

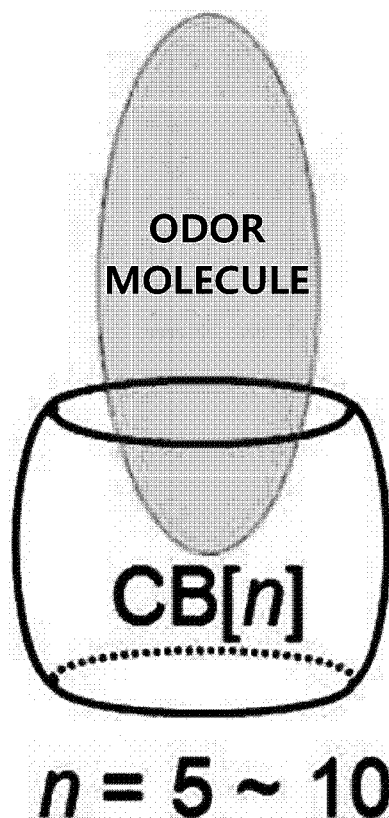


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[Continued on next page]

- (54) Title: COMPOSITION FOR ODOR REMOVAL COMPRISING CUCURBITURIL



(57) Abstract: Disclosed herein is a composition for removing odor, the composition including cucurbituril. The composition can capture and remove various kinds of odor materials (particularly, ammonium ion-containing bad odor, such as a fishy smell or the like) because cucurbituril can interact with various odor materials using its hydrophobic cavity and both inlets covered with carbonyl groups. Therefore, this composition can be practically applied to the cleaning of air and the improvement of an environment.



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Description

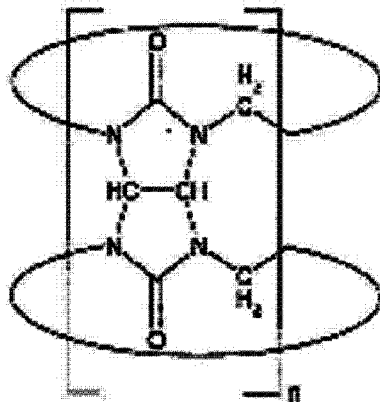
Title of Invention: COMPOSITION FOR ODOR REMOVAL COMPRISING CUCURBITURIL

Technical Field

- [1] The present invention relates to a composition for removing an odor, the composition including cucurbituril, and a method of preparing the same.

Background Art

- [2] There are many odor removing methods. For example, there are methods which use a material to block a receptor of an olfactory organ, methods whereby an odor material is oxidized, methods of removing odor-generating bacteria, methods that use a strong fragrant material, and methods of capturing an odor molecule using a porous material such as zeolite, silica or the like. Among these methods, the method of capturing an odor molecule using a porous material is efficient, but it is difficult to incorporate and apply this method in the form of spray. Therefore, recently, macrocyclic molecules that have water-soluble properties and can capture odor molecules have attracted considerable attention. Cyclodextrin is a typical example of such macrocyclic molecules. Cyclodextrin was discovered in 1891 and has been actively researched ever since it was discovered that cyclodextrin reacts with several compounds to form inclusion complexes. Thus, cyclodextrin has been utilized in various fields such as food manufacture, medicine and the like.
- [3] Meanwhile, cucurbituril is a material which was first reported by R. Behrend, E. Meyer and F. Rusche in 1905. In 1981, W. Mock and others discovered that this material is a macrocyclic compound composed of six monomers and having a chemical formula of $C_{36}H_{36}N_{24}O_{12}$, and they observed the structure thereof using X-ray diffraction (J. Am. Chem. Soc. 1981, 103, 7367). They called this compound "cucurbit[6]uril". Later, in 2000, K. Kim and others synthesized and separated cucurbit[n]urils ($n = 5, 7, 8$), as homologues of cucurbit[6]uril, as well as cucurbit[6]uril, by improving a conventional method of synthesizing cucurbit[6]uril, and they observed their respective structures using X-ray diffraction (J. Am. Chem. Soc. 2000, 122, 540). Meanwhile, International Patent Application Publication No. WO 00/68232 discloses cucurbit[n]uril which is represented by Chemical Formula below:
- [4] [Chemical Formula]
- [5]



[6] wherein n is an integer of 4 to 12.

[7] Each of the cucurbituril derivatives is a compound composed of glycoluril monomers having no substituent group (KR-A-10-2007-0073225).

[8] It is proposed in the prior patent of the present inventors that cucurbituril can be used as a bad odor remover or the like because it has a structure which can be bonded with an organic compound. However, experiments that may support this possibility have never been carried out by the present inventors or other researchers. Thus, the present inventors experimentally found that cucurbituril can fulfill a function of removing an odor, and the present inventors also found the optimal composition ratio of the complex and the optimal conditions required to exhibit functions. Based on these findings, the present invention was completed.

