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(81) **Designated States** *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

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Published:

— *with international search report (Art. 21(3))*

— *with amended claims (Art. 19(1))*

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(54) **Title**: COLLAGEN MIXTURE AND METHOD OF MAKING THE SAME

(57) **Abstract**: A collagen mixture includes a portion of unhydrolyzed eggshell membrane collagen and a portion of Avian collagen. The method of making the eggshell membrane collagen includes separating a membrane from an eggshell, drying the membrane, and pulverizing the membrane into a powder. The method of making the Avian collagen includes adding an enzyme mixture to a thawed Avian cartilage to break the cartilage down into liquid form, heating the liquid cartilage to form a layer of liquid digest, raising the pH level of the liquid digest, heating the raised pH level liquid digest to form a partial solid content, and spray drying the partial solid content.

TITLE: COLLAGEN MIXTURE AND METHOD OF MAKING THE SAME

BACKGROUND OF THE INVENTION

This invention is directed to creating a collagen mixture and more specifically, without limitation, creating a collagen mixture of unhydrolyzed eggshell membrane with LOS with Avian collagen.

Methods for producing collagen are known in the art. Such collagen is used for many things such as the healing of wounds, the production of skin creams and shampoo, the treatment of osteoarthritis and osteoporosis, and as an additive for human and pet food. While useful, the process is expensive, complicated, involving many steps, and requires harsh chemicals and inorganics which can damage unique properties. Accordingly, a method and mixture is needed in the art that addresses these deficiencies.

An objective of the present invention is to combine Type I, V, and X collagen with Type II collagen.

Another objective of the present invention is to provide a method of making unhydrolyzed eggshell membrane collagen with LOS that involves fewer steps and is undenatured.

A still further objective of the present invention is to provide a method of producing Avian collagen Type II with a greater molecular weight.

These and other objectives will be apparent to one of ordinary skill in the art based upon the following written description, drawings, and claims.

SUMMARY OF THE INVENTION

A collagen mixture includes a portion of unhydrolyzed eggshell membrane collagen and a portion of Avian collagen. The method of making the eggshell membrane collagen includes separating a membrane from an eggshell, drying the membrane, and pulverizing the membrane into a powder. The method of making the Avian collagen includes adding an enzyme mixture to a thawed Avian cartilage to break the cartilage down into liquid form, heating the liquid cartilage to form a layer of liquid digest, raising the pH level of the liquid digest, heating the raised pH level liquid digest to form a partial solid content, and spray drying the partial solid content.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a flow diagram of a method for making unhydrolyzed eggshell membrane collagen;

Fig. 2 is a schematic diagram of the environment for making unhydrolyzed eggshell membrane collagen;

Fig. 3 is a flow diagram for a method of making a collagen mixture; and

Fig. 4 is a schematic diagram of the environment for making a collagen mixture.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

Referring to the Figures, the process of making unhydrolyzed eggshell membrane collagen containing Type I, V, and X collagen and lysosyme, ovatransferin, and sialic acid (LOS) begins at step 100 by obtaining sanitized cracked eggshells 10. The cracked eggshells 10 are placed on a sifter 12 at step 110 where the membrane 14 of the eggshell 10 is separated from the shell 10. The sifter 12 is of any type and preferably includes a fine mesh through which the shell 10 falls while the membrane 14 is transported to the end of the sifter 12.

Once separated, the membrane 14 is placed in a hot dryer 16 to dry the membrane and kill bacteria at step 120. While the dryer 16 is of any type, a motion dryer is preferred to facilitate faster drying. Once dried, the membrane 14, which is in flake form, is pulverized into a powder at step 130. Once in powder form, the membrane is packaged preferably in a fiber drum or bag for further use and/or processing.

The process of making the Avian collagen with LOS begins by obtaining Avian cartilage 18, typically in frozen form at step 200. At step 210, the frozen cartilage 18 is thawed by placing the cartilage in a tank 20 containing hot water. Once thawed, an enzyme mixture 22 is added to the thawed cartilage 18 which breaks the cartilage down into liquid form at step 220. Preferably, the enzyme mixture 22 includes a liquid plant derived enzyme 24 such as papain, fien, or bromalin; a bacterial protolitic enzyme 26, such as acid, neutral, or alkelian; and a buffer 28 such as acetic acid or sodium hydroxide and water for a pH buffering range of 4.8 to 6.8. As the enzyme mixture 22 breaks down the cartilage 18, the tank is heated to preferably 140° - 160°F such that the cartilage 18 separates into a top layer of fat 30, a middle layer of liquid digest 32, and a bottom layer of bone chips 34.

