ABSTRACTS

Oils and Fats

Edited by
W. F. BOLLENS and M. M. PISKUR

PATENTS

Wool-fat alcohols. Deutsche Hydrierwerke A.-G. (Ludwig Prigge and Karl Hennig, inventors). Ger. 617,975, Aug. 30, 1935 (Cl. 12o. 5.02). Alcs. incapable of sapon. are obtained from wool fat and other high-mol. wax-like substances by sapong. the wool fat, etc., treating the product with at least 30% of a soap with no more than 18 C atoms in the mol., and finally distg. Thus, wool fat is sapond. with NaOH and water and palmitic soap, obtained by treating palm oil with NaOH, added. The mixt. is distd. with superheated steam to give a product of wool-fat alc. (C. A. 30, 893.)

Purifying fatty oils. Karl Merck, Louis Merck, Wilhelm Merck and Fritz Merck (trading as (firm of) E. Merck). Brit. 434,432, Sept. 2, 1935. Tunny-fish liver oil is freed from undesirable color, taste and odor by treating at ordinary temp. with aq. or alc. alkali and then sepg. the purified oil. The alkali removes the larger proportion of free fat acids without sapon., or loss of vitamin A. (C. A. 30, 894.)

Oils; vitamins. Aktieselskabet Ferrosan. Brit. 433,930, Aug. 22, 1935. The vitamin-contg. unsaponifiable components of fats and oils are extd. or concd. by sapong. the oil with a soln. of NH_3 or alkali metal hydroxide in a H_2O-sol. org solvent, e.g., Me_2CO, MeOH, EtOH, cooling or standing to sep. the soap and acidifying the residual soln. to ppt. fat acids from the soap which remains dissolved, said acids collecting as a layer with the unsaponifiable components. (C. A. 30, 894.)

Neutral bleached edible oils and fats. Wilhelm Gensecke (to American Lurgi Corp.). U. S. 2,003,076, May 28. After sepg. the albuminous and mucilaginous matter from a colored edible oil or fat such as a crude maize oil, the free fat acids are sepd. by neutralization with alkali, and the oil or fat is heated to about 280° for a sufficient time to effect bleaching, free fat acids being formed and distd. off in a vacuum with the aid of an inert gas.

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Soaps

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Medicinal Soaps. P. Martell, Allgem. Oel-u. Fett. Ztg. 32, 366-8 (1935). Tar and S-containing soaps are recommended for skin diseases, dandruff and falling hair. The S-containing soaps are also recommended for use by workers in metal industries, especially those who work with lead compounds. The addition of $HgCl_2$ to soap has the disadvantage of producing a grayish off-colored product; however, this soap finds application in disinfection of hands before operations and also for disinfecting surgical instruments. Other antiseptic soaps mentioned contain phenols, salicylic acid, formaldehyde or peroxides.
(C. A. 30, 1, 318, January 10, 1936.)

Lathering Power of Soaps. P. N. Das Gupta, Indian Soap Journal, 2, 2, 2, 1935. P. N. Das Gupta discusses the lathering power of soaps made from numerous oils and combinations of oils. The article contains a very useful tabulation of lathering indices.
(American Perfumery and Essential Oil Review, 31, 3, 98, November, 1935.)

The Oils, Fats and Soaps Industries in 1935. R. Furness, M.Sc., The Industrial Chemist XII, 132, 23 (January, 1936). It has been found by the I. G. Farbenindustrie A. G. that a product from paraffin wax (or other suitable mineral oil distillate) obtained by air oxidation at 120 to 150° C. in the presence of a suitable catalyst such as, for example, a manganese soap, can be made to furnish reasonably high quality soap-making fatty acids. The process consists in dissolving the oxidation product in a suitable solvent and removing therefrom, by means of saponifying alkali solutions, the fatty acid components.

The unreacted wax, etc., together with the oxidized products which are unsuitable for soap-making, remain in the solvent and may be recovered for re-use. The soap solution may be suitably worked up to relatively high quality products.