Disclosure of Invention

Technical Problem

[9] Accordingly, the present invention has been devised to solve the above-mentioned problems, and an object of the present invention is to provide a composition for removing an odor, the composition including cucurbituril, which can effectively remove odors caused by various kinds of materials.

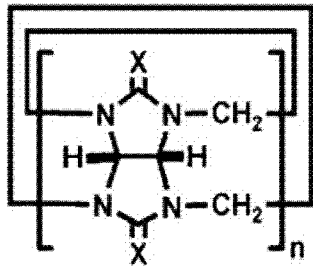
[10] However, an object to be accomplished by the present invention is not limited to the above-mentioned object, and other objects (not mentioned) will be clearly understood from the following descriptions by those skilled in the art.

Solution to Problem

[11] In order to accomplish the above object, an aspect of the present invention provides a composition for removing an odor, the composition including a compound represented by Chemical Formula 1 below:

[12] [Chemical Formula 1]

[13]



- [14] wherein n is an integer of 4 to 20, and X is O, S or NH, and
- [15] wherein the composition includes 45 to 100 wt% of the compound of Chemical Formula 1 of $n = 7$.
- [16] The compound of Chemical Formula 1 is cucurbituril.

Advantageous Effects of Invention

- [17] The composition of the present invention can capture and remove various kinds of odor materials (particularly, ammonium ion-containing bad odor, such as a fishy smell or the like) because cucurbituril can interact with various odor materials using its hydrophobic cavity and both inlets covered with carbonyl groups. Further, since the composition can effectively remove odors from various odor emission materials according to the composition ratio of cucurbituril homologues, it can be made into various products by suitably adjusting the composition ratio of cucurbituril homologues.

Brief Description of Drawings

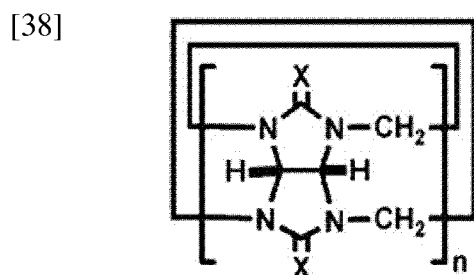
- [18] The above and other objects, features and advantages of the present invention will be more clearly understood from the following detailed description taken in conjunction with the accompanying drawings, in which:
- [19] FIG. 1 is a schematic view showing a complex of cucurbituril and an odor molecule;
- [20] FIG. 2A is a graph showing the effect of removing the odor of onion using cucurbituril;
- [21] FIG. 2B is a graph showing the effect of removing the odor of onion using beta-hydroxypropyl cyclodextrin (HY CD, deodorizer) and cucurbituril;
- [22] FIG. 2C is a graph showing the effect of removing the odor of onion using cucurbit[5]uril (CB[5]), cucurbit[6]uril (CB[6]), cucurbit[7]uril (CB[7]), cucurbit[8]uril (CB[8]) and crude cucurbit[7]uril (crude (CB[7]));
- [23] FIG. 3A is a graph showing the effect of removing the odor of garlic using cucurbituril;
- [24] FIG. 3B is a graph showing the effect of removing the odor of garlic using beta-hydroxypropyl cyclodextrin (HY CD, deodorizer) and cucurbituril;
- [25] FIG. 3C is a graph showing the effect of removing the odor of garlic using cucurbit[5]uril (CB[5]), cucurbit[6]uril (CB[6]), cucurbit[7]uril (CB[7]), cucurbit[8]uril (CB[8]) and crude cucurbit[7]uril (crude (CB[7]));

- [26] FIG. 4A is a graph showing the effect of removing the odor of pork belly using cucurbituril;
- [27] FIG. 4B is a graph showing the effect of removing the odor of pork belly using beta-hydroxypropyl cyclodextrin (HY CD, deodorizer) and cucurbituril;
- [28] FIG. 4C is a graph showing the effect of removing the odor of pork belly using cucurbit[5]uril (CB[5]), cucurbit[6]uril (CB[6]), cucurbit[7]uril (CB[7]), cucurbit[8]uril (CB[8]) and crude cucurbit[7]uril (crude (CB[7]));
- [29] FIG. 5A is a graph showing the effect of removing the odor of a japanese spanish mackerel using cucurbituril;
- [30] FIG. 5B is a graph showing the effect of removing the odor of a japanese spanish mackerel using beta-hydroxypropyl cyclodextrin (HY CD, deodorizer) and cucurbituril;
- [31] FIG. 5C is a graph showing the effect of removing the odor of a japanese spanish mackerel using cucurbit[5]uril (CB[5]), cucurbit[6]uril (CB[6]), cucurbit[7]uril (CB[7]), cucurbit[8]uril (CB[8]) and crude cucurbit[7]uril (crude (CB[7]));
- [32] FIG. 6A is a graph showing the effect of removing the odor of a fermented skate using cucurbituril;
- [33] FIG. 6B is a graph showing the effect of removing the odor of a fermented skate using beta-hydroxypropyl cyclodextrin (HY CD, deodorizer) and cucurbituril; and
- [34] FIG. 6C is a graph showing the effect of removing the odor of a fermented skate using cucurbit[5]uril (CB[5]), cucurbit[6]uril (CB[6]), cucurbit[7]uril (CB[7]), cucurbit[8]uril (CB[8]) and crude cucurbit[7]uril (crude (CB[7])).