Once separated, at step 230, the bone chips 34 are swept away from a valve 36 in the bottom of the tank 20 and the liquid digest 32 is pumped through the valve 36 to a holding

tank 38. Once pumped, at step 240, the pH level of the liquid digest 32 is raised by adding sodium hydroxide or potassium hydroxide 40 to the liquid digest 32 in the holding tank 38. Preferably the pH level of the liquid digest 32 is raised to a level of 8 to 11.

At step 250, after the pH level is raised, the liquid digest 32 is pumped into an evaporator 42 where the liquid digest 32 is heated to a solid content of between 10% to 75% such that the liquid has a syrup consistency. Preferably the liquid digest is heated at a temperature of 200° - 230°F to create a solid content of 35-40%.

Next, at step 260, the solid liquid digest 32 is transported to an area for spray drying 44. Preferably, the solid liquid digest is spray dried using pulse combustion. Also, a carrier such as maltodextrin is added to assist in pulling off extra water from the liquid digest 32. This results in a dry powder comprised of Avian collagen.

Once the Avian collagen has been spray dried it is mixed with the unhydrolyzed eggshell membrane collagen at step 270 to create a blended collagen mixture that includes Type I, II, III, IV, V and X collagen and LOS. Preferably the mixture has 70% Avian collagen to 30% eggshell membrane or alternatively 30% Avian collagen to 70% eggshell membrane collagen. Once combined, at step 280, the mixture is packaged and distributed.

Accordingly, a collagen mixture and method of making the same has been disclosed that, at the very least, meets all the stated objectives.

What is claimed is:

1. A method of making collagen comprising the steps of: obtaining sanitized cracked eggshells; separating a membrane from the eggshell; drying the membrane; pulverizing the dried membrane to form a powder.

2. The method of claim 1 wherein the membrane is separated from the eggshell using a sifter.

3. The method of claim 1 wherein the membrane is dried using a motion dryer.

4. A method of making collagen, comprising the steps of: obtaining Avian cartilage in frozen form; thawing the frozen cartilage; adding an enzyme mixture to the thawed cartilage to break the cartilage down into liquid form; heating the liquid cartilage to form a layer of liquid digest; raising the pH level of the liquid digest; heating the raised pH level liquid digest to form a partial solid content; and spray drying the heated partial solid content.

5. The method of claim 4 wherein the enzyme mixture includes a liquid plant derived enzyme, a bacterial protolitic enzyme, and a buffer.

6. The method of claim 4 wherein the pH level of the liquid digest is raised 8 to 11 pH.

7. The method of claim 4 wherein the heated partial solid content is spray dried using pulse combustion.

8. A collagen mixture comprising: a portion of unhydrolyzed eggshell membrane collagen; and a portion of Avian collagen.

AMENDED CLAIMS
received by the International Bureau on 03 January 2013 (03.01.2013)

What is claimed is:

1. A method of making collagen comprising the steps of: obtaining sanitized cracked eggshells; separating a membrane from the eggshell using a sifter having a fine mesh through which the eggshells fall while retaining the membrane; drying the membrane after the membrane is separated from the eggshell; and pulverizing the dried membrane to form a powder.

2. The method of claim 1 wherein the membrane is dried using a motion dryer.

3. A method of making Type II collagen, comprising the steps of: obtaining Avian cartilage in frozen form; thawing the frozen cartilage; adding an enzyme mixture comprising a plant derived proteolytic enzyme, a bacterial proteolytic enzyme and a buffer to the thawed cartilage to break the cartilage down into liquid form; heating the liquid cartilage to form a layer of liquid digest; raising the pH level of the liquid digest; heating the raised pH level liquid digest to 5-11 to form a partial solid content; and spray drying the heated partial solid content.

