

UNITED STATES PATENT OFFICE

1,940,372

METHOD OF OBTAINING PHYTOSTEROL,
FATTY ACIDS AND RESIN ACIDS FROM
RAW SOAP OR LIQUID RESIN OBTAINED
IN THE MANUFACTURE OF CELLULOSE
ACCORDING TO THE SULPHATE METHOD

Håkan Sandqvist, deceased, late of Stockholm,
Sweden, by Emma (Emelie) Sandqvist, Stock-
holm, Sweden, administratrix, and Torsten
Olof Hjalmarsson Lindström, Kosta, Sweden

No Drawing. Application August 12, 1931, Serial
No. 556,716, and in Sweden March 22, 1928

2 Claims. (Cl. 87-4)

In the manufacture of cellulose by means of an alkaline digesting liquor (the sulphate method) a saponaceous product, "sulphate soap", is precipitated from the obtained "black lye", said soap containing indifferent substances, resin- and fatty acids in addition to water and alkali. Liquid resin is generally obtained from said soap. Since the beginning of this century tests have been made technically to use the constituents of these products but still no satisfying solution of this problem seems to be at hand.

Among the indifferent substances of the said soap there are inter alia one or maybe two phytosterols. As phytosterols may be extensively employed in obtaining emulsions of a certain type inter alia of hydrated salves, the question of utilizing this soap may be regarded from quite a new point of view, for instance, in the production of water-absorbing salve bases by means of vaseline, paraffine and the like possesses a very high economic value.

According to the invention the raw soap or liquid resin, obtained in the manufacture of cellulose according to the sulphate process is extracted in a dried condition or in a highly concentrated solution by means of a suitable solvent, in which phytosterol and non-saponifiable substances are soluble. In the last-mentioned case the said soap is diluted with a suitable quantity of water for instance 1 to 2 parts, the solution being extracted for example by means of ether, carbon disulphide, carbon tetrachloride, benzol or the like. In the first-mentioned case the said soap is dried at about 100° C. at an extremely reduced pressure, for instance at a pressure below 30 mm. of mercury, and is then obtained in a porous condition being very suitable for the extraction. The extraction is carried out for instance by means of ether. Benzine is unsuitable as an extraction agent, phytosterol being comparatively difficultly soluble in the same. Benzol is unsuitable in the extraction of dried soap, benzol dissolving great quantities of the very soap. The extract obtained in one way or the other is shaken out by means of caustic soda lye, the treatment absorbing dark-colored and smeary impurities, which are then removed, after which the solvent is distilled from the extract and the remainder is recrystallized from alcohol. A white and fine crystallized phytosterol in a quantity of about 2% of the soap employed (with 30% of water) is obtained.

Fatty and resin acids are precipitated from the extracted soap by means of a diluted acid for instance sulphuric acid (whereby sodium

sulphate is obtained as a by-product about 10% of the weight of the soap). The fatty acids are extracted by means of petroleum ether, benzine, ligroin, or other suitable solvents, in which the resin acids are difficultly soluble. The fatty acids are extracted from the obtained acid mixture in its present state or after having been absorbed by some suitable solvent, such as ether and having been filtered and from which the solvent has been removed by distillation.

Having now described our invention, what we claim as new and desire to secure by Letters Patent is:

1. An improved method of obtaining phytosterol, fatty acids and resin acids from raw tall oil soap obtained in the manufacture of cellulose by the sulphate method, comprising drying said soap at an extremely reduced pressure, that is at a pressure below that represented by 30 mm. of mercury, extracting the raw tall oil soap in a concentrated state with a solvent in which phytosterol and non-saponifiable substances are soluble, removing impurities from the extract by shaking with an alkaline material, and distilling off the solvent and crystallizing the remainder to obtain pure phytosterol.

2. An improved method of obtaining phytosterol, fatty acids and resin acids from raw tall oil soap obtained in the manufacture of cellulose by the sulphate method, comprising drying said soap at an extremely reduced pressure, that is at a pressure below that represented by 30 mm. of mercury, extracting the raw tall oil soap in a concentrated state with a solvent in which phytosterol and non-saponifiable substances are soluble, treating the residue of extracted soap with dilute acid to decompose the same and precipitate organic acids, treating the precipitated organic acids with a solvent, such as for instance a solvent selected from the group consisting of petroleum ether, benzine and ligroin and in which the resin acids are difficultly soluble but in which the fatty acids are very soluble to separate the fatty and resin acids, and recovering the fatty acids from the solution.

EMMA (EMELIE) SANDQVIST,
Administratrix of the estate of Håkan Sandqvist,
deceased.

TORSTEN OLOF HJAL-
MARSSON LINDSTRÖM.