1. A process for the preparation of a refrigerated cooked product from a non-conventional starchy material containing substantially no gluten as hereinbefore
FORM 1
REGULATION 9

COMMONWEALTH OF AUSTRALIA

PATENTS ACT 1952

APPLICATION FOR A STANDARD PATENT

We, SOCIETE DES PRODUITS NESTLE S.A., a Swiss body corporate of Vevey, Switzerland, hereby apply for the grant of a Standard Patent for an invention entitled:

"PREPARATION OF REFRIGERATED PRODUCT"

which is described in the accompanying Complete Specification.

Details of basic application:-

Number: 07/271,045
Country: United States of America
Date: 14th November, 1988

Our address for service is: SHELSTON WATERS
55 Clarence Street
SYDNEY, N.S.W. 2000.

DATED this 20th Day of October, 1989
SOCIETE DES PRODUITS NESTLE S.A.

by

[Firm Signature]

Fellow Institution - Director Administration
of SHELSTON WATERS

To: The Commissioner of Patents
WODEN A.C.T. 2606
File: D.B. S-128
Fee: $164.00

SO10896 20/10/89
CONVENTION APPLICATION BY A COMPANY

FORM 8 – REGULATION 12 (2)

AUSTRALIA

PATENTS ACT 1952

DECLARATION IN SUPPORT OF A CONVENTION APPLICATION FOR A PATENT

(a) Here insert (in full) Name of Company.

(b) Here insert Title of Invention.

(c) and (d) Here insert Full Name and Address of Company Official authorized to make declaration.

SOCIETE DES PRODUITS NESTLE S.A.

"Preparation of refrigerated product"

En la Priauraz, 1807 BLONAY, Switzerland

Andrzej W. LEDZION

I do solemnly and sincerely declare as follows:

1. I am authorised by Applicant to make this declaration on its behalf.

2. The basic Application(s) as defined by section 141 of the Act was/were made in the United States on the 14th day of November 1988 by Jau Yann HSU, of 21 Flax Hill Road, BROOKFIELD, Connecticut 06804, U.S.A.

3. Jau Yann HSU, Eldon Chen-Hsiung LEE, Elaine Regina WEDRAL are/are the actual Inventor(s) of the invention and the facts upon which Applicant is entitled to make the Application are as follows:

the applicant is the assignee of the actual inventors of the invention subject of the application.

4. The basic Application(s) referred to in paragraph 2 of this Declaration was/were the first Application(s) made in a Convention country in respect of the invention, the subject of the Application.

DECLARED at Vevey, Switzerland 13th day of October, 1989

Preparation of Refrigerated Product
We have surprisingly found that the addition of certain functional proteins to the starchy material retards starch retrogradation on refrigerated storage of the cooked product.

The present invention relates to a process for the preparation of refrigerated cooked products e.g. pastas from non-conventional starchy materials containing substantially no gluten.

Examples of non-conventional starchy materials containing substantially no gluten include flours and starches derived from rice, maize and potatoes, reconstituted rice and similar starchy materials. The rice may be regular, parboiled, precooked, brown, long-grain, medium-grain, Basmati (from India), roasted wild rice and waxy (glutaneous/sweet) rice (from Japan and Taiwan).

By "functional protein" we mean an active protein which possesses its functional properties including binding capability, water absorption and protein-carbohydrate interaction which reduces starch retrogradation. The functional protein is preferably gluten, but other functional proteins may be used such as animal proteins e.g. egg albumen, gelatin or milk protein, plant proteins e.g. soy protein, and microbial proteins e.g. yeast protein.
1. A process for the preparation of a refrigerated cooked product from a non-conventional starchy material containing substantially no gluten as hereinbefore defined which comprises incorporating an effective amount of a functional protein into the uncooked starchy material, cooking the starchy material, and cooling to a refrigeration temperature.

5. A process according to claim 1 wherein the starchy material containing the functional protein is formed into a pasta before cooling.
COMMONWEALTH OF AUSTRALIA

FORM 10

PATENTS ACT 1952

COMPLETE SPECIFICATION

FOR OFFICE USE:

Class Int.Class

Application Number:  Lodged:

Complete Specification Lodged:
    Accepted:
    Published:

Priority:

Related Art:

Name of Applicant:  SOCIETE DES PRODUITS NESTLE S.A.