Best Mode for Carrying out the Invention

- [35] Hereinafter, preferred embodiments of the present invention will be described in detail with reference to the attached drawings.
- [36] The present invention provides a composition for removing odor, the composition including a compound represented by Chemical Formula 1 below:

[37] [Chemical Formula 1]



- [39] wherein n is an integer of 4 to 20, and X is O, S or NH, and
- [40] wherein the composition includes 45 to 100 wt% of the compound of Chemical Formula 1 of n = 7.

[41] The compound of Chemical Formula 1 is cucurbituril.

[42] According to the experiment of the present inventors (refer to Example 1), since the cavity of cucurbit[7]uril, which is the compound of Chemical Formula 1 of $n = 7$, is most easily bonded with a fragrant material, cucurbit[7]uril is most advantageous in terms of removing an odor. In this case, it is preferred that the ratio of the amount of cucurbit[7]uril to the total amount of cucurbituril be high. However, in order to increase the amount of cucurbit[7]uril at the time of synthesizing cucurbit[7]uril, a process of purifying the synthesized cucurbit[7]uril (crude cucurbit[7]uril) is additionally required, thus decreasing economical efficiency. Therefore, the synthesized crude cucurbit[7]uril itself can be used to remove an odor, and, in this case, the amount of cucurbit[7]uril in the crude cucurbit[7]uril may be 45% to 100% based on the total amount of cucurbituril. The composition may further include cucurbiturils of Chemical Formula 1 of $n = 4$ to 6 and 8 to 20 in an amount of 0 ~ 35%. Among the cucurbiturils of Chemical Formula 1 of $n = 4$ to 6 and 8 to 20, cucurbit[6]uril is mostly used. Besides, the composition may further include cucurbiturils of Chemical Formula 1 of $n = 4, 5$ and 8 to 20 in an amount of 0 ~ 5%.

[43] The crude cucurbit[7]uril is synthesized by a method including the following steps. However, the synthesis method thereof is not limited thereto.

[44] 1) Glycoluril 1.05 M (150g) and paraformaldehyde 2.2 M (67g) are mixed with a sufficient amount of HCl (225mL), and this mixture is heated to 85 ~ 90°C for 18 hours to obtain a cucurbituril solution.

[45] 2) H₂O (3L) is added to the cucurbituril solution, and then the solution is filtered to obtain a filtrate, and then the filtrate is mixed with methanol (2.4L) to obtain a solid-containing solution.

[46] 3) The solid-containing solution is filtered by a filter paper to obtain a solid compound, and then the solid compound is washed with methanol (2L) and dried to obtain crude cucurbit[7]uril.

[47] According to another embodiment of the present invention, the cucurbituril may be dissolved in a buffer. Examples of the buffer may include, but are not limited to, PBS (phosphate buffered saline), Tris-HCl, Tricine, HEPES (4-(2-hydroxyethyl)-1-piperazineethanesulfonic acid), MOPS (3-(N-morpholino)propanesulfonic acid), SSC (saline sodium citrate), and MES (2-(N-morpholino)ethanesulfonic acid).

[48] The composition of the present invention may further include an additive. In this case, examples of the additive may include, but are not limited to, fragrances such as ethyl butyrate, camphor, menthol, hexanal, eugenol, citronellol, linalool, patchoulol, santalol, phenyl ethyl alcohol, terpineol, vanillin, benzaldehyde, methyl salicylate, isoamyl acetate, d-limonene, L-limonene, α -pinene, 2-hydroxy-p-cymene, methyl ace-

tophenone, and cis-jasmone; and/or solvents such as cyclohexane, hexane, heptane, isooctane, isopentane, pentane, dimethyl ether, diethyl ether, methanol, ethanol, propanol, isopropanol, butanol, acetone and propellants.