4. The method of claim 3 wherein the enzyme mixture includes a liquid plant derived enzyme, a bacterial protolitic enzyme, and a buffer.

5. The method of claim 3 wherein the pH level of the liquid digest is raised 8 to 11 pH.

6. The method of claim 3 wherein the heated partial solid content is spray dried using pulse combustion.

7. A collagen mixture comprising: a portion of unhydrolyzed eggshell membrane collagen; and a portion of Avian collagen.

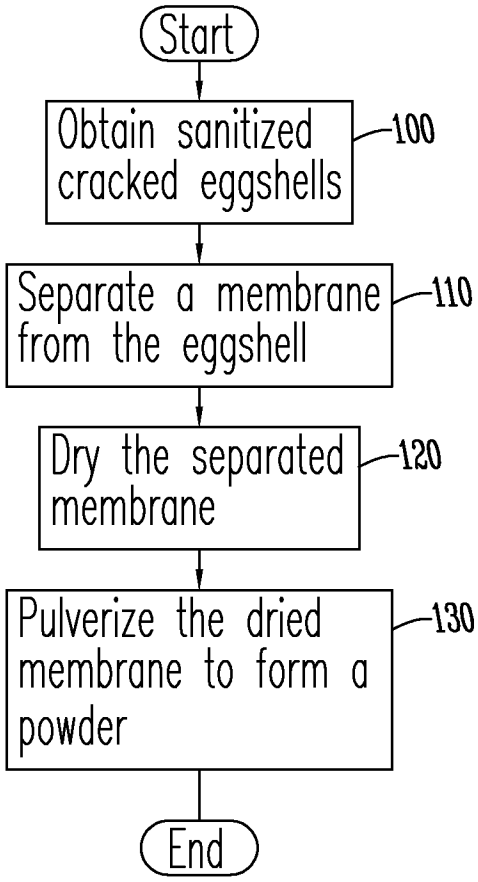

Start
Obtain sanitized cracked eggshells
100
Separate a membrane from the eggshell
110
Dry the separated membrane
120
Pulverize the dried membrane to form a powder
130
End

