Patented Feb. 16, 1932 1,845,461

UNITED STATES PATENT OFFICE

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PROCESS FOR RENDERING ODORLESS SOAPS BLEACHED WITH HYPOCHLOROUS ACID

No Drawing. Application filed May 12, 1931, Serial No. 536,935, and in Germany October 31, 1930.

Of all the soap bleaching agents which depend on oxidation, alkali hypochlorite shows the best bleaching effect. That this agent has not hitherto been more extensively applied in the soap industry is due to the fact that the soaps treated with hypochlorous acid acquire after the bleaching a disagreeable, very persistent odor similar to that of carbolic. Prolonged boiling, repeated salting out, steam blowing, addition of reducing agents, or the like, have not here led to the desired result; only the process already previously proposed by the applicant himself, i. e. directly separating out the fatty acids from the bleached soaps by addition of mineral acid, has frequently enabled the preparation of a satisfactory fatty acid free from this carbolic odor.

It has now been ascertained that the carbolic odor may be completely removed by raising the soap after bleaching to a high temperature in a pressure vessel by leading in steam with addition of an excess of alkali. Normally, a pressure of 5 atmospheres is sufficient for this purpose, but frequently the odor clings so persistently to many soaps that one is compelled to raise the pressure to 8–10 atmospheres, or, in special cases, still higher. The pressure necessary depends on the constitution and purity of the fatty acids on the one hand and on the alkali excess of the soap on the other hand. Usually a caustic alkali excess of a few percent is sufficient. Alkali carbonate may also be used with equal success, but in this case the pressure must be raised. The carbolic acid odor appears particularly strongly during the bleaching of coconut oil soaps, and up to now it has been impossible to remove it completely, so that hitherto one dared not bring soaps containing coconut oil into contact with hypochlorous acid.

It has now further been ascertained that if, in the pressure treatment aforedescribed, the pressure is taken sufficiently high, even coconut oil soaps may be freed from the disagreeable carbolic odor by heating with an excess of alkali. However, it has also been found that the same result may be obtained without one being compelled to operate at high pressure and high temperature, if small quantities of ammonia, or, better, of ammonium salts are added to the soap instead of, or as well as, the other alkali, as the result of which the carbolic acid odor already completely disappears at comparatively low temperature.

This fact justifies the assumption that the supposed carbolic odor is produced by chlorinated by-products, and that during the process according to this invention, these by-products are converted into hydroxy- or amino-compounds by the action of alkali or ammonia under pressure.

The new process has up to now been applied with satisfactory success in the case of all the known fatty acids.

Example.—10 t. of grain soap are made from about equal parts of dark ground nut oil fatty acid and waste coconut-palm kernel oil fatty acid in a vessel which is preferably lined with clay plates and provided with a good stirring mechanism. After good, if necessary repeated, salting out and removal of the lower layer of lye, bleaching is then effected with chlorine lye in the usual way after the temperature has fallen to 60–80° C. The ordinary chlorine lye which contains about 150 g. of chlorine per litre and which is preferably somewhat diluted, is here used. If during the supply of the lye, whilst the stirring mechanism is running the soap should become too thick, some salt or even lye is added to the soap as well. After, according to the purity of the fats, about 5–10% of chlorine lye, calculated on the saponified fat, have been used, this soap assumes an approximately white colour. It is now allowed to stand for a short time and is allowed to flow into a deep iron pressure vessel having a capacity of about 12 cbm.; about 1–3% of soda lye of 38° Bé. calculated on the soap or the equivalent quantity of solid soda are then added and, after closing the vessel, the whole is heated for 4–5 hours by means of a current of steam streaming in, until a pressure of 8–10 atm. is reached. The chlorine bodies formed are now completely decomposed and a test portion is free from odor. The contents of the vessel may then be forced

into another soap vessel again under their intrinsic pressure and then the soap is finished in the usual manner. In those cases in which fats have been heated having a particularly strong carbolic odor, for example coconut oil, some ammonium salt e. g. ammonium sulphate is also added as well, (about 5 kg. in the foregoing example) after which there is no longer any fear of the finished soap subsequently showing the carbolic odor again.

