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A process for the production of a skimmed milk

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A PROCESS FOR THE PRODUCTION OF A SKIMMED MILK

1 This invention relates to a process for the production
of a skimmed milk of reduced phosphate and calcium contents.

 It is known that cow's milk can be better adapted to
the nutritional and physiological needs of infants by
5 depletion in mineral salts and enrichment with milk serum
proteins more readily digestible than casein. It is also
known that the gastric secretion of acid and pepsin is
relatively low in infants and that a milk adapted to the
needs of infants should thus have a low buffering power.

10 Accordingly, one known process for the production of
a milk better adapted to the needs of infants comprises
demineralizing a lactoserum by ion exchange or by electro-
dialysis and adding a caseinate to the demineralized lacto-
serum. Another known process comprises recombining the
15 constituents of milk, particularly casein or caseinates,
ultrafiltered lactoserum proteins, lactose, salt and
vitamins, in adequate proportions.

 However, these processes are open to criticism in that
they involve the elimination of substantial quantities of
20 minor, known or unknown constituents of milk. However,
some of these minor constituents can be of considerable value
to the metabolism and physiology of infants.

 The object of the present invention is to propose a
new approach to these problems by adequate reduction of the
25 phosphate and calcium contents of the skimmed milk itself
and the use of the product obtained for the production of
a milk adapted to the needs of infants. Another object
of the present invention is to provide a milk adapted to
the needs of infants thus obtained which itself has a
30 reduced calcium content and a reduced phosphate content.

 To this end, the process for producing a skimmed milk

1 of reduced phosphate and calcium contents according to the
present invention is characterised in that a skimmed milk is
acidified to a pH of from 5.2 to 6.0 and from 3.5 to 18 litres
of the acidified skimmed milk per equivalent of exchanger are
5 passed over an anion exchanger, followed by neutralization.
The skimmed milk is preferably acidified to a pH of from 5.4 to 5.6.

It has been found that, if a skimmed milk is acidified
within the indicated limits, the phosphate ions are
unexpectedly retained on the anion exchanger preferentially
10 to the citrate and Cl^- ions in contrast to what is observed
during the demineralization of lactoserum by ion exchange.
It has also been found, again surprisingly, that not only
the phosphate ions, but also the Ca^{++} ions are thus re-
tained on the anion exchanger.

15 In the context of the invention, an "acidified skimmed
milk" is understood to be a mixture of skimmed milk and an
acidifying agent, irrespective of type. Thus, in one
embodiment of the process according to the invention where
a skimmed milk is acidified by addition of a decationized
20 lactoserum, the "acidified skimmed milk" is the mixture of
skimmed milk and decationized lactoserum.

In addition, the expression "demineralized lactoserum"
is used hereinafter to designate a decationized and de-
anionized lactoserum as opposed to a decationized but not
25 deanionized lactoserum.

Finally, the expression "total serum proteins" is used
to designate the total nitrogenous matter of the lactoserum
determined as total nitrogen multiplied by 6.38.

The use of the skimmed milk of reduced phosphate and
30 calcium contents obtained by the process according to the
invention for the production of a milk adapted to the needs
of infants is characterized in that a mixture is prepared
which contains, in percent by weight based on the dry matter
of the mixture, from 15 to 25% of dry matter of the skimmed
35 milk of reduced phosphate and calcium contents, from 45 to 55%

1 of dry matter of a demineralized lactoserum and from 25 to
30% of fats.

The milk adapted to the needs of infants obtained by
the use of the product of the process according to the
5 present invention is characterized in that it contains,
in percent by weight of dry matter,

from 4.5 to 5.5% of casein,
from 7.0 to 8.0% of total serum proteins,
from 25 to 30% of fats,
10 from 54 to 62% of lactose,
from 0.2 to 0.4% of calcium,
from 0.015 to 0.05% of magnesium,
from 0.10 to 0.17% of sodium,
from 0.4 to 0.6% of potassium,
15 from 0.4 to 0.6% of phosphate,
from 0.3 to 3% of citrate,
from 0.3 to 0.45% of chlorine.

