

Candidate's Answer Paper
(Examination Paper A - Chemistry)

CLAIMS

1. A glycerol ester having 3 to 10 glycerol units linked to each other by ether groups in a polyglycerol chain and having one or more hydroxyl groups esterified with a straight-chain fatty acid having a total of 12 to 26 carbon atoms.
2. An ester according to claim 1 having one or two fatty acid ester groups.
3. An ester according to claim 1 or claim 2 which is not water-soluble but is water-dispersible.
4. An ester according to any of claims 1 to 3 that is a palmitic or arachidic ester.
5. An ester according to any of claims 1 to 3 that is hexaglycerol distearate or decaglycerol distearate.
6. An ester according to any of claims 1 to 3 that is triglycerol monostearate.
7. A low calorie edible emulsion comprising at least 0.3% by weight of an ester according to any preceding claim and at least one edible ingredient.
8. An emulsion according to claim 7 wherein the amount of ester is 2 to 5% by weight of the emulsion.
9. An emulsion according to claim 8 that is in the form of a foam and which additionally comprises 0.2 to 3.5% by weight of a foam stabiliser.
10. An emulsion according to claim 8 that has been expanded 0.5 to 1.1-fold and which additionally comprises 0.2 to 0.5% by weight of a foam stabiliser.
11. A frozen dessert comprising an emulsion according to claim 10.
12. A process for preparing an expanded emulsion according to claim 9 or 10, comprising whipping the emulsion at 52°C to 57°C.
13. A process for preparing an ester as defined in any of claims 1 to 6, comprising reacting the polyglycerol chain with an ester of the fatty acid in the presence of a fatty acid soap and a catalyst, wherein at least 8% by weight of the fatty acid in the soap has a chain length of less than 15 carbon atoms, and the ratio of the soap to the polyglycerol is in the range 0.1:1 and 2.5:1 and the ratio of the ester to polyglycerol is in the range 10:1 and 20:1.
14. A process according to claim 13, wherein the fatty acid ester is a methyl ester.
15. A process according to claim 13 or 14, wherein the ratio of soap: polyglycerol is approximately 1.6:1.
16. A process according to any of claims 13 to 15 which is carried out at a temperature of 110 to 150°C.
17. A method of processing an ester as defined in any of claims 1 to 6,

comprising passing the ester in its molten state through a spray drier operated with a cooling medium having a temperature of less than 30°C.

18. Use of an ester according to any of claims 1 to 6 as an emulsifier.
19. A frozen dessert according to claim 11 that is an ice-cream substitute, and comprises up to 45% by weight bodying agents and up to 55% by weight non-aqueous ingredients.

Note to the Examiner

1. I have not used the two-part form of claim as this is not appropriate (Rule 29(1)(b) EPC) in this context.
2. The process claim of claim 12 refers to the esters of claims 1 to 6, but at page 6 of the client's letter it is clear that this process applies to other esters too. For purposes of unity I have not included a broad process claim, but a suitable one would be as laid out in claim 12 but not restricted to the fatty acid chain length. The processing method of claim 17 could similarly be broadened.

NOVEL GLYCEROL ESTERS SUITABLE FOR USE IN LOW-CALORIE FOODS AND METHODS FOR PREPARING AND PROCESSING THOSE ESTERS

The present invention relates to novel glycerol esters suitable for use as emulsifiers or foaming agents in low-calorie food products. The invention also relates to methods for preparing those esters and for processing them for easy handlability.

Glycerol esters as a class of compounds and for use as emulsifiers is known from Documents I and II.

Document II discloses the use of glycerol esters to form stable and spreadable low calorie foods. There is no disclosure of any specific esters that may be of particular use.

Document I is concerned with the harmful, cumulative effects that some known glycerol esters have on the body. It discloses that such effects can be avoided by the use of water-soluble esters, that are formed from polyglycerols and fatty acids having relatively short chain lengths. For instance, tetraglycerol monoactanoate and decaglycerol hexanoate are specifically mentioned.

The disadvantages with the glycerol esters known in the prior art is their limited applicability with regard to the type, or form, of product they can be used in.

The object of the present invention is to provide novel glycerol esters that can be used for a whole variety of purposes as emulsifiers, or foaming agents.

According to a first aspect of the present invention a glycerol ester has 3 to 10 glycerol units linked to each other by ether groups in a polyglycerol chain and having one or more hydroxyl groups esterified with a straight chain fatty acid having a total of 12 to 26 carbon atoms.

With the help of these esters, products of varying texture, ranging from creamy,

slightly expanded substances suitable for use as spreads, to products resembling highly foamed whipped cream can be prepared. The compounds can be used, for example to make a butter substitute. The properties of such products are significantly influenced by the emulsifiers or foaming agents used.

For instance, the ester of the invention can be used to prepare a kind of ice-cream substitute.

According to a second aspect of the invention, a low calorie edible emulsion comprises at least 0.3% by weight of an ester as defined above. Such emulsions can be formulated into a wide variety of food-product types.

The common feature of these products, which are normally based on aqueous emulsions, is that they have a significantly lower caloric value than their conventional equivalents. This is because the esters we have found make it possible, for example, to reduce the proportion of fatty ingredients, or indeed to eliminate such ingredients altogether, while taking account of the need to avoid impairing taste and other pleasant oral sensations (mouthfeel).

According to a third aspect of the present invention, a process for preparing the novel esters comprises reacting the polyglycerol chain with an ester of the fatty acid in the presence of a fatty acid soap and a catalyst ... [see remainder of claim 13].

It is quite surprising that this process, in its use of soap, does not produce an objectionable flavour in its products, as has been the case in the prior art [Document II for instance mentions this].

