

[54] PROCESS FOR THE MANUFACTURE OF FLOWABLE PASTY DETERGENTS CAPABLE OF BEING PUMPED	3,246,026	4/1966	Sowerby et al.	260/459
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FOREIGN PATENTS OR APPLICATIONS

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Related U.S. Application Data

[63] Continuation of Ser. No. 795,868, Feb. 3, 1969, abandoned.

Foreign Application Priority Data

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[58] **Field of Search**..... 252/550, 551, 554, 557, 252/558, 555, 545; 260/458, 459, 505 N, 671 B

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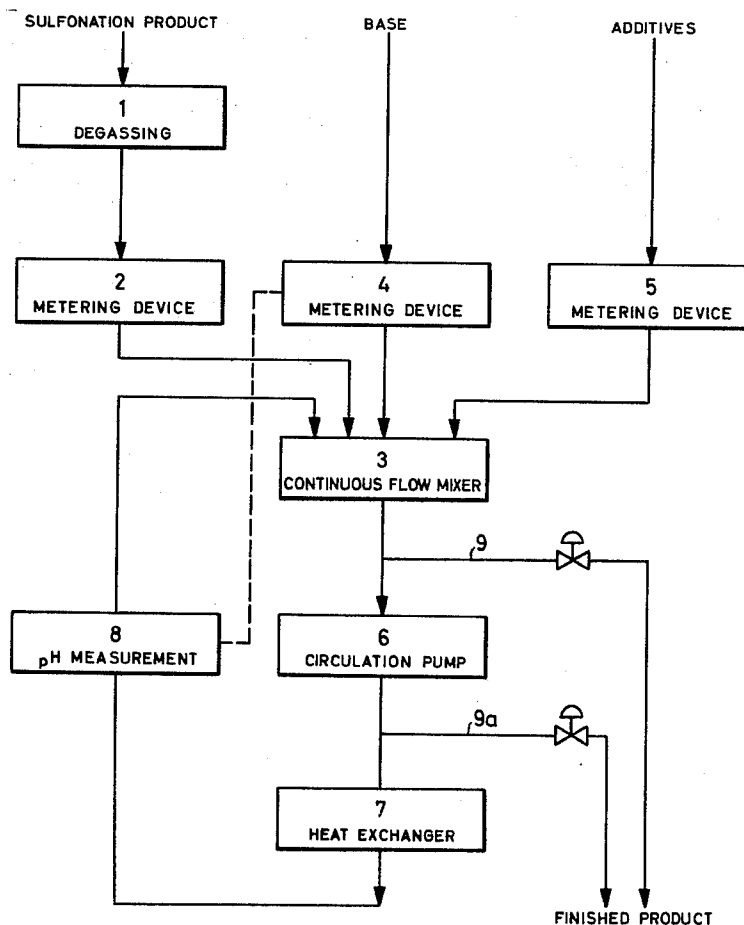
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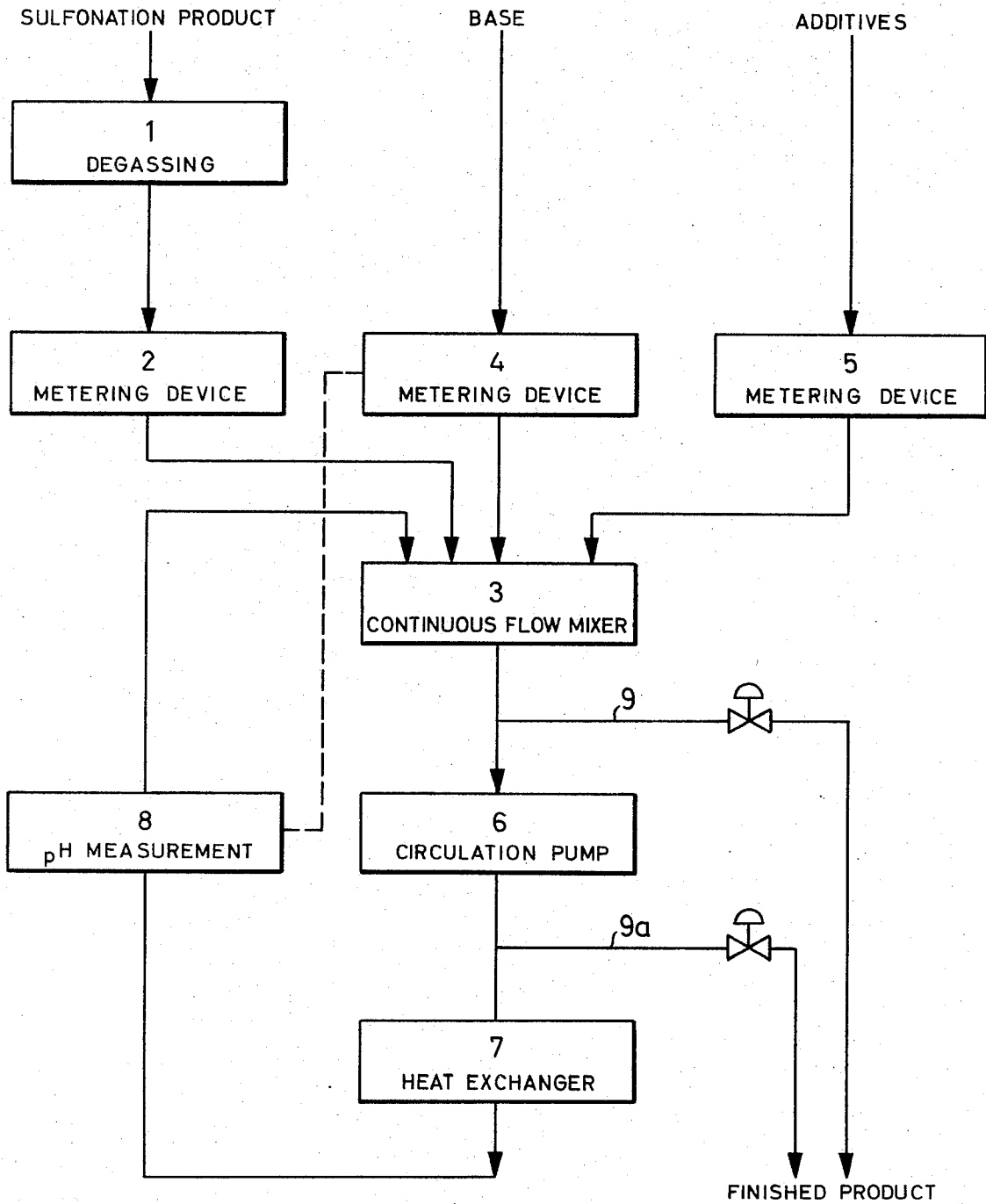
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[57] **ABSTRACT**