Again, with a view to improving the yields of useful detergent materials, the same company takes the crude mass resulting from the oxidation of wax, mineral oil distillates, etc., and subjects it to catalytic hydrogenation under conditions of high temperature and pressure such as are employed, for example, in ammonia synthesis, coal liquefaction, etc. By these means, which are now of course technically feasible, the aldehydic and ketonic portions of the oxidized hydrocarbons are converted into alcohols, whilst, although it is possible also to reduce the carboxylic compounds present in the oxidation product to alcohols by this catalytic process, the fatty acids remain as such. Thus, from an original hydrocarbon mixture, primarily converted by oxidation into a complex mixture of alcohols, esters, ketones, aldehydes, acids, anhydrides, etc., together with unconverted hydrocarbons, a hydrogenated product is obtained consisting mainly of alcohols, acids and unchanged hydrocarbons. This latter mixture is now sulphonated in such a way that only the alcoholic groups are affected so that the subsequent treatment of the sulphonated mass with alkali solution leads to the formation of a solution of alkali soaps and alkali salts of the sulphuric acid esters of higher alcohols—the unreacted hydrocarbons being removed for further

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use. It is claimed that the detergent mixture, when dried to a powder, is an attractive product.

Fatty acids from natural fats are at present required not only for soap production, but also for the production of the newer so-called "soapless" detergents of the cardinal and Igepon type, since they serve as raw materials for the catalytic reduction to the higher alcohols (which when sulphonated lead to preparations of the cardinal type) and also as integral parts of the sulphonated ethylene glycol-fatty acid condensation products of the Igepon type. It is not impossible that the higher alcohols will eventually be economically produced by the catalytic oxidation of higher boiling petroleum hydrocarbons.

Fatty Alcohol Sulfates Disappoint. Oskar Uhl, Seifensieder-Ztg. 62, 687-9, 719-20 (1935). Practical laundry tests in which the detergent efficiency of the fatty alcohol sulfates was compared with that of laundry soaps, indicated that the fatty alcohol sulfates do not always live up to expectations. Because of their high price as compared with soap it is claimed that they can be used in much smaller concentration, for example, one-sixth that of soap. Particularly at this low concentration the comparison becomes decidedly unfavorable. It is believed that in laundering certain classes of soiled material such as white goods, the very lack of alkalinity in the synthetic detergents, which is so much talked about, becomes a disadvantage as far as detergent efficiency is concerned. Used on white goods at or near the boiling point, the result is apt to be a gray finish. The conclusion is reached that the ideal detergent is yet to be found, since the synthetic products, either alone or added to soap, still leave something to be desired.

(Soap XII, 1, 71, January, 1936.)

Rosin Standards Modified, Effective Next May. Chemical and Metallurgical Engineering 42, 12, 693 (December, 1935). Rosin standards for the United States have been modified by order of the Secretary of Agriculture, the change to take place on May 6, 1936. The modification is in accordance with the procedure which has been prescribed by the provisions specified and enumerated in the Naval Stores Act.

Changes and improvements in the standards are briefly: Use of a more suitable glass to obtain light transmission qualities of "color composition" more nearly like that of rosin itself; higher brightness for most of the standards nearer that of clean well-strained rosin; more solid, serviceable assembly by cementing the glasses together and by eliminating the metal border around the faces of the standards to obtain a larger viewing face; and respacing of most of the standards to give a more systematic color difference. At present this color difference is too small in some cases, too great in others. Standards for X, N, M, K, I and H will be slightly redder than at present. Those for G, F, and E will be a little less red.

pH Determination and Control Discussed at Meeting of the Toronto Chemical Association. Soap XII, 1, 53 (January, 1936). J. W. McCutcheon of Lever Brothers, Ltd., Toronto, Canada, was a speaker at a recent meeting of the Toronto Chemical Association at which pH determination and control was discussed by several members. Mr. McCutcheon described the control of pH as a means of insuring the correct conditions for the removal of organic material from spent lye in the production of crude glycerine. The lye is acidified to pH between 3.4 to 6.2, depending on the nature of impurities to be removed, and aluminum and iron salts added. The metallic soaps are most completely precipitated at pH 6.2, but are then very difficult to filter. A compromise must therefore be made between precipitation and filtration characteristics. The complete removal of the aluminum and iron is accomplished by addition of alkali to pH about 7.4.

Soap-Making Materials from Petroleum. *Perfumery and Essential Oil Record,* 26, 9, 362 (September, 1935). We have referred from time to time to the use in the soap industry of naphthenic acids—acids obtained as a by-product in the refinement of petroleum, and though at one time owing to their unpleasant odor they could only be used in the cheapest kinds of soap, improved methods of purification have now rendered them much more useful material for soap-making. In many of their chemical properties they closely resemble coconut oil fatty acids, and in some cases they also have an odor somewhat suggestive of coconut fatty acids, but some of the naphthenic acids of American origin are now so well refined as to be odorless.