Fig. 1

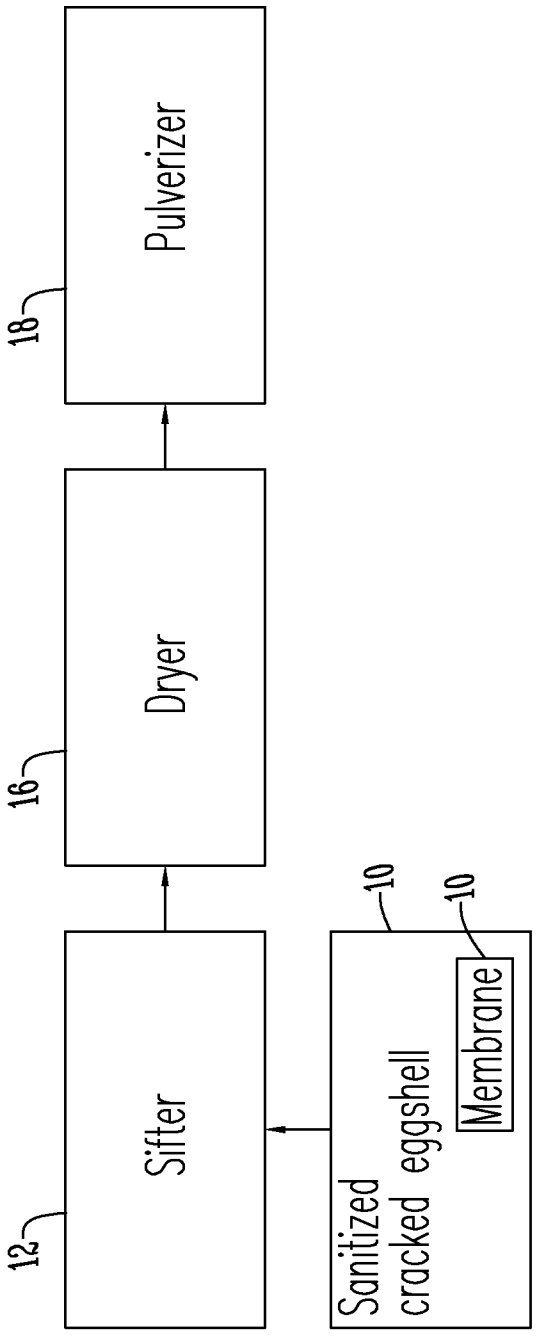

Pulverizer
18
Dryer
16
Sifter
12
Sanitized cracked eggshell
10
Membrane
10

Fig. 2

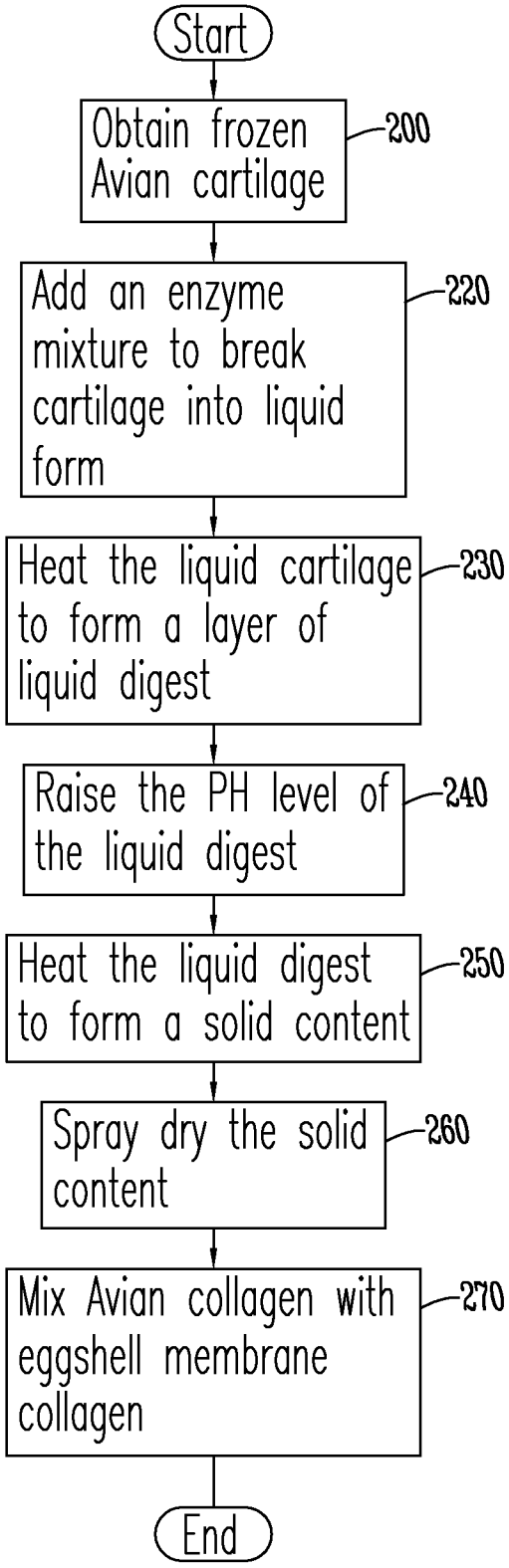

Start
Obtain frozen Avian cartilage
200
Add an enzyme mixture to break cartilage into liquid form
220
Heat the liquid cartilage to form a layer of liquid digest
230
Raise the PH level of the liquid digest
240
Heat the liquid digest to form a solid content
250
Spray dry the solid content
260
Mix Avian collagen with eggshell membrane collagen
270
End

