exposed to its action, which in turn depends upon the degree of subdivision of the seed being extracted.

2. The properties of the pigment glands have been utilized for the development of a method for mechanically fractionating cottonseed into pigment glands, embryo tissue (meal), and hull tissue. The process consists in the treatment of finely divided cottonseed with mixtures of inert liquids having densities intermediate between those of the seed parts being separated.

3. The fractionation method has been shown to be applicable to the preparation of pigment glands, and pigment-free oil and meal from defatted cottonseed.

4. The method has also been shown to be applicable to the separation of pigment glands from defatted cottonseed.

Acknowledgment

The authors wish to express their appreciation to A. L. Merrifield for his cooperation in this investigation, also to T. L. W. Bailey and M. E. Jefferson for the preparation of the photomicrographs reproduced here. We are indebted to J. Winston Neely and W. R. Paden for the samples of pure-bred varieties of cottonseed with which most of this investigation was carried out.

References


Selective Hydrogenation in the Preparation of Purified Oleic Acid From Animal Fats. Elimination of Extremely Low Crystallization Temperatures

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In recent publications from this laboratory procedures for the preparation of purified oleic acid (oleic acid content, more than 90%) from red oil (commercial oleic acid) (1) and tallow (2) were described. The presence of appreciable quantities of polyunsaturated acids in these starting materials necessitated crystallization of the oleic acid from a solvent at extremely low temperatures (-50° to -60° C.), with attendant loss of product. Although crystallization at these low temperatures is probably feasible on an industrial scale, a purification procedure employing temperatures not lower than -20° C. would be less costly and would not require especially elaborate and expensive processing equipment.

If inexpensive mixtures of fatty acids consisting only of oleic, stearic, and palmitic acids, with oleic acid predominating, were available from natural sources, purified oleic acid could be prepared by the relatively simple process of eliminating the saturated acids by solvent crystallization at temperatures between -20° and 0° C. Crystallization of the oleic acid itself would not be required. Unfortunately no such desirable mixture of fatty acids is available from natural sources. It was the purpose of the present investigation to prepare by a simple method from readily available and inexpensive starting materials a mixture of fatty acids which would simulate the hypothetical mixture referred to above and to determine the conditions required to give the best yield of high-purity oleic acid from such a mixture.

The fatty acids required were obtained from selectively hydrogenated inedible animal fats, such as the tallow and greases. Hydrogenation of triglycerides is a well-developed process, and it is possible to reduce the polyunsaturated content of a fat to a low level without appreciably hydrogenating the glycerides of oleic acid. This is illustrated in Table 1, which shows the effect of selective hydrogenation on the fatty acid composition of some typical animal and vegetable fats. When animal fats are hydrogenated under proper conditions, the fatty acids obtained by hydrolysis consist almost exclusively of oleic, palmitic and stearic acids, with only small proportions of isomeric oleic acids and minor quantities of polyunsaturated acids. This is in decided contrast to the vegetable oils, in which selective hydrogenation produces relatively large proportions of isomeric oleic acids. Hydrogenation of the animal fats at 150° C. under a hydrogen pressure of 10 to 15 pounds per square inch and with 0.1% nickel catalyst, is satisfactory.

In the work covered by this report the fatty acids from selectively hydrogenated Brown Grease (Table 1) were employed. The content of polyunsaturated acids was somewhat higher than that of other samples which we have processed, but this material was available in large quantities, and it was satisfactory for the purpose of illustrating the separations involved.
TABLE 1
Effect of Selective Hydrogenation on Fatty Acid Composition of Some Typical Animal and Vegetable Fats

<table>
<thead>
<tr>
<th></th>
<th>Before Hydrogenation</th>
<th>After Hydrogenation</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Iodine Number (%)</td>
<td>Oleic, %</td>
</tr>
<tr>
<td>Inedible tallow</td>
<td>59.9</td>
<td>51.6</td>
</tr>
<tr>
<td>Brown Grease</td>
<td>51.2</td>
<td>45.6</td>
</tr>
<tr>
<td>Cottonseed oil</td>
<td>103.9</td>
<td>27.1</td>
</tr>
<tr>
<td>Peanut oil</td>
<td>97.7</td>
<td>48.3</td>
</tr>
</tbody>
</table>


Two purification procedures were investigated. In one the mixed fatty acids were dissolved in acetone, and the solution was cooled to 0° C. The precipitate thus obtained contained most of the stearic acid originally present in the mixed fatty acids and some of the palmitic acid but only a small proportion of the isomeric oleic acids. The acids recovered from the filtrate were semi-solid at room temperature. In order to prepare high-purity oleic acid from this product fractional distillation was required. Owing to the presence of solid isomeric oleic acids as well as saturated acids the oleic acid obtained by this distillation was also semi-solid at room temperature. Most of these impurities were separated by crystallization from acetone at -20° C. This procedure is summarized in Figure 1.

In the other procedure the acetone solution was cooled to -20° C. The precipitate contained almost all the stearic acid and most of the palmitic and isomeric oleic acids originally present. The acids recovered from the filtrate were liquid at room temperature and were pale yellow. They contained at least 90% oleic acid. For some purposes this was sufficiently pure, but by fractional distillation a product containing at least 95% oleic acid and about 2.5% polyunsaturated acids was obtained. Starting with fatty acid mixtures containing only 0.3% polyunsaturated acids, we obtained final products containing about 97% oleic acid and about 1% polyunsaturated acids. This procedure, summarized in Figure 2, is more satisfactory than that shown in Figure 1 because the stearic acid and most of the solid isomeric oleic acids are removed in one step and the yields are better. In order to remove solid isomeric oleic acids crystallization at -20° C. is required. We have determined that above -15° C. these acids do not precipitate to any great extent from mixtures containing high percentages of oleic acid. Even when crystalli-