humidity were monitored when necessary. The sample was prepared by coating of a material at a fixed weight per unit surface area of boards. Sealing efficiency is estimated by (B – A) /B, provided that the emission weight from a sample and control board is A and B.

Evaluation of the ability of the material to adsorb formaldehyde and VOCs in surrounding air: Adsorption test was performed with a chamber of 6L shown in Figure 1(2). A sample was prepared to coat the material on a glass plate or a veneer board that the emission had been controlled enough of 20cm ×10cm. The chamber system has a stirring fan and an injector of formaldehyde or VOCs and a monitor inserted to the circulation line. After the sample or control board was inserted in the chamber, formaldehyde or VOCs was injected. Formaldehyde was prepared by vaporization from formarine and monitored with a potential-controlled gas sensor. A VOC was prepared by injection of the vapor with a syringe and monitored with a gas sensor on semiconductor. When concentrations of monitor became constant, the concentrations for the sample and control (A and B) were read. Removal efficiency was estimated by (B – A) / B.

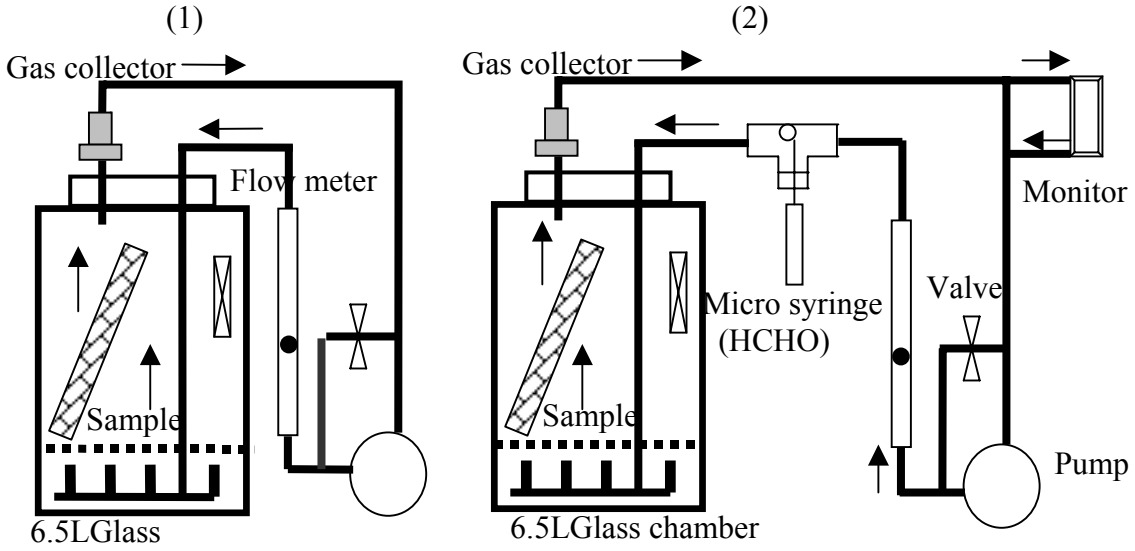

Figure 1. Simple chamber system for emission test (1) and adsorption test (2).

The concentrations of formaldehyde and VOCs in fields, respectively, were measured according to determination method of ISO 16000-3 and 6. The sampling was performed for 30 minutes after elapse of 5 hours from time when doors and windows had been shut. Emission rate of formaldehyde in field: TEA-Dish method developed by Hori et al was applied to the measurement. It is a simple method for measurement of formaldehyde emission rate parallel to many points in field. The procedure is as follows: A filter of cell rose permeated with 10 % triethanolamine(TEA) fixed on inner surface of a dish of aluminum of 20 cm in diameter, which was put on surface of parts such as ceiling and wall for 24h. Formaldehyde collected with the filter was determined with AHMT absorption method after abstraction to water.

RESULTS AND DISCUSSION

Effect of function groups of material on sealing efficiency.

Sealing efficiency of kinds of function groups added to a base of glucose on emission rate from coated veneer boards was as follows: Carboxyl group 52%; Carboxyl and amino group 72%, Carboxyl, amino and sulfonic group(sulfonic 0.5) 82%, Carboxyl, amino and sulfonic group (sulfonic 1.4) 85%. Emission rate of formaldehyde from control veneer board was approximately 20 μg /m^2 h. Addition of amino and sulfonic group was effective for formaldehyde, and that of carboxyl group was slightly effective. It is assumed that amino(NH_2-) and sulfonic(-$C_6H_4SO_3H$) groups bring about chemical adsorption of formaldehyde, and coating of carboxyl group and urea bring about only physical sealing effect. Material of amino, sulfonic and carboxyl group addition was used in this study.