Flowable pasty raw materials for detergents capable of being pumped and having a content of washing active substance of more than 60% are obtained by neutralizing organic sulfuric acid semi-esters or sulfonic acids with alkalis. Before neutralization the sulfuric acid semi-esters or sulfonic acids are degassed and neutralization is carried out in a continuous flow mixer. The greater proportion of the mixture is conducted in a cycle whereas a smaller proportion is withdrawn as finished product. In the continuous flow mixer the usual buffer substances and preserving agents may be added to the products.

1 Claim, 1 Drawing Figure





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**PROCESS FOR THE MANUFACTURE OF
FLOWABLE PASTY DETERGENTS CAPABLE OF
BEING PUMPED**

This application is a continuation of application Ser. No. 795,868, filed Feb. 3, 1969 and now abandoned.

The present invention relates to a process for the manufacture of flowable pasty detergents capable of being pumped by neutralizing with alkalis sulfuric acid semi-esters or sulfonic acids, which pasty detergents have a high content of washing active substances, especially more than 60 % of washing active substance.

By the term "organic sulfuric acid semi-esters or sulfonic acids" there are to be understood semi-esters of fatty alcohols and of hydroxy-ethers of fatty alcohols and alkylarylsulfonic acids and olefin-sulfonic acids as well as the product mixtures obtained in the olefin sulfonation which are known as anionic constituents of raw materials for detergents. There are mentioned by way of example semi-esters of fatty alcohols having a chain length of 12 to 18 carbon atoms, oxyethylation products thereof containing 1 to 5 moles of ethylene oxide per mole of fatty alcohol, fatty acid and fatty acid amine oxyethylation products having the same chain length and the same content of ethylene oxide as well as mixtures thereof, aryl and alkylarylsulfonic acids having straightchain or branched alkyl radicals with up to 12 carbon atoms and olefin-sulfonates having a chain length of 15 to 22 carbon atoms, or mixtures of the aforesaid classes of compounds. All these raw materials may be of natural or synthetic origin.

Commercial raw materials for detergents capable of being pumped contain a relatively low proportion of washing active substance. Concentrated detergents having a content of washing active substance of up to about 60 % are rather stiff pastes which cannot be pumped with tolerable expense. It is, therefore, impossible to store them in tanks or to transport them in tank cars without considerable costs.

The stiff consistency of the pastes is a result of the high air and electrolyte content originating from the manufacturing conditions. The air content can be reduced by prolonged storage, however to a limited extent only. To prepare pastes having a low content of electrolyte it is necessary to neutralize the organic sulfuric acid semi-esters or sulfonic acids in small quantities with careful control. This procedure considerably increases the price of the products.