The starting material used for carrying out the
process according to the invention may be fresh or re-
20 constituted skimmed cow's milk. This milk is acidified to
a pH of from 5.2 to 6.0 and preferably to a pH of from 5.4
to 5.6. The pH range is limited at the lower end by the
precipitation of casein. At its upper end, it is limited
by the disappearance of the surprising preferential retention
25 of the phosphate to the benefit of the retention of citrate
and chloride.

Acidification may be carried out, for example, with
citric acid or hydrochloric acid.

In a preferred variant mentioned earlier on, a de-
30 cationized lactoserum is used. It is preferred to use a
soft lactoserum, of the type produced, for example, by
cheesemakers making pressed curd and cooked curd cheeses
and by manufacturers of rennet casein, decationized by
passage over a cation exchanger, particularly an exchanger
35 of the sulfonic type. A lactoserum such as this may

1 advantageously have a pH of from 1.2 to 3.0. It is
assumed that, if a decationized lactoserum is particularly
suitable for carrying out the process according to the
invention, it is because, by dilution, chelation and
5 acidification, it exerts a triple effect in releasing
the Ca from the casein micelle which in turn releases
the inorganic phosphate from the casein micelle.

In the following step of the process according to the
invention, the acidified skimmed milk is passed over an
10 anion exchanger. This exchanger may be in particular of
the quaternary ammonium or secondary or tertiary amine type.
The anion exchanger may be used in the OH^- form. In that case,
the skimmed milk which was passed over the exchanger has
a mean pH of from about 7.5 to 9 and is neutralized,
15 preferably with citric acid. The anion exchanger may
also be used in the form of citrate and/or Cl^- anions.
In that case, the phosphate anions are directly exchanged
for the citrate and/or Cl^- anions and no increase in the
pH is observed during passage over the exchanger.

20 The skimmed milk is passed over the anion
exchanger in a quantity of from 3.5 to 18 liters of skimmed
milk per equivalent of exchanger capacity. By remaining
below the minimum ratio thus indicated, an unnecessarily
high volume of exchanger is used. By exceeding the maximum
25 ratio thus indicated, the phosphate content of skimmed milk
is not sufficiently reduced.

The skimmed milk of reduced phosphate and calcium
contents obtained by the process according to the invention
is thus an intermediate product intended for use in the
30 production of milks adapted to the needs of infants. As
indicated above, this use is best effected by preparing a
mixture containing, in percent by weight based on the dry
matter of the mixture, from 15 to 25% of dry matter of the
skimmed milk of reduced phosphate and calcium contents,
35 from 45 to 55% of dry matter of a demineralized lactoserum

1 and from 25 to 30% of fats. A lactoserum demineralized
by ion exchange or by electrodialysis for example may be used
to prepare this mixture, although it is preferred to use a
sweet lactoserum demineralized by decationization on a
5 cation exchanger followed by deanionization on an anion
exchanger. So far as the fat is concerned, it is preferred
to use a milk fat containing a small amount of added
vegetable fat.

A dry or liquid mixture may be prepared, depending on
10 whether or not the skimmed milk of reduced phosphate and
calcium contents and/or the demineralized lactoserum was
dried beforehand, for example by spray-drying. If
necessary, the liquid mixture itself may be spray-dried for
example. The milk thus obtained adapted to the needs of
15 infants itself has a reduced phosphate content and a
reduced calcium content. Its composition is shown above.

The invention is illustrated by the following
Examples in which the percentages quoted are by weight.

EXAMPLE 1

20 A skimmed milk having a pH of 6.6 and a dry matter
content of 9% and containing, expressed in mg of ions
per 100 ml of skimmed milk,
270 mg/100 ml of phosphate
180 mg/100 ml of citrate
25 120 mg/100 ml of calcium
is acidified to pH 5.5 by addition of 1.9 g of citric acid
per liter of skimmed milk.