[49] The composition of the present invention is very effective at capturing an odor molecule having an amino group or a carboxyl group, but can effectively capture other odor molecules. The odor which can be removed by the composition of the present invention may be emitted from an odor emission material such as onion, garlic, pork, fish, fermented fish, mold, bacteria, body fluids, leather, sewage, industrial wastewater or the like. However, the kinds of the odor emission material are not limited thereto.

[50] The composition of the present invention can remove an odor from an odor emission material by 30% or more, which was measured by an odor measuring device.

[51] Cucurbituril, which is a macrocyclic compound, has a hydrophobic cavity, and has hydrophilic inlets at both sides thereof. Therefore, hydrophobic interaction occurs in the cavity of cucurbituril, and hydrogen bonding, polar interaction, cation-polar interaction and the like occur at the upper and lower inlets thereof at which six carbonyl groups are located, so cucurbituril exhibits inclusion effects using extremely stable noncovalent binding with various kinds of compounds. Particularly, cucurbituril is formed into a complex by extremely stable noncovalent binding with compounds having a functional group such as an amino group, a carboxyl group or the like. Thanks to it having such characteristics, cucurbituril can effectively capture various kinds of odor molecules, and thus cucurbituril can be used a deodorizer which is superior compared to conventional deodorizers.

[52] In Example 1, an odor removing effect was evaluated using a mixture of cucurbituril homologues, and in Examples 2 and 3, the odor removing effect of each cucurbituril was compared to that of a commercially available deodorizer.

[53] The odor removing effects in Examples may be different from each other according to the amount of an odor material, the kind of the odor material and the used portion of the odor material (for example, the degree of odor of onion is different with respect to each portion). The values (Y axis in graph) measured by an odor measuring device used in the experiment are relative values indicating the degree of strength and weakness of odor, so it is difficult to indicate these values as absolute values. Therefore, the comparison of the odor removing effect of each cucurbituril or each deodorizer may be regarded independently.

[54]

[55] Hereinafter, the present invention will be described in more detail with reference to the following Examples. However, these Examples are set forth to illustrate the present invention, and the scope of the present invention is not limited thereto.

[56]

[57] [Examples]

[58] **Example 1: Preparation of odor removing composition including cucurbituril and evaluation of odor removing effect thereof**

[59] Cucurbit[6]uril (CB[6]) and a mixture of cucurbituril homologues (CB[n], composition ratio: CB[5]:CB[6]:CB[7]:CB[8]= 12.5%: 52.0%: 25.6%: 9.4%) were respectively dissolved in a buffer (PBS, pH 7.2) to obtain a solution (1 w/v %). The solution was sprayed onto odor emission materials such as onion, garlic, pork (pork belly), fish (japanese spanish mackerel), fermented fish (sliced raw skate) and the like to such a degree that each of the odor emission materials was sufficiently impregnated with the solution. After 1 hour, the degree of odor emission was measured using an odor measuring device (COSMOS, XP-329 m). The degree of reduction of odor emission was evaluated by the values measured by the odor measuring device on the basis of that obtained when only PBS buffer was sprayed. The average and standard deviation of the values obtained by testing the same sample three times were calculated and charted. The results thereof are shown in FIGS. 2A, 3A, 4A, 5A and 6A.

[60]

[61] **Example 2: Preparation of odor removing composition including cucurbituril and β -Hydroxypropyl Cyclodextrin and evaluation of odor removing effect thereof**

[62] In order to compare the odor removing effect of each cucurbituril homologue with that of β -hydroxypropyl cyclodextrin, which is generally used as a deodorizer for textile materials, the following experiment was carried out.

[63] Cucurbit[6]uril (CB[6]), cucurbit[7]uril (CB[7]) and β -hydroxypropyl cyclodextrin (Hy CD) were respectively dissolved in a buffer (PBS, pH 7.2) to obtain a solution (1 w/v %). 1 mL of the solution was sprayed onto odor emission materials such as onion, garlic, pork (pork belly), fish (japanese spanish mackerel), fermented fish (sliced raw skate) and the like. Each of the odor emission materials sprayed with the solution was shaken at a rotation speed of 60 rpm using a shaker machine (N-BIOTEK, ORBITAL SHAKER NB-101S) in order to uniformly disperse the solution, and the degree of odor emission was measured using an odor measuring device (COSMOS, XP-329 m). The results thereof are shown in FIGS. 2B, 3B, 4B, 5B and 6B.

[64]

[65] **Example 3: Preparation of odor removing composition according to the kinds of cucurbituril homologues and evaluation of odor removing effect thereof**

[66] In order to compare the odor removing effects of cucurbituril homologues with each other, the following experiment was carried out.