Address of Applicant:  Vevey, Switzerland

Actual Inventor:  Jau Yann Hsu, Eldon Chen-Hsiung Lee and Elaine Regina Wedral

Address for Service:  SHELSTON WATERS, 55 Clarence Street, Sydney

Complete Specification for the Invention entitled:

"PREPARATION OF REFRIGERATED PRODUCT"

The following statement is a full description of this invention, including the best method of performing it known to us:-

- 1 -
Preparation of Refrigerated Product

The present invention relates to a process for the preparation of refrigerated cooked products e.g. pastas from non-conventional starchy materials containing substantially no gluten.

There has recently been an increasing market demand for high quality culinary chilled food products. However, retrogradation has been a common problem in starch-dominant products, particularly on refrigeration storage and cooked rice used in chilled foods shows this problem which involves firming texture (staling) progressively on refrigeration.

When used in the preparation of pastas, non-conventional starchy materials such as rice, maize and potatoes which contain substantially no gluten, generally require a binder to form a good shape during extrusion and to prevent pasta stickiness during cooking. Binders that have been reported to be suitable for such purposes include gelatinised starch (British Patent No. 1384149), gums such as alginates and certain proteins. However, we have found that the addition of gelatinised starch or alginates to such starchy products does not retard starch retrogradation on refrigerated storage. Chemically modified starches such as certain organic acid derivatives (United States Patent No. 3953616) and certain emulsifiers have also been reported to have a lower staling effect. However, consumers generally prefer natural substances.

Although pasta formulations containing added proteins have been described in which the proteins are added for protein enrichment or as binding agents, there have been no reports on the use of added proteins to retard starch retrogradation in refrigerated cooked products. We have surprisingly found that the addition of certain...
functional proteins to the starchy material retards starch retrogradation on refrigerated storage of the cooked product.

Accordingly, the present invention provides a process for the preparation of a refrigerated cooked product from a non-conventional starchy material containing substantially no gluten as hereinafter defined which comprises incorporating an effective amount of a functional protein into the uncooked starchy material, cooking the starchy material and cooling to a refrigeration temperature.

By a non-conventional starchy material containing substantially no gluten we mean a starch or a flour containing substantially low levels of proteins and proteins of lower functionality. The starches contain substantially no gluten/proteins whereas the flours contain some gluten but in substantially low quantities and low functionality.

Examples of non-conventional starchy materials containing substantially no gluten include flours and starches derived from rice, maize and potatoes, reconstituted rice and similar starchy materials. The rice may be regular, parboiled, precooked, brown, long-grain, medium-grain, Basmati (from India), roasted wild rice and waxy (glutaneous/sweet) rice (from Japan and Taiwan).

By "functional protein" we mean an active protein which possesses its functional properties including binding capability, water absorption and protein-carbohydrate interaction which reduces starch retrogradation. The functional protein is preferably gluten, but other functional proteins may be used such as animal proteins e.g. egg albumen, gelatin or milk protein, plant proteins e.g. soy protein, and microbial proteins e.g. yeast protein. The amount of active protein added to the starchy material may be from 1 to 20% preferably from 2
to 10% and especially from 2.5 to 7.5% by weight based on the weight of the starchy material.

If desired, other additives such as alginate gums may be added to the starchy material.

The starchy material containing the functional protein may be formed into a pasta, for example by extrusion followed by cooking or extrusion/cooking, before cooling. The pastas may be formed by extrusion or by the dough kneading/sheeting process.

By refrigeration temperature we mean above 0°C to about 10°C, usually from about 2°C to about 8°C, and the present invention also comprises a refrigerated product prepared by the hereinbefore described process.

The following Examples further illustrate the present invention. Parts are given by weight.

Example 1

81 parts of ungelatinised rice flour and 13 parts of pregelatinised rice flour were mixed with 5 parts of wheat gluten, 0.5 parts of sodium alginate, 0.5 parts of propylene glycol alginate and 40 parts of water until uniform. The dough was then extruded through a low-pressure, pasta-type extruder to form a simulated rice grain shape. The rice pasta was cooked in boiling water for 3 minutes, drained, cooled and placed in a refrigerator at 5°C.