Comparison with the other kinds of coating material

Examples of sealing efficiency and adsorption efficiency in some kinds of material are shown in Table 1. Sealing test was performed, and subsequently adsorption test was performed. When the sealing efficiency of a sample is low in the sealing test, the adsorption efficiency was estimated to correct the concentration of formaldehyde emitted in the sealing test. Therefore it is presumed that the measurement values are low in precision. The result shows that the coating material of No.9 is superior to the others on market in Japan for sealing and adsorption of formaldehyde. Adsorption test of this material is going to be performed in the concentration of 0.1 to 0.2 ppm

Table 1. Examples of sealing efficiency and adsorption efficiency of formaldehyde in some kinds of material.

	Sample	Non-coating (μg/h)	Emission rate (μg/h)	Sealing (%)	Adsorption (%)
1	Natural organic	14	0.47	82	25
2	Synthetic paint(1)	14	0.42	97	47
3	Synthetic paint(2)	14	0.58	96	70
4	Synthetic paint(3)	14	2.7	5.5	20
5	Ceramic paint	14	5.5	61	40
6	Synthetic coating (1 time)	31	20	35	-
7	Synthetic coating (2 times)	31	10	67	-
8	Natural inorganic	14	0.79	98	89
9	Present material	13	0.31	98	94
10	Cell rose sheet*				97

Control(non- coating): veneer board of 20×10×9 cm

* Function group was added directly on seats of Cell rose with graft polymerization.

Mount of formaldehyde injected in adsorption test was 9.9 μg(1.2ppm).

Samples of No.2,3,4 were measured with a potential-controlled monitor, and the others with DNPH method because of interference of VOC .

The sealing of formaldehyde is assumed to result from decrease of diffusion area on coating membrane. This effect also affects for VOCs emitted from inner building materials. Concentrations of formaldehyde in the chamber decreased when the material was inserted in the chamber. The result means that formaldehyde adsorbed to surface of the coating material. Formaldehyde is assumed to adsorb on the amino group because of high polarity of formaldehyde, but it is not clear for the adsorption to be followed by dehydration from HCHO

and NH_2- such as polymerization of urea resin. It has a limited adsorption amount of formaldehyde and VOCs. Therefore this material is presumed to be effective for improvement of IAQ as amount of them emitted to an indoor air is limited, and it is necessary to evaluate the effect again after 1 or 2 years.

Field test

This coating material was applied in new multi-family houses of 38 m^2 in area. One of them was treated with the coating material which was 2.4 L in volume of undiluted solution and about 2 g/m^2 in dry weight in the surface. In addition the sheets of No.10 in Table 1 and 18 m^2 in total area were inserted under wall and flooring. After two weeks sampling of air in both of the room and the other that had not been treated was performed. The measurement values is shown in Figure 2. The concentration of formaldehyde decreased to one fifth. Some kinds of VOC except ethanol that the concentration decreased more than the other VOCs in this field test is shown. The ethanol comes from a polymerization adjustor. The concentration of ethanol is presumed to decrease rapidly because of vaporization from the surface.