[67] Cucurbit[5]uril (CB[5]), cucurbit[6]uril (CB[6]), cucurbit[7]uril (CB[7]), cucurbit[8]uril (CB[8]) and crude cucurbit[7]uril ((crude CB[7]), a complex having a

composition ratio of CB7:CB6 = 2:1) were respectively dissolved in a buffer (PBS, pH 7.2) to obtain a solution (1 w/v %). 1 mL of the solution was sprayed onto odor emission materials such as onion, garlic, pork (pork belly), fish (japanese spanish mackerel), fermented fish (sliced raw skate) and the like. Each of the odor emission materials sprayed with the solution was shaken at a rotation speed of 60 rpm using a shaker machine (N-BIOTEK, ORBITAL SHAKER NB-101S) in order to uniformly disperse the solution, and the degree of odor emission was measured using an odor measuring device (COSMOS, XP-329 m). The results thereof are shown in FIGS. 2C, 3C, 4C, 5C and 6C.

[68]

[69] Although the preferred embodiments of the present invention have been disclosed for illustrative purposes, those skilled in the art will appreciate that various modifications, additions and substitutions are possible, without departing from the scope and spirit of the invention as disclosed in the accompanying claims.

[70]

Industrial Applicability

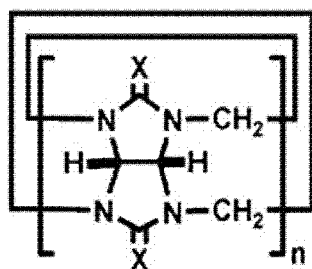
[71]

The composition can capture and remove various kinds of odor materials (particularly, ammonium ion-containing bad odor, such as a fishy smell or the like) because cucurbituril can interact with various odor materials using its hydrophobic cavity and both inlets covered with carbonyl groups. Therefore, this composition can be practically applied to the cleaning of air and the improvement of an environment.

Claims

[Claim 1] A composition for removing odor, the composition comprising a compound represented by Chemical Formula 1 below:

[Chemical Formula 1]



wherein, n is an integer of 4 to 20, and X is O, S or NH, and wherein the composition comprises 45 to 100 wt% of the compound of Chemical Formula 1 of n = 7.

[Claim 2] The composition of claim 1, wherein the compound of Chemical Formula 1 is dissolved in a buffer.

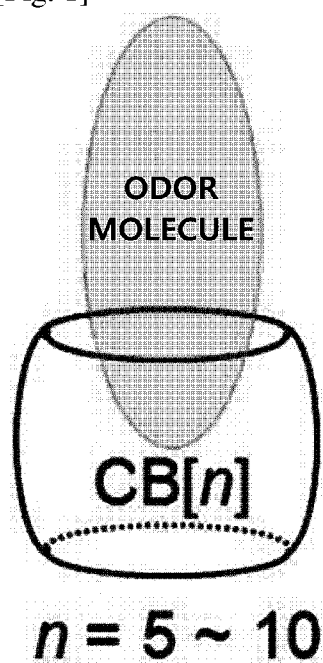
[Claim 3] The composition of claim 2, wherein the buffer is selected from the group consisting of PBS (phosphate buffered saline), Tris-HCl, Tricine, HEPES (4-(2-hydroxyethyl)-1-piperazineethanesulfonic acid), MOPS (3-(N-morpholino)propanesulfonic acid), SSC (saline sodium citrate), and MES (2-(N-morpholino)ethanesulfonic acid).

[Claim 4] The composition of claim 1, further comprising at least one additive selected from the group consisting of ethyl butyrate, camphor, menthol, hexanal, eugenol, citronellol, linalool, patchoulol, santalol, phenyl ethyl alcohol, terpineol, vanillin, benzaldehyde, methyl salicylate, isoamyl acetate, d-limonene, L-limonene, α -pinene, 2-hydroxy-p-cymene, methyl acetophenone, cis-jasmone, cyclohexane, hexane, heptane, isooctane, isopentane, pentane, dimethyl ether, diethyl ether, methanol, ethanol, propanol, isopropanol, butanol, acetone and propellants.

[Claim 5] The composition of claim 1, wherein the composition captures an odor molecule having an amino group or a carboxyl group.