To measure the retrogradation after a period of storage in the refrigerator, a 15 g sample of the pasta was placed in a Kramer shear cell apparatus. The compressibility of the samples was performed with the following parameters:
The height of peaks was calculated to an equivalent weight load of 15 g sample. The percent of retrogradation after storage was expressed as the percent of the increasing weight load based on the initial sample before storage. The initial Instron Compressibility was 9 kg and after 18 days it was 13 kg which indicates a starch retrogradation of 44%.

**Comparative Example A**

Example 1 was repeated except that no gluten was added. The initial Instron Compressibility was 11 kg and after 18 days storage at 5°C was 19.4 kg which indicates a starch retrogradation of 76%.

In the following Examples the tests for retrogradation were carried out as follows:

To measure the retrogradation after a period of storage in the refrigerator at approximately 5°C, a 30g sample of the pasta was placed even in a Kramer shear cell apparatus. The Instron compressibility of the samples was performed with the following parameters: full scale load, 100 Kg; crosshead speed, 50mm/min; chart drive, 50mm/min. The percent of retrogradation after storage was expressed as the percent of the increasing weight load based on the initial sample before storage.

The thermal characterization for retrograded starch was performed by using differential scanning calorimetry (DSC). The pasta sample after a period of storage was ground to paste form, weighed and encapsulated in a stainless steel pan. The sample pan was scanned from 25 to 150°C at a rate of 10°C/min.
Examples 2 to 6

80 parts of uncooked long grain rice flour, 15 parts of pre-gelatinized long grain rice flour and 41 parts of water were mixed with 5 parts of wheat gluten, egg white, whole egg, sodium caseinate, or Torula yeast, in separate trials, until uniform, and extruded through a low-pressure, pasta type extruder to form a desired pasta shape. The pasta was cooked in boiling water, then cooled and packed under a controlled atmosphere using a gas mixture of carbon dioxide and nitrogen to extend its shelf-life. The results of the retrogradation tests are given in Table 1.

Comparative Example B

A similar procedure to that described for Examples 2 to 6 was carried out except that no functional protein was added. The results of the retrogradation tests are given in Table 1.

TABLE 1

I. Instron Compressibility:
Kg/30 g pasta sample in a Kramer shear cell

<table>
<thead>
<tr>
<th>Example</th>
<th>Description</th>
<th>Days at 5°C</th>
<th>Retrogradation, %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Initial</td>
<td>9 days</td>
</tr>
<tr>
<td>B</td>
<td>Control (none)</td>
<td>14.2</td>
<td>31.0</td>
</tr>
<tr>
<td>2</td>
<td>Wheat Gluten</td>
<td>9.8</td>
<td>12.0</td>
</tr>
<tr>
<td>3</td>
<td>Egg White</td>
<td>15.4</td>
<td>25.0</td>
</tr>
<tr>
<td>4</td>
<td>Whole Egg</td>
<td>11.0</td>
<td>21.0</td>
</tr>
<tr>
<td>5</td>
<td>Caseinate</td>
<td>10.8</td>
<td>16.0</td>
</tr>
<tr>
<td>6</td>
<td>Torula Yeast</td>
<td>12.4</td>
<td>23.0</td>
</tr>
</tbody>
</table>
II. DSC Analysis: 12 days at 5°C

<table>
<thead>
<tr>
<th>Example</th>
<th>Description</th>
<th>Peak Temperature 50-55°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>3.65</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Wheat Gluten</td>
<td>1.51</td>
</tr>
<tr>
<td>3</td>
<td>Egg White</td>
<td>1.90</td>
</tr>
<tr>
<td>4</td>
<td>Whole Egg</td>
<td>2.50</td>
</tr>
<tr>
<td>5</td>
<td>Caseinate</td>
<td>2.47</td>
</tr>
<tr>
<td>6</td>
<td>Torula Yeast</td>
<td>---</td>
</tr>
</tbody>
</table>

As shown in the TABLE 1, the addition of functional proteins, wheat gluten, egg white, whole egg, sodium caseinate, and Torula yeast showed less retrogradation than the comparative control with no added protein. Wheat gluten demonstrated the most effective protein for minimizing starch retrogradation with stable low Instron compressibility and low enthalpy (H) of retrograded starch. The endotherm of retrograded starch showed the peak maximum at approximately 50-55°C.

