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PREPARATION OF SAPONINS

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This invention relates to the extraction of saponins from vegetable matter.

The ordinary methods for extracting saponins from vegetable matter also result in the extraction of large proportions of tannins and coloring substances at the same time. The presence of these latter substances in the product make it unfit for many intended uses. To purify mixtures thus obtained, it is necessary to employ a number of complicated purification steps such as precipitation, recrystallization and the use of decolorizing agents.

This invention has for its object to overcome the above difficulties. Another object it to provide a process whereby saponins may be prepared in improved form by means of a simple solvent extraction process. A further object is to provide an improved process for extracting saponins from vegetable matter by means of an organic solvent. Other objects will appear hereinafter.

These and other objects are accomplished by the herein described invention which comprises extracting a saponin from vegetable matter by means of an organic solvent while in the presence of a compound having an alkaline reaction.

Material to be extracted, such as soap bark, is contacted in a chopped, macerated or finely divided form with an organic solvent while in the presence of an alkaline compound. Contact between the substance extracted and the solvent may be brought about in conventional solvent extraction apparatus or the two components may merely be placed in a container and allowed to soak for a period of time, such as over night. The soaking procedure is preferred, since it results in a better recovery of the desired constituent. Repeated extraction with successive portions of solvent may be made in order to complete the recovery.

The alkali may be added to the solvent before it is contacted with the vegetable matter. This procedure is preferred since the alkali is present during the entire period of extraction. Equivalent procedure would be to add the alkali to the vegetable matter prior to the addition of the solvent. The solvent may be added to the vegetable matter and the alkali added subsequently, but extraction of coloring matter and tannin will take place until the alkali has been added. It is, therefore, desirable to have the alkali present as soon as the extraction starts. The amount of alkali used can be varied greatly without materially affecting the extraction process. Proportions of between .1 to 20% of alkaline compound based on the amount of solvent used are generally most useful, and amounts of between 1 and 10% are preferred. With strong alkali proportions in the lower portion of the range mentioned are best while with weaker bases the higher concentrations are more useful. Any substance having an alkaline reaction, such as sodium hydroxide, ammonia, ammonium hydroxide, borax, trisodium phosphate, methyl amine (33% in water), diethyl amine, triethanol amine, etc. may be used.

It is desirable to heat the mixture of solvent, alkali and vegetable material after it has soaked for a period of time, since this procedure increases the yield. For instance the mixture may be heated to boiling temperature and the solvent then separated and filtered to remove suspended vegetable matter. When the soaking procedure is not employed and the material is directly extracted with a solvent, a hot or boiling solvent may be used.

The extract or filtrates from the successive extractions are preferably combined and are then treated by any one of a number of different methods to recover the dissolved saponin. One method of recovery is to completely or partially remove the solvent by evaporation or distillation, preferably under reduced pressure. The evaporation may be continued to dryness, but it is desirable to proceed only to a point where a precipitate first forms. This first precipitate is often of a resinous nature and may be undesirable, in which case, it is removed from the hot solution by filtration. When the concentrated extract is cooled, a portion of the dissolved saponin separates out. Super cooling may be employed to increase the precipitation.

In some cases, the solubility of the saponin in the solvent is such that cooling does not cause much, if any precipitation. In such cases a precipitating agent is added. The precipitating agent should be one which is soluble in the particular solvent containing the saponins but which has little or no solvent action for the saponins. The temperature of the solution at the time of addition of the precipitating agent has a marked effect on the physical properties of the saponin precipitated. Precipitation from a cold solution produces a finely divided precipitate, while that obtained from a hot solution is granular. The former dries to a chalky mass, while the latter dies to a resinous mass. The precipitated saponins are separated from the residual solvent by filtration and dried. If desired, they may be washed with one of the precipitating agents, prior to the drying step. Examples of suitable

precipitating agents are acetone, methyl-ethyl ketone, ethyl ether, and isopropyl ether.