Where they are suspected in a soap, the best method for their detection is to carry out an ordinary Polenske determination on the fatty acids from the soap, and to the neutralized Polenske acids add a small quantity of copper sulphate solution. The precipitated copper soaps are filtered off and dried, and then tested for solubility in petroleum spirit. Copper naphthenate is soluble in petroleum spirit so that if the soap contained naphthenic acids, a green solution is obtained.

The shortage of ordinary soap-making fats and oils in Russia has led the Soviet authorities to seek to augment these by fatty acids obtained by the oxidation of petroleum. This subject has attracted research workers for many years, from 1884 onwards, with but little success though some progress has been made using paraffin wax as the starting point, and oxidizing it either with air in the presence of some catalyst, or with ozonized air. Two large research stations have now been organized in Russia to carry out intensive investigation of the subject, and an account of their work is given by Heublum ("Seifen. Zeit.," 1935, 421-3, 443-4). Blowing air through vaseline oil for about 48 hours was first tried, but the yield of acids was small; an improved yield was obtained by working under a pressure of about 50 atmospheres, in contact with a 2N. solution of caustic soda, a method used by Fischer and Scheider ("Chem. Zeit.," 1921, 327). Various catalysts are being experimented with, and so far calcium and magnesium naphthenates have been found more efficient than the stearates. The soaps obtained from these oxidation products appear to be inferior to those prepared from ordinary fats and oils.

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A Volumetric Determination of Fat Acids in Soap. W. Stüwe, *Chem.-Ztg.* 59, 468-9 (1935). A 100-cc. conical flask having a neck holding 6 cc. graduated to 1/100 cc. is used for the determination. About 3.5 g. of olein equal to 3.91 cc. is weighed into the tared flask. About 2 g. of the soap sample is dissolved in H_2O and transferred to the flask, the soap is decomposed with RCl and 15 g. NaCl is added. The fat acids mixed with the olein rise to the top and are forced into the neck by adding warm saturated NaCl solution. The flask is placed in a water bath to melt the acids and after cooling to 20° the volume is read and the volume of olein deducted. The percentage of fat acids is calculated by multiplying the volume by the specific gravity of the fat acids and dividing by 2. Thus for common fats whose composition is known the specific gravity of the fat acids is obtained by multiplying the percentage of each fat acid in the fat by its specific gravity and combining them. The method is not highly accurate, but is suitable for practical work. (C. A. 29, 16, 5685, August 22, 1935.)

Lecithin Soaps. Dr. Karl Braun. *Deutsch. Parf. Zeit.,* 1935, 238. Although Lecithin is known to have a beneficial effect when properly incorporated in soaps, improving both their therapeutic and detergent properties, it is sometimes difficult to form a homogeneous mixture with the soap owing to the wax-like nature of the lecithin. A new lecithin preparation is intended to overcome this difficulty and facilitate the incorporation of lecithin in soaps. A high disperse substance is added to the lecithin whereby it is rendered much more liquescent, and a 55 per cent product of syrupy consistency may be obtained which may be easily and effectively mixed with a soap. A soap is thus obtained which has marked cleansing power and a very agreeable action as a toilet soap, with remarkable lathering properties. (*Oil and Colour Trades Journal* 88, 1932, 1247, October 25, 1935.)

Soap Used in Production of Gramophone Records. *The Industrial Chemist* 129, 11, 400 (October, 1935). It is found that the introduction of various soaps confers desirable properties on the wax used for making gramophone records. Great elasticity, as well as toughness and durability, are thus obtained. The production of soaps naturally needs waxes of an acid nature, and it was found that very good results are obtained by using ordinary distillation stearine with a melting point of 120/2° F.

Soaps are introduced by heating the stearine with the oxides, hydroxides or carbonates of the metals whose soaps are required, and the product is known as recording wax. The metals used mainly for this purpose are lead and sodium, and each of these produces a definite effect on the wax. The lead soaps act as hardeners and destroy the crystalline structure of stearine, whilst the sodium soaps give transparency, elasticity, and minimum shrinkage. This combination, when used in the correct proportions, gives a wax which has the required properties for recording.