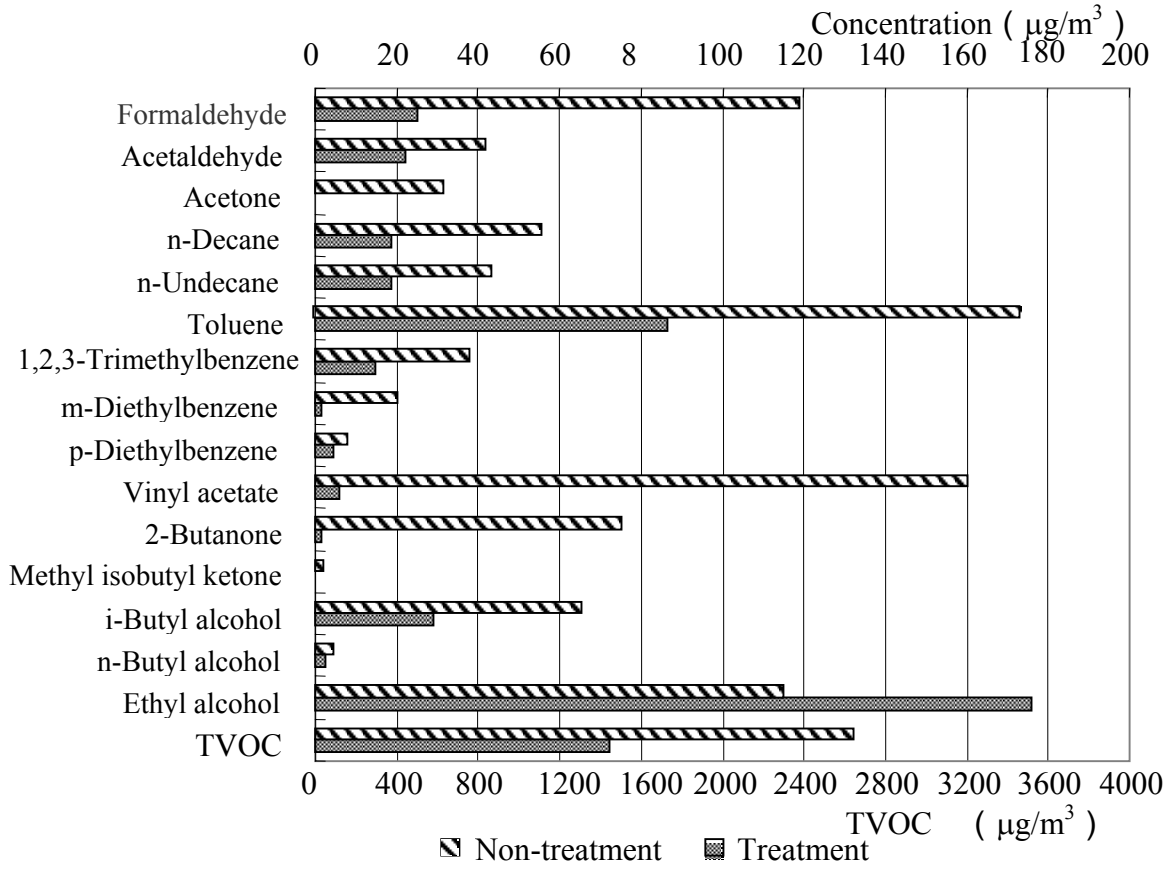

Figure 2. Comparison of the concentration of formaldehyde and VOCs in a room treated with the coating material with that in a non-treated room.

The room has two ventilation fans only in a bathroom, a toilet and a kitchen and does not air supply holes. These fans are operated in use time. Ventilation rates of the room with and without operation of the fans of bathroom and a toilet, respectively, were 0.17 and 0.4 (1/h). The concentration was measured after elapse of 5 h without operation of the fan from when windows and doors had been shut. We ordinary dwell in the conditions. If HVAC of 0.5 in ventilation rate is applied, the concentration of formaldehyde would be estimated to be 40 $\mu g/m^3$. In resent years new residential houses of a full time ventilation has increased in Japan, but the great portion of new houses does not have it, even if airtight houses. Therefore the

improvement of IAQ with the building materials is considered to be valid

Emission rate of formaldehyde from individual parts of building material used in the other new multi-family house was measured with TEA-Dish method. The effect of treatment by the coating material was evaluated to compare emission rate of a part in a treated room with that of the same part in non-treated room. Amount emitted from a part was estimated by product of surface area of the part and the measured emission rate. Contribution(%) of a part such as wall is shown as ratio of amount emitted from the part to total amount. The results are shown in Figure 3 and 4. The sampling was performed for 24h after 3 days. Total amount of formaldehyde emitted decreased one sixth. The result shows that coating to both of wall and ceiling was effective, and that of flooring was not effective. After treatment both of door and

