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PLASTICIZED SPONGE MATERIAL AND
METHOD OF MAKING SAME

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This invention relates to a sponge material comprising partially formalized polyvinyl alcohol, and relates particularly to such a sponge material having incorporated therein a plasticizer to maintain the sponge in a soft and resilient condition.

Sponge and expanded materials comprising partially formalized polyvinyl alcohol and their preparation are described in my copending application Serial No. 29,657, filed May 27, 1948, and now Patent No. 2,609,347, which is a continuation-in-part of my prior copending application Serial No. 769,537, filed August 19, 1947, now abandoned. These sponge materials are prepared by reacting the formaldehyde with polyvinyl alcohol so that only a portion of the hydroxyl groups of the alcohol enters into the reaction. Preferably solution of the materials is expanded as by beating air into the solution and the reaction is then permitted to proceed so that a solid is formed containing openings formerly occupied by the bubbles of air or other gas. In the preferred procedure, an acid catalyst is employed in order to speed up the reaction.

As further set forth in the above mentioned Patent 2,609,347, the sponge materials described therein are made by a process comprising providing an aqueous solution of formaldehyde, polyvinyl alcohol containing less than 10% residual hydrolyzable material in the molecule, an acid catalyst in which the acid functional group is inorganic and having a dissociation constant of at least 10^{-2} and a froth-stabilizing wetting agent soluble in and substantially stable to said solution; forming a substantially stable and homogeneous froth with said solution stabilized with the wetting agent; reacting the polyvinyl alcohol and the formaldehyde in the solution thereby causing gelation and solidification of the froth with the production of interconnected pores simultaneously with the progression of the reaction, the froth being formed prior to appreciable reaction between the polyvinyl alcohol and the formaldehyde; and stopping said reaction when approximately 35-80% of the hydroxyl groups of the polyvinyl alcohol have been reacted with the formaldehyde.

Sponge materials prepared in the above manner are soft and resilient when they contain appreciable amounts of water. When the sponge materials become dry, however, they shrink to a certain extent and become hard and rigid. They are immediately softened as soon as water is reintroduced into the sponge material. Although this re-wetting of the sponge material immediately changes it from a rigid solid to a resilient elastic material and the drying out of the material causes no deleterious effects, the dry sponge material does not have as pleasing an appearance to the prospective purchaser.

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I have discovered that the sponge material may be maintained soft and resilient if a plasticizer is incorporated therewith. These plasticizers are polyhydric alcohols and derivatives thereof and may be introduced by soaking the sponge material in an aqueous solution of the alcohol. The polyhydric alcohols are preferably those that are liquid at ordinary room temperatures. After soaking in the solution, the excess solution is removed. These plasticizers maintain the sponge material in a soft, resilient condition.

One of the features of this invention is to provide a method of making a soft, resilient sponge material comprising providing a partially formalized polyvinyl alcohol sponge and soaking the sponge in an aqueous solution of a polyhydric alcohol; another feature of this invention is the provision of a partially formalized polyvinyl alcohol sponge material containing as a plasticizer a polyhydric alcohol. Other features and advantages of the invention will become apparent from the following description and the accompanying claims.

Among the suitable plasticizers that have been discovered are diethylene glycol; triethylene glycol and other polyethylene glycols having a molecular weight of at least 200; trimethylene glycol; tetramethylene glycol; pentamethylene glycol; 2-methyl-1,3-pentanediol; 2-methyl-2,4-pentanediol; 1,2,4-butanetriol; 1,2,5-pentanetriol; mixtures of pentanediols constituting the residue of hydrogenated furfural; butanediol-1,3; butanetriols; pentanetriols; ethylene glycol monomethyl ether; diethylene glycol monoethyl ether and propylene glycol. These plasticizers may be used alone or in combinations of two or more and may be used in combination with hygroscopic materials such as glycerol and calcium chloride. Glycerol alone may be employed as a plasticizer, but this is not preferred. The best plasticizers appear to be diols or those compounds containing two hydroxy groups in the organic molecule. Diethylene glycol, triethylene glycol and pentamethylene glycol have been found to be particularly effective. These diols appear to have a somewhat better softening effect than other hydroxy compounds and are effective for greater lengths of time.

Any amount of plasticizer desired may be used so long as a plasticizing effect is achieved. In general, approximately 50% of the anhydrous plasticizer should be introduced into the sponge by weight of the dry sponge material. In actual practice, it has been found that excellent results are achieved when at least 30% of the plasticizer is employed by weight of the dry sponge material. Ordinarily not more than about 100% is required, but in some instances it may be advisable to use more. Under conditions of high humidity, the minimum amount may be reduced below 30%.

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For example, 20% is satisfactory if the relative humidity of the atmosphere surrounding the sponge is at least 85-90%.

