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INVESTIGATIONS ON THE BY-PRODUCTS OF THE RICE MILLING INDUSTRY

Part II.—Vitamin E Concentrate from Rice Polishings

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Preliminary examination of the various by-products of the rice milling industry, described in the previous communication,[1] has shown that fine 'kutti' (rice polishing) is richest in vitamin B complex, vitamin E and other nutrients. It yields about 10% of oil with a vitamin E content of 0.16% on the weight of oil, which compares favourably with other natural sources of vitamin E, e.g. corn, cottonseed, soy-bean and wheat germ oils, the tocopherol content of which ranges from 0.1 – 0.3%. Although wheat germ oil is regarded as the main source of vitamin E concentrates, occasional references[2, 3, 4] are to be found regarding the industrial utilisation of rice germ for the production of vitamin E enriched oil, which is finding increasing application as an antioxidant in food technology. It was therefore considered of importance to evolve a suitable method for the production of vitamin E concentrates from fine kutti.

Conventional methods for the preparation of low-potency vitamin E concentrates are based on saponification of the oil with subsequent extraction and concentration of the tocopherols in the unsaponifiable fraction, and they give approximately eight-fold concentration of the tocopherols in wheat germ oil.[5] Direct extraction of wheat germ with hot ethanol followed by low temperature treatment of the extract to freeze out the extracted liquid material, and removal of the solvent also yields a tocopherol-enriched oil.[6] In another method, adsorption chromatography [7,8,9] has been used for the large scale production of tocopherols, while trans-esterification of the rice germ oil with ethyl alcohol using sulphuric acid as a catalyst has been exploited to get 10% concentrates of vitamin E.[4,10] However, the most important industrial method for the concentration of vitamin E from natural oils is the short-path, high-vacuum, molecular distillation method.[2,3] In the present investigation a new method has been devised to prepare from kutti oil a ten-fold concentrate of vitamin E by a simple process based on the insolubility of the sodium soaps in acetone.

Method and Results

The oil, with a saponification value of 179 and an acidity of 60.3% as oleic acid, was extracted with petroleum ether (b. p.40° – 60 °C.) from fine kutti (procured from Abdullah Rice Milling Factory, Larkana, Sind). One hundred grams of this oil was taken up in 130 ml. of acetone, and to this was gradually added a concentrated aqueous solution of 13 g. of sodium hydroxide. The temperature of the mixture rose to about 40 °C., by the heat of reaction which resulted in the neutralization and partial saponification of the oil. Three hundred ml. of acetone was then added with constant stirring. Most of the sodium salts of the fatty acids were precipitated and removed by centrifuging. The precipitate was repeatedly washed with acetone and the washings added on to the acetone solution, which was then kept overnight at 0°C. whereby a further quantity of sodium salts was removed. The solvent was then distilled off and the residue, consisting of oil, unsaponifiable matter, residual soaps, excess alkali and traces

Table 1.—Recovery of Vitamin E with Different Solvents

Process		Starting material		Concentrate		Recovery of vitamin E %
		Wt. of oil	Potency of oil (1 %)	Weight (g)	Potency (% vitamin E)	
Acetone process	Ether	100	0.15	7.61	1.6	81
	Petroleum ether ..	100	0.15	6.60	1.3	57
Normal saponification process ..		100	0.15	9.67	1.1	71

of acetone was designated 'crude concentrate'.

In an effort to obtain the maximum possible extraction of vitamin E from the crude concentrate, a comparison of two possible solvents, ether and petroleum ether, was made under controlled conditions. The soaps and excess alkali were removed by repeated washings with water, the ethereal solution was dried over anhydrous sodium sulphate, and freed of the solvent, the last traces of it being removed in an atmosphere of nitrogen. The results obtained with ether and petroleum ether as solvents are compared in Table 1 with those from the normal hot saponification process.

The concentrate in each case was a dark yellow viscous oil at room temperature. The vitamin E content was estimated by the α,α-dipyridyl-ferric chloride method of Emmerie and Engel,[11] the intensity of the red colour produced being measured on a Hilger photoelecrtic colorimeter using Filter 52(520 mμ).

From the above data it is evident that ether is much more efficient as a solvent than petroleum ether for the recovery of vitamin E from the crude concentrate. It may be noted here that a great advantage of the acetone process described in this paper over the saponification-ether extraction method lies in the fact that all the free fatty acids and a major portion of the glycerides are removed without using higher temperatures, which affect the recovery of vitamin E. Moreover, the subsequent extraction of non-saponifiables and the unreacted oil proceeds more smoothly and requires much smaller quantities of ether, cutting down the manufacturing cost.

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