The acidified skimmed milk is passed over an anion
exchanger in the OH^- -form in a quantity of 3.5 l of milk
30 per equivalent of exchanger capacity. The pH of the acidified
skimmed milk which was passed over the exchanger initially
rises to around 11.5 and then drops back to about 8.5. The
mean pH of the milk passed over the exchanger is 9.0. After
neutralization to pH 7.0 with citric acid, a skimmed milk
35 of reduced phosphate and calcium contents is obtained which

1 has a dry matter content of 9% and contains, in mg of
ions per 100 ml of skimmed milk,
120 mg/100 ml of phosphate
150 mg/100 ml of citrate
5 57 mg/100 ml of calcium

COMPARISON EXAMPLE

The procedure is as described in Example 1, except
that the skimmed milk is passed directly over the anion
exchanger without acidification. The pH of the skimmed
10 milk which was passed over the exchanger again rises but
the phosphate content remains constant. Only the citrate
and Cl^- ions are exchanged for OH^- .

EXAMPLE 2

The same skimmed milk as in Example 1 is acidified
15 to pH 5.5 by addition of decationized soft lactoserum
having a pH of 1.8 and a dry matter content of 5.5% in a
quantity of 0.4 l of lactoserum per liter of skimmed
milk.

The acidified skimmed milk is passed over an anion
20 exchanger in the OH^- -form in a quantity of 3.5 l of milk
per equivalent of exchanger capacity. The mean pH of
the milk which was passed over the exchanger is 9.0.
After neutralization to pH 7.0 with citric acid, a
skimmed milk of reduced phosphate and calcium contents
25 is obtained which has a dry matter content of 8% and
which contains, in mg of ions per 100 ml of skimmed
milk,

134 mg/100 ml of phosphate
134 mg/100 ml of citrate
30 46 mg/100 ml of calcium.

EXAMPLES 3-5

Various skimmed milks of reduced phosphate and
calcium contents are prepared in the same way as in
Example 2, except that the acidified skimmed milk is
35 passed over the ion exchanger in different volumes of
milk per equivalent of exchanger capacity.

The respective values of the volumes of acidified
skimmed

1 milk passed over the anion exchanger (vol. passed), the mean
pH's of the milk which was passed over the exchanger (mean
pH), the dry matter content of the skimmed milk which was
passed over the exchanger (dry matter) and the ion content of
5 the milk after neutralization to pH 7.0 (ions) are
shown in the following Table. The corresponding values
taken from Examples 1 and 2 are repeated therein. The
data of the starting skimmed milk are shown in the
extension of the pH, dry matter and ions columns to
10 facilitate comparison.

	Ex. No.	Vol. passed l/equ.	Mean pH	Dry matter %	phosphate	Ions citrate mg/100 ml	Ca
15	1	3.5	9	9	120	150	57
	2	3.5	9	8	134	134	46
	3	7	8.9	8	120	121	44
20	4	10.5	8	8	140	143	51
	5	17.5	7.7	8	150	130	55
25	Starting skimmed milk		6.6	9	270	180	120

EXAMPLE 6

A skimmed milk is acidified by addition of a de-
30 cationized sweet lactoserum. The acidified skimmed milk
is passed over an anion exchanger in the OH⁻-form in a
quantity of 10.5 l of acidified skimmed milk per exchanger
equivalent.

A powdered milk adapted to the needs of infants is
35 prepared by mixing the skimmed milk of reduced phosphate

1 and calcium contents thus obtained with a demineralized
lactoserum and fat in such proportions that the mixture
contains, in percent based on the dry matter of the
mixture, 21% of dry matter of this skimmed milk of
5 reduced phosphate and calcium contents, 51.5% of dry
matter of the demineralized lactoserum and 27.5% of fat.
The mixture is concentrated by evaporation in vacuo to a
dry matter content of 40% and then spray-dried. The
powder obtained has a moisture content of approximately
10 5%.