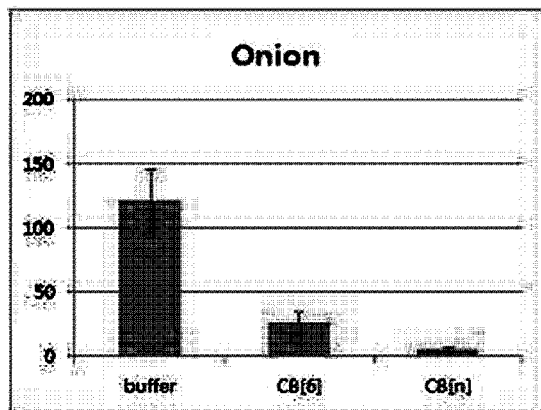
[Claim 6] The composition of claim 1, wherein the odor is emitted from an odor emission material selected from onion, garlic, pork, fish, fermented fish, mold, bacteria, body fluids, leather, sewage, and industrial wastewater.

[Fig. 1]

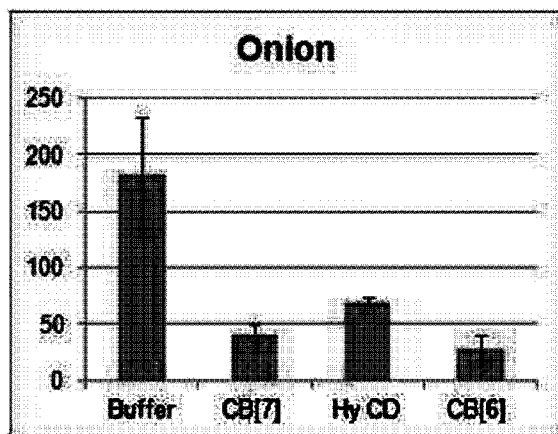


[Fig. 2]

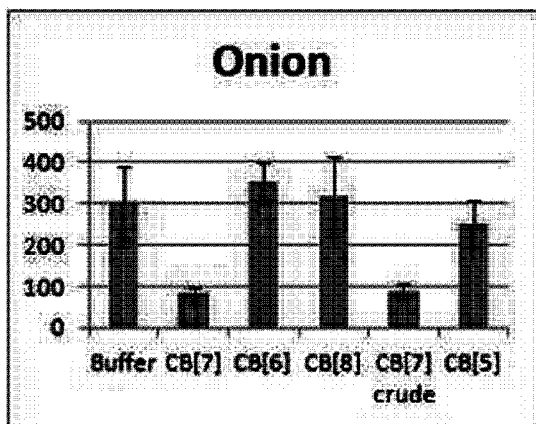
(A)



(B)

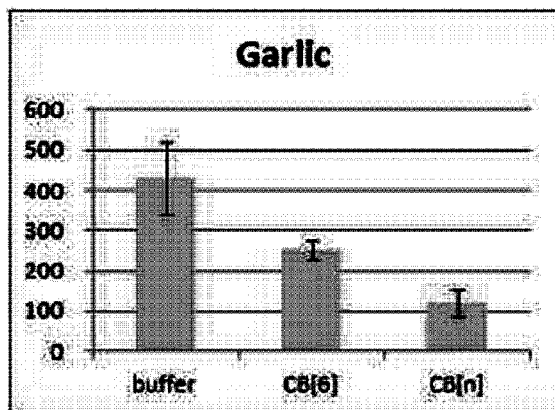


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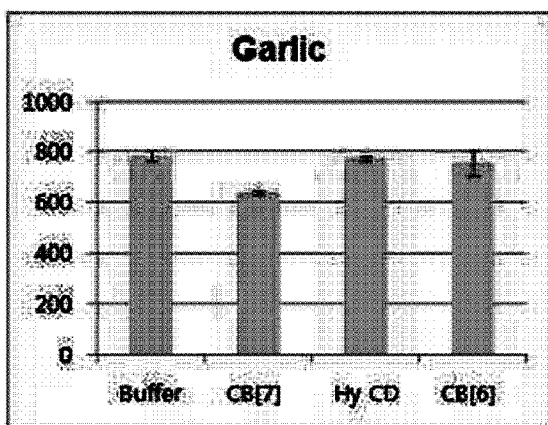


[Fig. 3]