Fig. 3

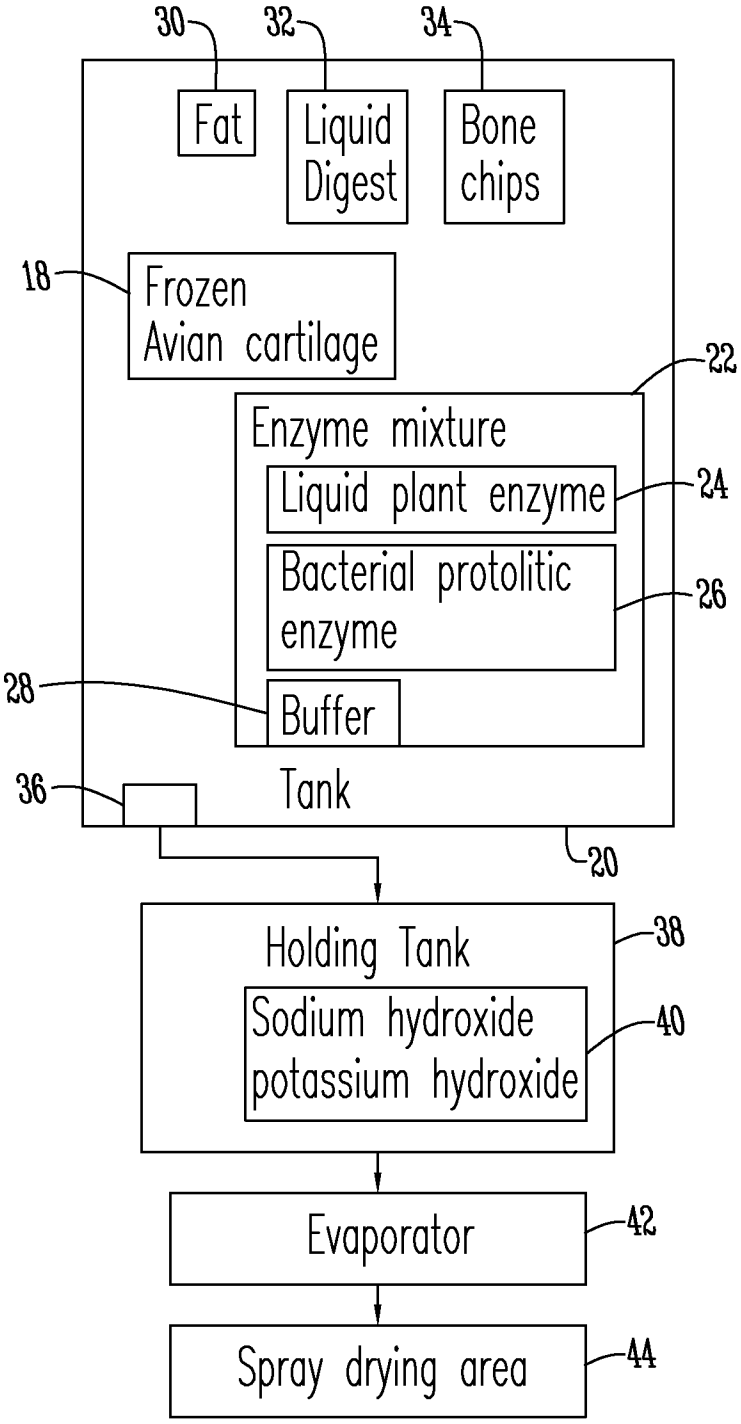

30
32
34
Fat
Liquid Digest
Bone chips
18
Frozen Avian cartilage
22
Enzyme mixture
Liquid plant enzyme
24
Bacterial protolitic enzyme
26
28
Buffer
36
Tank
20
Holding Tank
38
Sodium hydroxide potassium hydroxide
40
Evaporator
42
Spray drying area
44

Fig. 4

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US 12/49545

A. CLASSIFICATION OF SUBJECT MATTER
IPC(8) - A61K 38/39 (2012.01)
USPC - 530/356
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)
IPC(8)- A61K 38/39 (2012.01);
USPC- 530/356

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
Patents and NPL (classification, keyword; search terms below)

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
PubWest, PatBase (USPTO, EPO, JPO, WIPO), GoogleScholar (PL, NPL), FreePatentsOnline (USPTO, EPO, JPO, WIPO, NPL); search terms: egg, eggshell, collagen, dry, powder, pulverize, granulate, particle, cartilage, thaw, hydrolyze, hydrolysis, digest, spray, dry, pulse, combustion, aviary, chicken, poultry