The following examples illustrate methods of practicing this invention:

Example 1

An 8.9 gram specimen of dry resin material prepared as described in my prior Patent No. 2,609,347 was soaked in a mixture of 125 parts by weight of ethylene glycol and 36 parts of water. The sponge material was then centrifuged in a 5 inch basket at 5,000 R. P. M. to remove the excess liquid. The sponge material retained 8.85 grams of the liquid and remained flexible for an appreciable period of time.

Example 2

5 grams of the dry sponge material was soaked in a solution of 73 parts by weight of glycerol and 36 parts of water. The wet sponge material was centrifuged as described in Example 1 and retained 2.8 grams of the liquid. The resulting sponge material was quite flexible, but retained these properties only in atmospheres having a humidity above 85%.

Example 3

7 grams of dry sponge material was soaked in a mixture of 12 parts by weight of pentamethylene glycol, 5 parts of calcium chloride and 20 parts of water. The sponge material retained 10 grams of the solution after centrifuging as described above and was found to be quite soft and flexible for a relatively long period of time.

Example 4

20 grams of the dry sponge material was soaked in a mixture of 14.4 parts by volume of water, 7 parts of pentamethylene glycol and 25 parts of 1,2-pentanediol. The sponge material after centrifuging retained 30 grams of the solution. The sponge material remained quite flexible for a long period of time when exposed to air at room temperatures.

Example 5

6.5 grams of dry sponge material was soaked in a mixture of 73 parts by volume of glycerol, 25 parts of pentamethylene glycol and 36 parts of water. After centrifuging, the sponge material retained 5 grams of the mixture and remained flexible.

Example 6

The dry sponge material was treated with a mixture of 36 parts by weight of water and 80 parts of 1,2,5-pentanetriol. After centrifuging, the sponge material remained flexible when exposed to air.

Example 7

13.5 grams of dry sponge material containing 16 grams of a mixture of 36 parts by weight glycerol, 73 parts of 2-methyl-2,4-pentanediol, 36 parts of ethylene glycol and 216 parts of water remained flexible upon exposure to air.

Example 8

A dry sponge material containing an equal weight of a mixture of 73 parts by weight of glycerol, 90 parts by weight of 1,3-butanediol and 144 parts of water remained flexible when exposed to air.

Example 9

10 grams of the dry sponge material was treated

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with a solution of 11.5 parts by volume of 2-methyl-1,3-pentanediol and 45 parts of water. After centrifuging, 22 grams of this solution was retained. The sponge material remained flexible upon exposure to air.

Plasticizers for the partially formalized polyvinyl alcohol sponges consisting of either polyethylene glycols or polyalkanol amines are described and claimed in my copending application Serial No. 116,643, filed September 19, 1949.

Having described my invention as related to various embodiments of the same, it is my intention that the invention be not limited by any of the details of description unless otherwise specified, but rather be construed broadly within its spirit and scope as set out in the accompanying claims.

I claim:

1. The method of making a soft, resilient sponge material having interconnected pores by reacting polyvinyl alcohol and formaldehyde, which comprises: providing an aqueous solution of formaldehyde, polyvinyl alcohol containing less than 10% residual hydrolyzable material in the molecule, an acid catalyst in which the acid functional group is inorganic and having a dissociation constant of at least 10^{-2} and a froth-stabilizing wetting agent soluble in and substantially stable to said solution; forming a substantially stable and homogeneous froth with said solution stabilized with the wetting agent; reacting the polyvinyl alcohol and formaldehyde in the solution thereby causing gelation and solidification of the froth with the production of interconnected pores simultaneously with the progression of the reaction, the froth being formed prior to appreciable reaction between the polyvinyl alcohol and the formaldehyde; stopping said reaction when approximately 35-80% of the hydroxyl groups of the polyvinyl alcohol have been reacted with the formaldehyde; whereby a sponge material is formed impregnating said sponge with an aqueous solution of a polyhydric alcohol and then removing excess of said solution until there remains deposited on said sponge about 20-100% of said polyhydric alcohol based on the dry weight of the sponge.
2. A method according to claim 1 in which said polyhydric alcohol is pentamethylene glycol.
3. A method according to claim 1 in which said polyhydric alcohol is butanetriol.
4. A method according to claim 1 in which said polyhydric alcohol is diethylene glycol.
5. A method according to claim 1 in which said polyhydric alcohol is ethylene glycol.
6. A method according to claim 1 in which said polyhydric alcohol is triethylene glycol.
7. A sponge material prepared by the method of claim 1.

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References Cited in the file of this patent
UNITED STATES PATENTS

Number	Name	Date
1,779,011	Pearce	Oct. 21, 1930
2,037,049	Sager	Apr. 14, 1936
2,056,496	Macht et al.	Oct. 6, 1936
2,370,126	Danie	Feb. 27, 1945
2,398,049	Schneider et al.	Apr. 9, 1946
2,478,879	Ten Broeck	Aug. 9, 1949

FOREIGN PATENTS

Number	Country	Date
573,966	Great Britain	Dec. 14, 1945