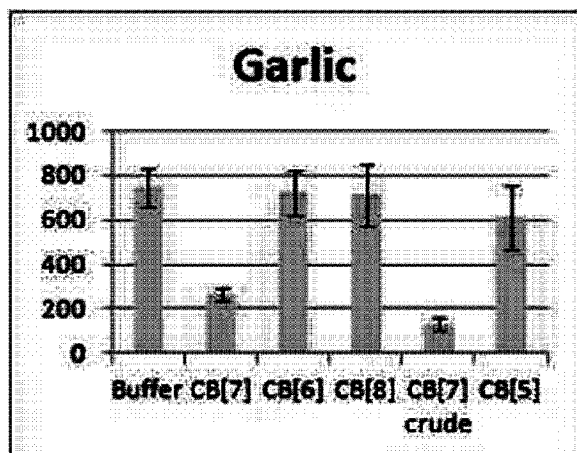
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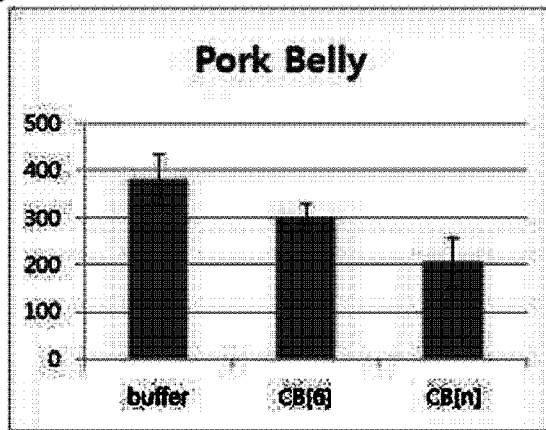


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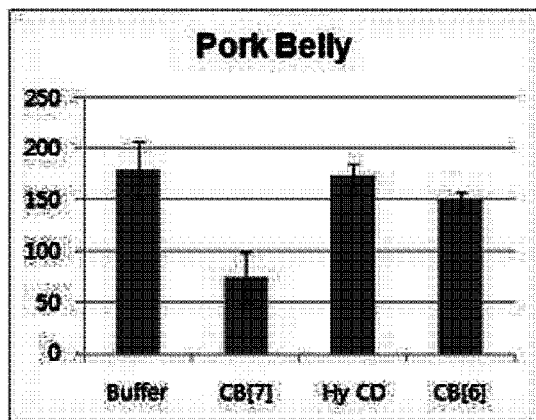


[Fig. 4]

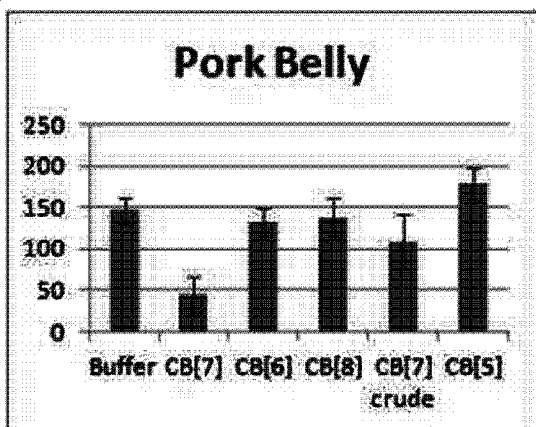
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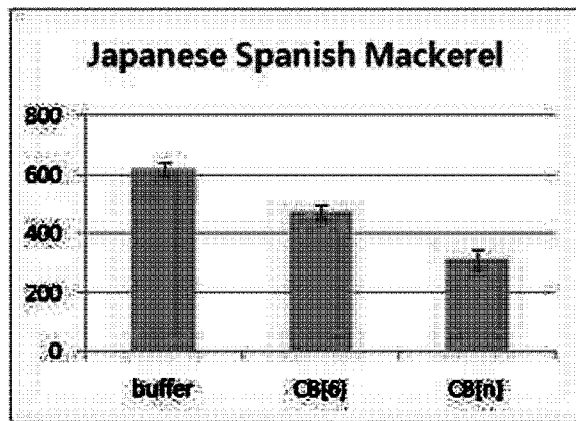


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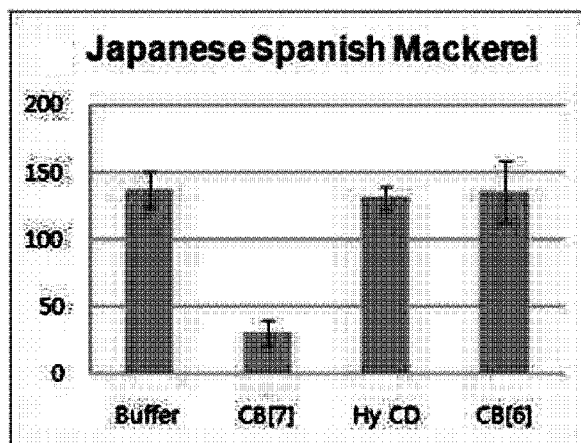


[Fig. 5]