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2003/0209617 A1 (MACNEIL) 13 November 2003 (13.11.2003), Fig. 2; para [0019], [0021], [0025], [0026], [0031], [0038], [0051], [0055]	1-3
X	US 2011/0033440 A1 (STROHBEHN et al.) 10 February 2011 (10.02.2011), Figs. 1, 2; para [0032], [0043], [0044]	8
Y	US 2002/0012714 A1 (OLSON) 31 January 2002 (31.01.2002), para [0007], [0014], [0024]	4-7
Y	US 2006/0045920 A1 (ISHAQ) 02 March 2006 (02.03.2006), para [0017], [0020], [0029]	4-7
Y	US 6,028,118 A (DUPONT et al.) 22 February 2000 (22.02.2000), col 3, ln 54 to col 4, ln 4; col 7, ln 9-39	4-7
Y	US 2006/0188892 A1 (LATHAM et al.) 24 August 2006 (24.08.2006), para [0010], [0011], [0054]	5
Y	US 2009/0061002 A1 (VENBROCKS et al.) 05 March 2009 (05.03.2009), para [0005], [0092], [0101]	7
Y	US 2010/0175570 A1 (VLAD) 15 July 2010 (15.07.2010), para [0028]-[0052]	1-3, 8
Y	US 2009/0324777 A1 (WILLIAMS et al.) 31 December 2009 (31.12.2009), para [0052]	4-7
Y	US 2004/0037813 A1 (SIMPSON et al.) 26 February 2004 (26.02.2004), para [0007], [0077], [0190]	4-7

☒ Further documents are listed in the continuation of Box C. ☐

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier application or patent but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"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	Date of mailing of the international search report
16 November 2012 (16.11.2012)	07 DEC 2012
Name and mailing address of the ISA/US Mail Stop PCT, Attn: ISA/US, Commissioner for Patents P.O. Box 1450, Alexandria, Virginia 22313-1450 Facsimile No. 571-273-3201	Authorized officer: Lee W. Young PCT Helpdesk: 571-272-4300 PCT OSP: 571-272-7774

Form PCT/ISA/210 (second sheet) (July 2009)

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US 12/49545

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:
This application contains the following inventions or groups of inventions which are not so linked as to form a single general inventive concept under PCT Rule 13.1. In order for all inventions to be examined, the appropriate additional examination fees must be paid.

Group I: claims 1-3 and 8: directed to a method of making a collagen comprising the steps of: obtaining sanitized cracked eggshells; separating a membrane from the eggshell; drying the membrane; and pulverizing the dried membrane to form a powder.

Group II: claims 4-8: directed to a method of making collagen, comprising the steps of: obtaining frozen Avian cartilage; thawing it; treating it with enzymes to liquify the cartilage; adjusting the pH of the liquified cartilage; and spray drying it.

-- Please see Extra Sheet --

1. ☒ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.

2. ☐ As all searchable claims could be searched without effort justifying additional fees, this Authority did not invite payment of additional fees.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

☐ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.

☐ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.

☒ No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US 12/49545

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 6,649,203 B1 (THOROSKI) 18 November 2003 (18.11.2003), col 3-11	1-3, 8
Y	ES 2 181 580 A1 (MONFERRER et al.) 16 February 2003 (16.02.2003), English Abstract only	1-3, 8

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US 12/49545

Box No. III. Observations where unity of invention is lacking:

This application contains the following inventions or groups of inventions which are not so linked as to form a single general inventive concept under PCT Rule 13.1. In order for all inventions to be examined, the appropriate additional examination fees must be paid.

Group I: claims 1-3 and 8: directed to a method of making a collagen comprising the steps of: obtaining sanitized cracked eggshells; separating a membrane from the eggshell; drying the membrane; and pulverizing the dried membrane to form a powder.

Group II: claims 4-8: directed to a method of making collagen, comprising the steps of: obtaining frozen Avian cartilage; thawing it; treating it with enzymes to liquify the cartilage; adjusting the pH of the liquified cartilage; and spray drying it.

The inventions listed as Groups I and II do not relate to a single general inventive concept under PCT Rule 13.1 because, under PCT Rule 13.2, they lack the same or corresponding special technical features for the following reasons:

Group I does not include the inventive concept of making collagen from eggshells, as required by Group II.

Group II does not include the inventive concept of making collagen from frozen Avian cartilage, as required by Group I.

Groups I and II therefore lack unity under PCT Rule 13 because they do not share a same or corresponding special technical feature.