By means of this treatment a very cheap removal of the odor is produced. If merely alkali is used in the process, the latter proceeds perfectly smoothly, without any disturbance and also without danger. On the other hand if ammonia or ammonium salts are used then as far as possible only those quantities of ammonia must be added to the soap which are just necessary (to be ascertained practically by experiments), since too great an excess in the pressure vessel produces a gas pressure, due to ammonia, which could exceed the normal strength limits of the vessel. This can be recognized by the fact that the pressure prevailing in the pressure vessel no longer agrees with the corresponding temperature of the soap material. If this is avoided, the operation with ammonia also is completely free from danger, and proceeds smoothly and simply. In place of ammonia substitution products thereof, such as, for example, methylamine, dimethylamine, trimethylamine, or even aniline, or methylaniline etc., may also be used, provided these do not exert any detrimental effect or can be readily removed again. It may also be remarked that ammonia is used in all cases in which the odor persists so obstinately that it cannot be removed with soda or caustic soda.

It must be particularly emphasized that the process here described relates to the treatment of soaps merely with that quantity of hypochlorous acid which is necessary for producing sufficient bleaching, and not to the attachment of hypochlorous acid to the unsaturated linkages of the fatty acids contained in the soap owing to the addition of large quantities of hypochlorous acid, in which case, as is well known, the chlorine is eliminated by the action of alkali or ammonia. The production of hydroxy-fatty acids or amino-fatty acids is not the purpose of the process here described, but only the production of an odorless bleached soap.

On the other hand the object here aimed at is not attained if, as has been proposed from other quarters, steam is blown through the aqueous liquid soap under pressure at temperatures of about 200° C. or above. This process can only be used when the odor is brought about by readily volatile substances and not substances bound to alkali.

What I claim is:

1. Process for removing the odor from soap bleached with hypochlorous acid consisting in heating the bleached soap under pressure with small quantities of excess alkali.

2. Process for removing the odor from soap bleached with hypochlorous acid consisting in heating the bleached soap under pressure with small quantities of excess caustic alkali.

3. Process for removing the odor from soap bleached with hypochlorous acid consisting in heating the bleached soap under pressure with small quantities of excess alkali carbonate.

4. Process for removing the odor from soap bleached with hypochlorous acid consisting in heating the bleached soap under pressure with small quantities of excess ammonia.

5. Process for removing the odor from soap bleached with hypochlorous acid consisting in heating the bleached soap under pressure with small quantities of excess ammonium salt.

6. Process for removing the odor from soap bleached with hypochlorous acid consisting in heating the bleached soap under pressure with small quantities of excess alkali together with another alkali.

7. Process for removing the odor from soap bleached with hypochlorous acid consisting in heating the bleached soap under pressure with small quantities of excess alkali together with ammonia.

8. Process for removing the odor from soap bleached with hypochlorous acid consisting in heating the bleached soap under pressure with small quantities of excess alkali together with an ammonium salt.

9. Process for removing the odor from soap bleached with hypochlorous acid consisting in heating the bleached soap under pressure with small quantities of a substitution product of ammonia.

10. Process for removing the odor from soap bleached with hypochlorous acid consisting in heating the bleached soap under pressure with small quantities of a salt of a substitution product of ammonia.

11. Process for removing the odor from soap bleached with hypochlorous acid consisting in heating the bleached soap under pressure with small quantities of excess alkali together with a substitution product of ammonia.

12. Process for removing the odor from soap bleached with hypochlorous acid consisting in heating the bleached soap under pressure with small quantities of excess alkali together with a salt of a substitution product of ammonia.

13. Process for removing the odor from soap bleached with hypochlorous acid con-

sisting in heating the bleached soap under pressure with small quantities of excess alkali together with aniline.

14. Process for removing the odor from soap bleached with hypochlorous acid consisting in heating the bleached soap under pressure with small quantities of excess alkali together with methyl aniline.

15. Process for removing the odor from soap bleached with hypochlorous acid consisting in heating the bleached soap under pressure with small quantities of excess alkali together with methylamine.

16. Process for removing the odor from soap bleached with hypochlorous acid consisting in heating the bleached soap under pressure with small quantities of excess alkali together with dimethylamine.

17. Process for removing the odor from soap bleached with hypochlorous acid consisting in heating the bleached soap under pressure with small quantities of excess alkali together with trimethylamine.

In testimony whereof I have signed my name to this specification.

ADOLF WELTER.