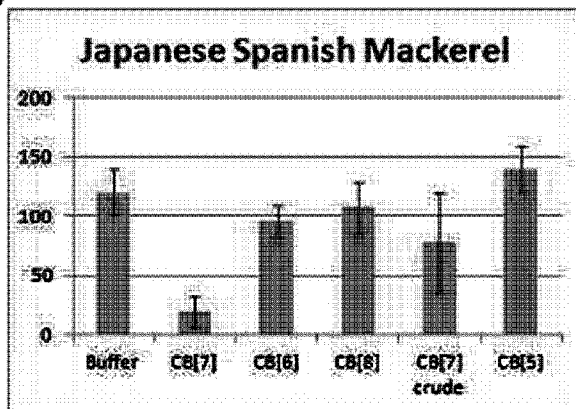
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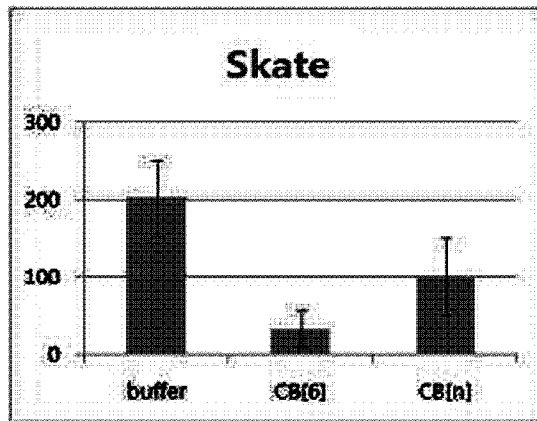


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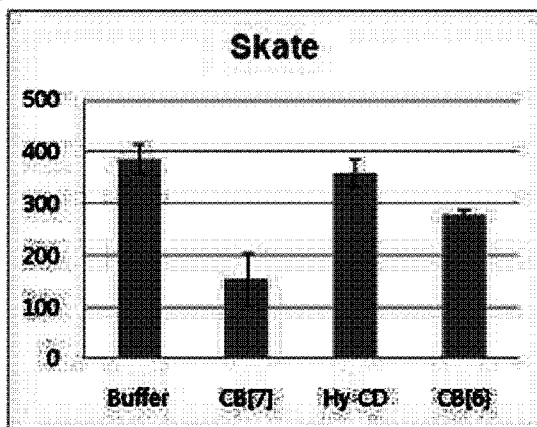


[Fig. 6]

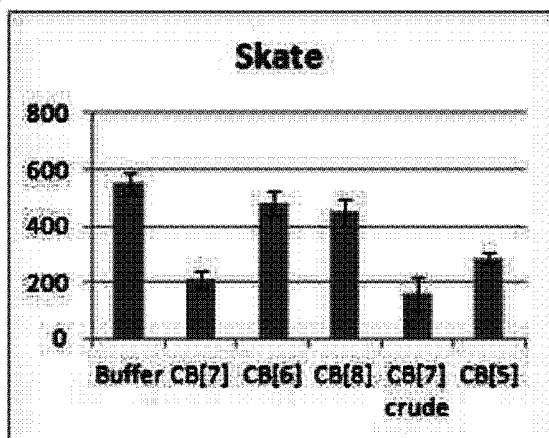
(A)



(B)



(C)



INTERNATIONAL SEARCH REPORT

International application No.
PCT/KR2013/010436**A. CLASSIFICATION OF SUBJECT MATTER****A61L 9/01(2006.01)i, A61L 9/04(2006.01)i**

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

A61L 9/01; A61K 9/14; A61K 38/28; B01D 53/48; C07D 487/22; C09K 3/00; B01D 53/14; A61L 9/04

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Korean utility models and applications for utility models

Japanese utility models and applications for utility models

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

eKOMPASS(KIPO internal) & keywords: cucurbitril, ordor, fragrance, buffer

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	KIM et al., `Functionalized cucurbiturils and their applications` Chemical Society Reviews, 07 November 2006, Vol. 36, pp. 267-279 See abstract; page 269, lines 1-9, page 278, lines 18-24.	1-6
A	US 2007-0154553 A1 (KIM et al.) 05 July 2007 See abstract; claims 1-4.	1-6
A	US 2002-0133003 A1 (KIM et al.) 19 September 2002 See abstract; claim 1.	1-6
A	US 2010-0028232 A1 (BEDELL) 04 February 2010 See abstract; claims 1-14.	1-6
A	US 2003-0140787 A1 (DAY et al.) 31 July 2003 See abstract; paragraphs [0032]-[0033]; claims 1-24.	1-6



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:

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"E" earlier application or patent but published on or after the international filing date

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

07 February 2014 (07.02.2014)

Date of mailing of the international search report

07 February 2014 (07.02.2014)

Name and mailing address of the ISA/KR

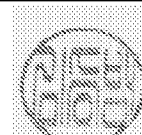
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/KR2013/010436

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 2007-0154553 A1	05/07/2007	KR 10-0750320 B1 KR 10-2007-0073225 A US 7850992 B2	17/08/2007 10/07/2007 14/12/2010
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