The saponin is dried either in air or, when an especially light colored product is desired, drying in the absence of air, such as in the presence of an inert atmosphere or in vacuo, is recommended. To provide an inert atmosphere inert gases, such as carbon dioxide, nitrogen, or vapors of solvents, such as carbon tetrachloride, may be employed. The product may be ground to fine powder if desired and is ready for immediate use.

The extraction of the saponin from the vegetable matter can be accomplished using any one or a mixture of a large number of organic solvents for the saponin. A preferred type of solvent is a low molecular weight aliphatic alcohol, such as methyl or ethyl alcohol, but other solvents, such as glycerine, ethylene glycol, allyl alcohol, furfuryl alcohol, benzyl alcohol, diacetin, pyridine and ethylene chlorohydrin may be employed. Proportions of solvent may vary greatly as may also the number of extractions made. For the initial extraction proportions of five parts of solvent to one of vegetable matter have been found to be satisfactory. Smaller amounts of solvent can be used for the subsequent extractions.

Example

Pour 5 lbs. of methanol, containing 25 gms. of ammonium hydroxide (28% NH_3), over 1 lb. of soap bark. Allow this mixture to stand 15 hours. Heat to boiling and filter while hot. Evaporate the filtrate to one-fifth its original volume, and filter off and discard the precipitate which appears. Heat the filtrate to 120° F. and add an equal volume of acetone. The precipitated saponin settles out rapidly. Decant the supernatant liquor, add more acetone and decant again. Filter and place the precipitate in a desiccator in an atmosphere of carbon dioxide. Evacuate the desiccator and allow the precipitate to dry over night. Grind the dry mass to a powder and the product is ready for use.

While I have given specific examples of materials which could be extracted, the procedure is applicable to other vegetable substances containing saponins. Examples of other materials are soapwort, soaproot and members of the Smilax family.

What I claim is:

1. A process which comprises extracting vegetable matter containing a saponin with an organic saponin solvent which is substantially free of water, while in the presence of a compound having an alkaline reaction.

2. A process which comprises extracting a member of the group consisting of soap bark, soapwort and soaproot with an organic solvent for the saponin, which is substantially free of water and which contains a compound having an alkaline reaction which is more than feeble.

3. The process for the preparation of saponin which comprises extracting soap bark with an organic solvent for the saponin, which is substantially free of water and which contains a substance having an alkaline reaction.

4. The process of extracting saponins which comprises extracting vegetable matter containing a saponin with an alcohol, which is substantially free of water and which contains a compound having a basic reaction which is more than feeble.

5. A process of preparing saponins which comprises extracting a member of the group consisting of soap bark, soapwort, and soaproot with an alcohol, which is substantially free of water and which contains an inorganic compound having an alkaline reaction.

6. A process which comprises extracting vegetable matter containing a saponin with an aliphatic alcohol, which is substantially free of water and which contains a basic substance.

7. A process which comprises extracting soap bark with an aliphatic alcohol, which is substantially free of water and which contains an inorganic compound having an alkaline reaction, adding a precipitating agent to the extract and separating precipitated saponin.

8. A process which comprises extracting a member of the group consisting of soap bark, soapwort and soaproot with methyl alcohol, which is substantially free of water and to which has been added an inorganic compound having an alkaline reaction.

9. The process of preparing a saponin which comprises extracting vegetable matter containing a saponin with an organic solvent, which is substantially free of water and which contains between about .1 and 20 per cent of a compound having an alkaline reaction.

10. The process which comprises extracting vegetable matter containing a saponin with an aliphatic alcohol, which is substantially free of water and which contains 1–10 per cent of an inorganic base.

11. A process which comprises extracting a member of the group consisting of soap bark, soapwort, and soaproot with an aliphatic alcohol, which is substantially free of water and which contains 1–10 per cent of an inorganic compound having an alkaline reaction.

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