

UNITED STATES PATENT OFFICE

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PROCESS OF TREATING TALLOEL

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Numerous experiments have been made for some time with a view to splitting up talloel into its two fundamental constituents, fatty acid and resinic acid, the economic result hitherto obtained has not been satisfactory however.

The invention is concerned with the same problem, resulting in the separation of the majority of the fatty acid from the talloel (in some cases up to 90%). There are two ways of obtaining this result. One well known method consists in first of all distilling the talloel and thereupon subjecting the distillate to a further fractional distillation.

Applicant's method which is described subsequently is based on esterifying the talloel according to methods which merely effect an esterification of the fatty acids but not of the resinic acids contained in the talloel. for instance the talloel is treated with alcohol and concentrated sulphuric acid; about 100 kg. talloel, 25 kg. alcohol and 4 kg. concentrated sulphuric acid of 66° Bé. The talloel esterified in this manner is then distilled in any known way. The term "talloel" which literally means "pine oil" is a "liquid resin."

As a specific example, I mix 100 kilos of talloel with 25 kilos alcohol and 4 kilos sulphuric acid of 66° Bé and heat the mixture under a reflux for two hours. The reaction mixture is then left at rest until the sulphuric acid layer has separated out as a bottom layer. The sulphuric acid layer is drawn off and discarded.

The ester mixture is now mixed with such a quantity of aqueous caustic soda solution that the unesterified resin-acid and residue of sulphuric acid are neutralized. The end or final point of the neutralization is ascertained by testing with an indicator, for instance, phenolphthalein. A slight excess of caustic soda should preferably be used. The quantity of alkali used depends on the content of resin-acid which considerably varies in talloel and on the content of sulphuric acid.

The cooled solution is then stirred thoroughly twice with benzene or, for instance, with about 25% of the neutralized esterifying mixture. Two layers are formed. The upper layer contains the fatty acid ethyl es-

ters and unsaponifiable matter dissolved in benzene, the lower layer the soda soaps of the resin-acids in aqueous-alcoholic solution.

The fatty acid ester solution is washed out with H₂O or 50% alcohol after separation of the lower layer, then freed from benzene by distillation and now forms a yellow oil, which smells like olive oil.

By careful distillation, for instance, with steam or under vacuum or both, one can separate the fatty acid esters from the unsaponifiable matter and thus obtain the esters absolutely pure.

The alcohol is removed from the aqueous-alcoholic resin soap solution by distillation, the remaining aqueous soap solution is decomposed by diluted sulphuric acid whilst stirring with benzene. The extraction of esters and the unsaponifiable matter takes place only after the neutralization of the resin acids with soda lye. The benzene dissolves the resin acids. After washing the benzene-resin solution with water, the benzene is distilled off. Hard resin-acid remains as residue. The cleavage of the esters into free fatty acid may be effected by boiling with aqueous soda lye while simultaneously recovering the split off or separated alcohol. The acids are separated from the soap by decomposition with dilute mineral acid.

According to the methods referred to it is possible to obtain from the talloel, fatty acids which contain such a small amount of resin acids, that they can be further used industrially, which was impossible according to methods employed hitherto owing to the detrimental content of resin.

Having now particularly described and ascertained the nature of my said invention and in what manner the same is to be performed, I declare that what I claim is:—

1. The process of treating talloel to separate the liquid fatty acid components thereof from the resinic acid components thereof which comprises subjecting the talloel to an esterifying agent to esterify only the liquid fatty acids contained therein and then separating the esterified fatty acids from the unesterified resinic acid components of the esterification mixture.

2. The process of treating talloel to separate the liquid fatty acid components thereof from the resinic acid components thereof which comprises treating the talloel with a mixture of alcohol and sulphuric acid to esterify only the liquid fatty acids, and then separating the esterified fatty acids from the esterification mixture.

3. In the process of treating talloel to separate the liquid fatty acid components thereof from the resinic acid components thereof, the step which comprises subjecting the talloel to the action of an esterifying agent to esterify only the liquid fatty acid components.

4. In the process of treating talloel to separate the liquid fatty acid components thereof from the resinic acid components thereof, the step which comprises subjecting the talloel to the action of an esterifying mixture comprising alcohol and sulphuric acid to esterify only the fatty acid components.

5. The process of recovering the liquid fatty acids occurring in talloel which comprises subjecting the talloel to the action of an esterifying agent to esterify only the liquid fatty acids contained therein, separating the fatty acid esters so prepared from the esterification mixture and then treating the esters with a hydrolytic agent to obtain the free fatty acids.

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

WILLI SCHULTZE.