Examples 7 to 11

55 parts of precooked potato flour from ground potato flake, 40 parts of potato starch and 45 parts of water were mixed with 5 parts of wheat gluten, egg white, whole egg, sodium caseinate or Torula yeast, in separate trials, until uniform, and extruded through a low-pressure, pasta type extruder to form a desired pasta shape. The pasta was cooked in boiling water or steam, then cooled and packed under a controlled atmosphere using a gas mixture of carbon dioxide and nitrogen to extend its shelf life. The results of the retrogradation tests are given in TABLE 2.
Comparative Example C

A similar procedure to that described for Examples 7 to 11 was carried out except that no functional protein was added. The results of the retrogradation tests are given in TABLE 2.

**TABLE 2**

I. Instron Compressibility

Kg/30g pasta sample in a Kramer shear cell

<table>
<thead>
<tr>
<th>Example</th>
<th>Description</th>
<th>Initial</th>
<th>7 days</th>
<th>20 days</th>
<th>Retrograd., %</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>Control (none)</td>
<td>7.8</td>
<td>22.5</td>
<td>22.5</td>
<td>188</td>
</tr>
<tr>
<td>7</td>
<td>Wheat Gluten</td>
<td>4.0</td>
<td>4.8</td>
<td>4.8</td>
<td>20</td>
</tr>
<tr>
<td>8</td>
<td>Egg White</td>
<td>7.6</td>
<td>13.4</td>
<td>14.2</td>
<td>76</td>
</tr>
<tr>
<td>9</td>
<td>Whole Egg</td>
<td>5.6</td>
<td>13.4</td>
<td>15.4</td>
<td>139</td>
</tr>
<tr>
<td>10</td>
<td>Caseinate</td>
<td>7.2</td>
<td>15.0</td>
<td>15.2</td>
<td>108</td>
</tr>
<tr>
<td>11</td>
<td>Torula Yeast</td>
<td>5.8</td>
<td>14.0</td>
<td>15.6</td>
<td>141</td>
</tr>
</tbody>
</table>

II. DSC Analysis: 20 days at 5°C storage

<table>
<thead>
<tr>
<th>Example</th>
<th>Description</th>
<th>Peak Temperature 50-55°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>Control (none)</td>
<td>4.55</td>
</tr>
<tr>
<td>7</td>
<td>Wheat Gluten</td>
<td>2.54</td>
</tr>
<tr>
<td>8</td>
<td>Egg White</td>
<td>2.07</td>
</tr>
<tr>
<td>9</td>
<td>Whole Egg</td>
<td>3.41</td>
</tr>
<tr>
<td>10</td>
<td>Caseinate</td>
<td>3.43</td>
</tr>
<tr>
<td>11</td>
<td>Torula Yeast</td>
<td>3.48</td>
</tr>
</tbody>
</table>
As shown in the TABLE 2, the addition of wheat gluten, egg protein, whole egg, sodium caseinate and Torula yeast showed less retrogradation than the comparative control with no added protein. Wheat gluten demonstrated the most effective functional protein for minimizing starch retrogradation with stable low Instron compressibility and low enthalpy ($H$) of retrograded starch.

Examples 12 to 17

60 parts of corn flour, 20 parts of maize starch, 15 parts of pre-cooked corn flour and 41 parts of water were mixed with 5 parts of wheat gluten, egg white, whole egg, sodium caseinate, Torula yeast or gelatin, in separate trials, until uniform, and extruded through a low-pressure, pasta-type extruder to form a desired pasta shape. The pasta was cooked in boiling water, then cooled and packed under a controlled atmosphere using a gas mixture of carbon dioxide and nitrogen to extend its shelf life. The results of the retrogradation tests are given in Table 3.