For the production of brown masses the wax can be colored by the addition of up to 2 per cent of fatty acid pitch depending on the shade required. A black wax can be made by this addition of pitch and a little fat soluble nigrosine.

"Free Fat" in Soap. *Perfumery and Essential Oil Record,* 26, 8, 322 (August, 1935). A Sub-Committee of the Society of Public Analysts was appointed a year or two ago to recommend a process for the determination of free fat, which might be generally adopted.

The work of the sub-committee has resolved itself into two parts, (1) the determination of free fatty acids, and (2) the determination of unsaponified neutral fat. For the first, various methods have been tried, but the sub-committee finally recommends solution of the soap in 95 percent alcohol, and, without filtration, titration at 70° C. with N/10 alcoholic potassium hydroxide solution, using 0.5 cc. of a 0.5 percent solution of phenol-phthalein as indicator. It is pointed out that if the solution is filtered, a slightly high result may be obtained, owing principally to absorption to carbon dioxide during filtration. In their recommendation to titrate the solution of the soap without filtration the sub-committee are conforming to the views of Davidson and other Continental workers on the subject, and to the practice recommended by Wizoff.

For the determination of unsaponified neutral fat the ether extraction method already recommended by the sub-committee for determination of unsaponifiable matter ("P. & E. O. R.," 1933, p. 142) is adopted, with slight modifications in the washing procedure. About 5 grams of the soap are dissolved in a mixture of 50 cc. of 95 percent alcohol and 100 cc. of water, and extracted successively with 100, 50 and 50 cc. of ether, the mixed ethereal extracts being then washed with 20 cc. quantities of water until the wash-water, when acidified, becomes not more than faintly turbid. The ethereal solution is then washed alternatively with 20 cc. N/2 aqueous potassium hydroxide, and 20 cc. water, until the alkaline extract no longer becomes turbid when acidified. It is then washed with water until free from alkali when tested with phenol-phthalein solution, evaporated to dryness, and the residue dried and weighed. This gives the unsaponifiable matter, together with any unsaponified saponifiable matter, and if, after saponification with 25 cc. N/2 alcoholic potassium hydroxide, the mixture is again extracted in the manner described above, one obtains the unsaponifiable matter only, so that any difference between the results obtained represents the unsaponified saponifiable matter.

Estimation of Perborate in Soap Powders. *Oil and Colour Trades Journal* 88, 1932, 1243 (October 25, 1935). The following suggestions are put forward by van Kregten ("Chem. Weekbl.," 1935, p. 81). The soap powder is added to a 2N solution of sulphuric acid, and the fatty acids extracted with chloroform. The perboric acid in the aqueous layer is determined by (a) titration with standard potassium permanganate, (b) treatment with a known volume of standard N/20 potassium iodide for an hour and the liberated iodine titrated with standard sodium thiosulphate, (c) treatment with an excess of sodium hypoiodite and titrating back the excess with sodium thiosulphate. Of these, method (a) gives the best results.

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PATENTS

Fat Acid Nitriles. French 785,622, August 14, 1935. I. G. Farbenind. A.-G. Nitriles are formed by passing vapors of higher fat acids free from OH groups and capable of forming soaps, e.g., oleic acid, coco-or palm-oil fat acid or functional derivatives of these fat acids, at the same time as NH_3, over a catalyst capable of eliminating water and at a temperature of 320-420°.

(C. A. 30, 2, 491, January 20, 1936.)

Production Fatty Esters of Mono and Di-Glycerides. U. S. 2,022,494. Carl W. Christensen to Armour and Company, both of Chicago. Production fatty esters of mono and di-glycerides.

(Chemical Industries 38, 1, 41, January, 1936.)

Production High Molecular Alcohols and Unsaturated Hydrocarbons. U. S. 2,022,894. Walter H. McAllister, Wyoming Ohio, to The Procter & Gamble Company, Cincinnati, Ohio. Production high molecular alcohols and unsaturated hydrocarbons.

(Chemical Industries 38, 1, 42, January, 1936.)

Processes of Preparing Glycerides. U. S. 2,022,-493. Patented November 26, 1935, by Carl W. Christensen, Chicago, Illinois, assignor to Armour & Company, Chicago, Illinois, a corporation of Illinois. Processes of Preparing Glycerides. In the process of preparing fatty esters of the class of mono and diglycerides the step which comprises heating, at a temperature of about 100° C. to 200° C., a mixture of a higher fatty acid and a glycerate of an alkali-forming metal, the glycerate having at least one free hydroxyl hydrogen.