The present invention provides a process for the manufacture of pasty detergents containing a high proportion of washing active substance and capable of being pumped by neutralization of organic sulfuric acid semi-esters or sulfonic acids with alkalis which comprises degassing the organic sulfuric acid semi-esters or sulfonic acids before they are neutralized, mixing them with the alkali and optionally other additives in a continuous flow mixer and thereby neutralizing them, after the mixture has left the continuous flow mixer withdrawing a smaller proportion thereof as finished product and recycling the larger proportion thereof as cycle product through a cooler and into the continuous flow mixer.

The ratio of withdrawn finished product to cycle product is in the range of from about 1 : 5 to about 1 : 30, advantageously about 1 : 10 to about 1 : 20.

The process of the invention is now described in greater detail with reference to the accompanying drawing.

The organic sulfuric acid semi-ester or the organic sulfonic acid used as starting product is degassed in degassing device 1. The degassing device is suitably a vacuum degassing machine with rapidly rotating atomization means. The degassing is especially important with starting products which have been produced with the use of gas mixtures containing SO₂ or SO₃.

The degassed starting product is passed over a metering device 2 into continuous flow mixer 3 into which the alkali and optionally other common additives, for example buffer substances or preserving agents and the like, are introduced through further metering devices 4 and 5 that can be regulated in dependence of the pH value of the product by means of pH measuring device (8). After the product has left the continuous flow mixer a smaller proportion thereof is withdrawn through conduit 9 as finished product, whereas the greater proportion thereof is recycled as cycle product into continuous flow mixer 3 through circulation pump 6 and heat exchanger 7.

As continuous flow mixer 3 a toothed disk mixer is suitably used. It is likewise possible, however, to use a mixer with pronounced conveying effect, such as a mixing pump. In the latter case, circulation pump 6 can be dispensed with. The finished product can also be withdrawn through conduit 9a after the circulation pump, this being of special advantage when after withdrawal the finished product must be conveyed through a long conduit.

As heat exchanger 7 a plate cooler is preferably used in order to maintain low the pressure loss.

The process of the invention has a number of advantages over the known processes. When the detergent is neutralized in a continuous flow mixer instead of, for example, in a kneader, air is practically not introduced into the paste. The increase in temperature during the neutralization is kept within moderate limits by the high proportion of cycle product. Thermal damage of the paste which causes the formation of electrolyte is thus substantially avoided.

The following example serves to illustrate the invention but it is not intended to limit it thereto.

Example

In a device as specified above, 0.66 kilogram of lauryldiglycol ether-sulfuric acid semi-ester was neutralized with 0.33 kilogram of sodium hydroxide solution of 25 % strength with the addition of 0.04 kilogram of sodium citrate solution at a cycle temperature of 20°C (behind the heat exchanger) and with a ratio of cycle product to finished product of 15 : 1. 1 kilogram of a paste was obtained having a content of washing active substance according to Epton of 70.1 %, a content of electrolyte of 1.0 % and an iron content of less than 5 ppm. The paste had good flowing properties.

Commercial pasty detergents generally have a content of electrolyte of 4 to 5 % and a content of specific detergent of 60 %. They constitute stiff pastes that are hardly flowable.

What is claimed is:

1. In a process for the manufacture of flowable pasty detergents capable of being pumped by neutralization of organic sulfuric acid semi-esters or organic sulfonic acids selected from the group consisting of sulfuric acid semi-esters of fatty alcohols having a chain length of 12

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to 18 carbon atoms, oxyethylation products thereof containing 1 to 5 moles of ethylene oxide per mole of fatty alcohol, fatty acid and fatty acid amide oxyethylation products having a chain length of 12 to 18 carbons and containing 1 to 5 moles of ethylene oxide per mole of said fatty acid or fatty acid amide as well as mixtures thereof, aryl and alkylaryl-sulfonic acids having straight-chain or branched alkyl radicals with up to 12 carbon atoms, olefin sulfonic acids having a chain length of 15 to 22 carbon atoms, and mixtures of the aforesaid classes of compounds, the improvement which comprises removing gases by application of vac-

uum from the organic sulfuric acid semi-esters or sulfonic acids before they are neutralized, neutralizing them with an alkali in a continuous flow mixer, withdrawing a smaller proportion of the mixture as finished product after it has left the continuous flow mixer, and recycling the larger proportion of the mixture as cycle product into the continuous flow mixer through a cooler, the ratio of said withdrawn finished product to said cycle product being in the range of from about 1:5 to 1:30, the resulting finished product having a washing active substance content of 60 to 70 percent.

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