Comparative Example D

A similar procedure to that described for Examples 12 to 17 was carried out except that no functional protein was added. The results of the retrogradation tests are given in Table 3.
TABLE 3

I. Instron Compressibility

Kg/15g pasta sample in a Kramer shear cell

<table>
<thead>
<tr>
<th>Example</th>
<th>Description</th>
<th>Initial</th>
<th>1 day</th>
<th>5 days</th>
<th>10 days</th>
<th>18 days</th>
<th>18 days at 5°C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>Control (none)</td>
<td>4.2</td>
<td>7.7</td>
<td>10.8</td>
<td>14.0</td>
<td>233</td>
<td></td>
</tr>
<tr>
<td>12</td>
<td>Wheat Gluten</td>
<td>2.8</td>
<td>4.4</td>
<td>4.5</td>
<td>4.6</td>
<td>64</td>
<td></td>
</tr>
<tr>
<td>13</td>
<td>Egg White</td>
<td>2.8</td>
<td>5.8</td>
<td>5.8</td>
<td>6.8</td>
<td>143</td>
<td></td>
</tr>
<tr>
<td>14</td>
<td>Whole Egg</td>
<td>2.7</td>
<td>5.6</td>
<td>5.8</td>
<td>8.0</td>
<td>196</td>
<td></td>
</tr>
<tr>
<td>15</td>
<td>Caseinate</td>
<td>3.4</td>
<td>7.0</td>
<td>7.5</td>
<td>8.2</td>
<td>141</td>
<td></td>
</tr>
<tr>
<td>16</td>
<td>Torula Yeast</td>
<td>2.7</td>
<td>6.2</td>
<td>7.6</td>
<td>8.2</td>
<td>204</td>
<td></td>
</tr>
<tr>
<td>17</td>
<td>Gelatin</td>
<td>2.4</td>
<td>5.9</td>
<td>7.0</td>
<td>7.2</td>
<td>200</td>
<td></td>
</tr>
</tbody>
</table>

II. DSC Analysis: 18 days at 5°C storage

<table>
<thead>
<tr>
<th>Example</th>
<th>Description</th>
<th>Peak Temperature 50-55°C</th>
<th>△H.J/g</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>25</td>
<td>Control (none)</td>
<td></td>
<td>3.76</td>
</tr>
<tr>
<td>12</td>
<td>Wheat Gluten</td>
<td></td>
<td>2.42</td>
</tr>
<tr>
<td>13</td>
<td>Egg White</td>
<td></td>
<td>2.67</td>
</tr>
<tr>
<td>14</td>
<td>Whole Egg</td>
<td></td>
<td>2.85</td>
</tr>
<tr>
<td>15</td>
<td>Caseinate</td>
<td></td>
<td>2.68</td>
</tr>
<tr>
<td>30</td>
<td>Torula Yeast</td>
<td></td>
<td>2.68</td>
</tr>
<tr>
<td>17</td>
<td>Gelatin</td>
<td></td>
<td>2.70</td>
</tr>
</tbody>
</table>

As shown in the TABLE 3, the addition of wheat gluten, egg protein, whole egg, sodium caseinate, Torula yeast and gelatin showed less retrogradation than the comparative control with no added protein. Wheat gluten demonstrated the most effective functional protein for
minimizing starch retrogradation with stable low Instron compressibility and low enthalpy ($\Delta H$) of retrograded starch.
THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:-

1. A process for the preparation of a refrigerated cooked product from a non-conventional starchy material containing substantially no gluten as hereinbefore defined which comprises incorporating an effective amount of a functional protein into the uncooked starchy material, cooking the starchy material, and cooling to a refrigeration temperature.

2. A process according to claim 1 wherein the starchy material is a flour or starch derived from rice, maize or potatoes.

3. A process according to claim 1 wherein the functional protein is gluten.

4. A process according to claim 1 wherein the amount of active functional protein added to the starchy material is from 1% to 20% by weight based on the weight of the starchy material.

5. A process according to claim 1 wherein the starchy material containing the functional protein is formed into a pasta before cooling.

6. A refrigerated cooked product whenever prepared by a process according to claim 1.

7. A process for the preparation of a refrigerated cooked product substantially as herein described with reference to any one of Examples 1 to 17 including Comparative Examples.

DATED this 20th Day of October, 1989

SOCIETE DES PRODUITS NESTLE S.A.

Attorney: IAN ERNST
Fellow Institute of Patent Attorneys of Au of SHELSTON WATERS