(Soap XII, 1, 75, January, 1936.)

Soaps; Cosmetics; Germicidal Composition. British 432,689, January 23, 1935. Lever Bros., Ltd., Port Sunlight, Cheshire.— (Lever Bros. Co., Cambridge, Massachusetts, U. S. A.)

Germicidal soaps, cosmetics and similar compositions are made by mixing soaps or similar saponaceous substances with a mercury compound of the general type (R Hg) x R^1 described in the parent specification, in which R is an aromatic nucleus with or without side chains in which the nuclear and side chain carbon atoms are not directly connected to any element other than carbon, hydrogen, or mercury, and R^1 is a radicle containing one or more imido groups, the nitrogen atom of which is directly connected to the R. Hg. group. The aromatic group include polycyclic radicles such as diphenyl-, tolyl-, xylyl- and naphthyl-groups. Mercury compounds of the following compounds are specified: Hydraze benzene, guanidine, phthalimide, o-benzine sulphinide, succinimide, pyrolle, naphthalimide, indole, isatin, carbazole, piperidine, 3-nitro phthalimide, diketopiperazine, acridone, pyrollidine, emetine, 1:3 dimethyl xanthine, barbituric acid, parabanic acid, uric acid, theobromine, alloxan, quinone imide, allantoin; and such dyes as auramine, indigo, and indanthrene red violet. These are mixed in small amount with powdered soap, sodium salts of sulphonated higher fatty alcohols or with the substances sold under the registered trade marks "Gardinol" and "Igepon." Dyes or perfumes may be added. After mixing, the soap may be plodded, milled or stamped.

(The Industrial Chemist Pharmaceutical and Cosmetic Supplement, page 13, January, 1936.)

Wetting, Washing and Dispersing Agents; Soaps. British 433,305, August 8, 1935. I. G. Farbenindustrie A.-G. Oxidation products of nonaromatic hydrocarbons of high molecular weight are dissolved in a H_2O-insoluble solvent or mixture of solvents, the solution is treated with an alkali substance dissolved in H_2O in such amount that the acid portions of the oxidation products are neutralized, the aqueous soap solution is separated and the unsaponifiable constituents may then be extracted from it by means of the same or another H_2O-insoluble solvent. The soap solution may be evaporated to dryness and formed into shaped soaps, soap powder, etc. Among examples, hard paraffin is oxidized with air by heating in the presence of Na and Mn palmitates, the product is dissolved in a mixture of benzine and alcohol and stirred with warm Na_2CO_3 solution, the soap solution is removed after settling, extracted with benzine and evaporated in a roll drier to form a soap powder.

(C. A. 30, 2, 539, January 20, 1936.)

Textile Lubricants. British 431,964, July 18, 1935. British Celanese Ltd. A textile lubricant comprises a substantially H_2O-insoluble oil or fat or higher fatty acid and a small proportion of an aromatic compound containing an unsaturated aliphatic substituent group, e.g., vinyl, propenyl, allyl or crotonyl. The aromatic compound may contain other groups, e.g., OH, ether, halo or SO_3H groups. Substitued PhOH, cresol, naphthol, catechol and resorcinol, e.g., or substituted phenol ethers such as anisole and phenetole may be used, and especially the propenyl, etc., derivatives of aromatic compounds containing both ether and OH groups, e.g., guaethol and guaiacol. Animal or vegetable oils or fats, e.g., olive, castor, coconut, neatsfoot and, in general, glycerides of oleic, stearic, palmitic, or other higher fatty acids, or the acids themselves may be used. Assistants, e.g., mineral lubricating oils, glycol, diethylene glycol, glycerol, etc., may be employed. The lubricant may be added to the spinning solution or to the formed material.

(C. A. 30, 1, 303, January 10, 1936.)

Production of an Alcoholysis Product. U. S. 2,015,145, September 29, 1935. L. P. Hubbuck to E. I. du Pont de Nemours & Company. An ester of an alcoholysis product obtained by heating together a polyhydric alcohol and a hydrogenated castor oil consisting essentially of 12-hydroxystearin, said ester being one in which substantially all of the alcoholic hydroxyl groups of said product are esterified by a monocarboxylic acid.