The details concerning the characteristics and
composition of the various products involved or
obtained in this Example are shown in the following
Table in which the percentages quoted are based on the
15 total weight in the first four columns and on the weight
of dry matter in the last three columns. To accommodate
them on the page, the column headings have been
abbreviated. The full headings are as follows:

	1st column (sk. milk):	starting skimmed milk
20	2nd column (dec. lact.):	decationized lactoserum
	3rd column (ac. sk. milk):	acidified skimmed milk
	4th column (red. sk. milk):	skimmed milk of 25 reduced phosphate and calcium contents
	5th column (red. sk. milk):	skimmed milk of reduced phosphate and calcium contents
30	6th column (dem. lact.):	demineralized lactoserum
	7th column (adapt. milk):	milk adapted to the needs of infants.

	Sk. milk	dec. lact.	ac. sk. milk	red sk. milk	red. sk. milk	dem. lact.	adapt. milk
pH	6.7	1.8	5.5	7.5	7.5	6.7	6.8
Dry matter %	9.0	5.5	8.0	8.0	8.0	20	95
Casein %	2.8	0	2.0	2.0	25	0	5.0
Total Serum proteins %	0.6	0.6	0.6	0.6	7.5	12	7.4
Fats %	0.1	0.05	0.08	0.08	1.0	1.0	27.5
Lactose %	5.5	4.9	5.3	5.3	61	86	58
Ca %	0.115	0.002	0.081	0.042	0.58	0.34	0.28
Mg %	0.011	0.001	0.008	0.006	0.06	0.02	0.02
Na %	0.048	0.06	0.036	0.038	0.40	0.10	0.15
K %	0.165	0.016	0.116	0.122	1.40	0.30	0.45
Phosphate %	0.270	0.150	0.235	0.130	1.70	0.35	0.51
Citrate %	0.180	0.160	0.174	0.106	1.25	0.50	0.50
Cl %	0.120	0.100	0.112	0.093	0.90	0.08	0.22

The Cl content of the adapted milk is standardized during mixing to a value of from 0.3 to 0.45% by addition of KCl, $MgCl_2$ and/or $CaCl_2$.

CLAIMS

1. A process for the production of a skimmed milk of reduced phosphate and calcium contents,
5 wherein a skimmed milk is acidified to a pH of from 5.2 to 6.0 and from 3.5 to 18 litres of the acidified skimmed milk per equivalent of exchanger are passed over an anion exchanger, followed by neutralization.
- 10 2. A process as claimed in Claim 1, wherein the skimmed milk is acidified to a pH of from 5.4 to 5.6.
- 15 3. A process as claimed in Claim 1, wherein the skimmed milk is acidified with citric acid, hydrochloric acid, or a decationized lactoserum.
- 20 4. A process as claimed in Claim 1, wherein the skimmed milk is acidified with a decationized lactoserum having a pH of from 1.2 to 3.0.
- 25 5. A process as claimed in Claim 1, wherein the anion exchanger resin is of the quaternary ammonium type or secondary or tertiary amine type.
6. A process as claimed in Claim 1 wherein the anion exchanger is in the OH^- form or in the form of citrate and/or Cl^- anions.
- 30 7. The use of the skimmed milk of reduced phosphate and calcium content obtained by the process claimed in Claim 1

for the production of a milk adapted to the needs of infants, wherein a mixture is prepared which contains, in percent by weight based on the dry matter of the mixture, from 15 to 25 % of dry matter of the skimmed milk of reduced phosphate and calcium contents, from 45 to 55 % of dry matter of a demineralized lactoserum and from 25 to 30 % of fats.

8. A milk adapted to the needs of infants obtained by the use claimed in Claim 7, which contains in percent by weight of dry matter,

from 4.5 to 5.5 % of casein,
from 7.0 to 8.0 % of total serum proteins,
from 25 to 30 % of fats,
from 54 to 62 % of lactose,
from 0.2 to 0.4 % of calcium,
from 0.015 to 0.05 % of magnesium,
from 0.10 to 0.17 % of sodium,
from 0.4 to 0.6 % of potassium,
from 0.4 to 0.6 % of phosphate,
from 0.3 to 3 % of citrate,
from 0.3 to 0.45 % of chlorine.

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