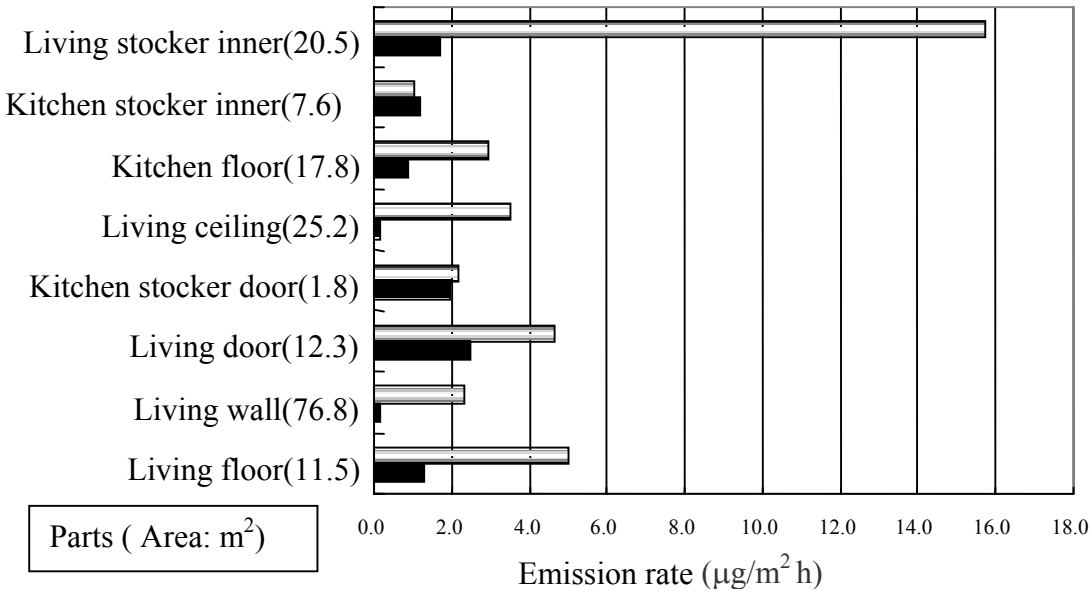

Figure 3. Emission rate of formaldehyde emitted from individual parts; Comparison of non-treatment with treatment. The surface area of each parts was described in parentheses. Upper bar: Non-treatment; Lower bar: Treatment. Surface of the kitchen stocker was not treated.

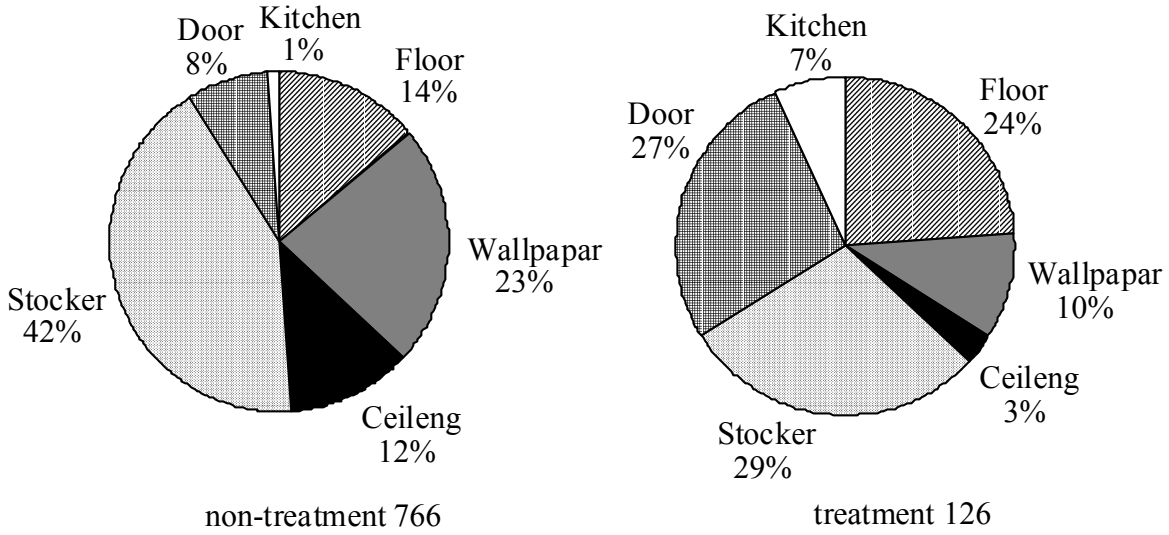

Figure 4. Contribution of individual parts for emission of formaldehyde in treatment and non-treatment room. Number of bottom is total emission amounts (μg/h) in the room calculated by ∑ RA; R: Emission rate of parts constructed with a material and A: Total area of the part. They are described in Figure 3.

Surface of stocker made relatively large contributions to emission of formaldehyde. It is necessary to study how to coat effectively to surface of melamine resin of stocker and flooring. This coating material is valid to improve IAQ in not only new houses but also established sick houses on formaldehyde and some kinds of VOCs.

REFERENCES

Hori M. 1997. Proposal of sampling methods and a simple method for emission rate measurement of formaldehyde and VOC, *The Proceedings of Academic Lecture Meeting on Heating, Air-conditioning and Sanitary Engineering,* Ⅱ pp 405-408(in Japanese).

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Ohkawara, T et al. 1996. Adsorptive materials and process for producing them, United States Patent 5,506,188, April 9.