Once the esters of the invention have been prepared, they have to be converted into a usable form. The problem is that they have a wax-like consistency which makes it difficult to blend them. One known process for handling wax-like compounds involves blending them in a molten state - at a temperature above their melting-point, e.g. at least 70°C - with some of the other solid ingredients with which they are to be further processed. Another known method of processing wax-like products is to grind the deep-frozen materials to a powder at temperatures ensuring that they remain sufficiently brittle. However, during this grinding the process conditions must be controlled in a very complicated and costly way and the grinder often becomes clogged.

It is a further object of the invention to provide an improved method for processing the esters of the invention.

According to a fourth aspect of the present invention, a method of processing the esters of the invention comprises ... [see claim 17].

A suitable coding medium is, for instance, air.

On emerging from the spray nozzle, the material solidifies into fine particles. As in the case of the grinding method, other ingredients may be added at this point to prevent the fine particles from sticking together.

This is a particularly elegant processing method with regard to the esters of the invention.

Suitable fatty acids for reaction with the polyglycerol chain range from straight-chain carboxylic acids with a total of 12 carbon atoms (lauric acid) to those with a total of 26 carbon atoms (cerotic acid) and include those with 14

(myristic acid), 16 (palmitic acid), 18 (stearic acid) and 20 (arachidic acid) carbon atoms. These can be used to prepare compounds which are completely water-soluble or compounds which are completely oil-soluble.

The esters have one or two fatty acid ester groups, and are preferably those which are not water-soluble but show good water-dispersability. With the palmitic and arachidic esters we obtained particularly stable emulsions which lend themselves well to further processing and can be foamed. The stearates are even better suited to our purposes. Here, special mention is made of hexaglycerol distearate and decaglycerol distearate. The best product of all is triglycerol monostearate.

The polyglycerol ester has a sufficient emulsifying effect in amounts of at least 0.3% by weight. The texture of the resultant emulsion varies according to the amount of the ester. If the concentration exceeds 5% by weight, the product takes on a fatty, butter-like consistency, even if whipped with air. In the course of our experiments we found that a concentration of 2 to 5% by weight of the polyglycerol ester yields a mix, which does not resemble butter and which can be foamed to provide a high degree of expansion and is suitable for use as a topping resembling whipped cream. The amount of expansion is determined by the extent of whipping or mixing.

For this purpose a suitable amount of hot water and the other ingredients are added to the polyglycerol ester and so much air is beaten into the mixture with a mechanical mixer (an ordinary household electric mixer will do) that the resultant foam does not expand any further. The temperature of the water is not particularly important: it can be anywhere within the range of 50 to 100°C. However, to improve the texture of the mixture, the temperature at the time of whipping must be between 50 and 60°C, preferably between 52 and 57°C.

Other ingredients may also be added: for example, fats, flavouring, colouring and bodying agents, such as milk powder, whey powder, dried egg, sugar or artificial sweetener. Dextran is an example of a suitable bodying agent, if a very low caloric value is desired.

The fully expanded foam products collapse after a short time unless a hydrophilic vegetable gum is added as a foam stabiliser, in an amount ranging from 0.2 to 3.5% by weight of the aqueous phase. Gum arabic (acacia gum), xanthan gum, guar gum or carrageenan are particularly suitable. However, it must be remembered that vegetable gums tend to inhibit foaming or even prevent it altogether. When preparing fully expanded foams (e.g. for toppings), the gums can therefore only be added after the foam has developed. The resultant mixture is smooth and substantially tasteless.

After numerous unsuccessful attempts we found that, for frozen desserts such as ice-cream, the amount of bodying agents and the amount of the above-mentioned non-aqueous ingredients altogether should not exceed, respectively, 45 and 55% by weight of the total mixture. Thereto our polyglycerol ester (0.5-1% by weight of the total) and water are added. Blending and whipping are adjusted to provide a 0.5 to 1.1-fold expansion (a so-called overrun of 50 to 110%), thereby obtaining a stable emulsion with good mouthfeel.

As well as acting as a stabiliser, the vegetable gum also improves the texture of the final product. For example, it delays or prevents unwanted crystallisation of ice or sugar. For frozen desserts, a liquid melt is desired. Excess stabiliser prevents this and leads to the formation of a stiff, pudding-like melt - a phenomenon known as "melt resistance". In the present case, the amount of

stabiliser is therefore limited to between 0.2 and 0.5% by weight.

As mentioned above, the process we used to produce these esters consists in converting the relevant polyglycerol with an ester of the chosen straight-chain fatty acid in the presence of fatty acid soap and a catalyst. Methyl esters, or esters with another alcohol boiling at up to about 100°C are particularly suitable.

Almost any fatty acid soap can be used for the process, provided that a certain proportion of it has a fatty acid chain length of less than 15 carbon atoms. This proportion should be at least 8% by weight of the total amount of the fatty acids bound in the soap. Soaps with a very short chain length (6 carbon atoms or more) may be used, but best results are obtained using soaps with a chain length of 10 to 12 carbon atoms.

The molar ratio between the reactants is also very important. The ratio of soap to polyol must be in the range 0.1:1 and 2.5:1; with regard to yield and reaction time, the optimum ratio is approximately 1.6:1. The ratio of ester to polyol must be in the range 10:1 and 20:1.

Any of the normal catalysts for this type of reaction may be used; for example, any strong base, such as sodium or potassium hydroxide, may be used. Solvents can also be used. The pressure is adjusted so that the low-boiling alcohol released during the reaction can be removed by distillation. The temperature is normally maintained within the range of 100 to 180°C, preferably within the range of 110 to 150°C.