

was dissolved in dilute hydrochloric acid; treated with charcoal and filtered. The filtrate was made basic with 30% aqueous sodium hydroxide solution in cold (10–15°C), and solid collected by filtration was crystallized from ethanol: yield 3.1 g (\approx 50%), mp 210–12°C (Found: C, 66.58; H, 6.41; N, 27.61%; Calcd. for $C_{10}H_{20}N_6$: C, 66.23; H, 6.50; N, 27.27%).

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POLYMERISATION OF SESAME OIL

UNSATURATED oils can undergo polymerisation by auto-oxidation, heating in air or oxygen, in presence of catalysts like clay, activated earths and metal ions¹⁻⁵. The polymerisation of a number of oils, synthetic fatty acid esters, and glycerides have been studied. In the present investigation, the polymerisation of sesame oil in presence of red lead (Pb_3O_4) has been studied.

Sesame oil was polymerised by suspending red lead (Pb_3O_4) and spreading the mixture in a glass dish. It was allowed to stand at room temperature for 3 weeks. The clear pale yellow oil becomes viscous and orange red in colour. This was decanted and centrifuged to remove suspended red lead.

The iodine number, saponification value, peroxide value and refractive index were determined for the oil before and after reaction according to the AOAC procedures⁶. The intrinsic viscosity was determined in acetone using an Ubbelohde viscometer. Gel permeation chromatography was carried out in a 60 × 1 cm column packed with sephadex LH 20 in ethanol. The eluted products were estimated by dichromate oxidation⁷. The triglycerides of sesame oil before and after reaction were separated by TLC, transmethylated with methanolic HCl and the methyl esters of the fatty

acids characterised by GLC in a varian aerograph series 1400 using a 8' × 1/8" stainless steel column packed with 15% DEGS on chromosorb Q. The individual triglyceride species before and after reaction were separated by argentation TLC⁸.

Sesame oil before reaction has an iodine number of 116 saponification value 210, refractive index at 25°C 1.467 and intrinsic viscosity 0.035 dl/gm. After the reaction the iodine value decreased to 47.5, saponification value increased to 253, refractive index to 1.473 and intrinsic viscosity to 0.058 dl/gm. The peroxide value of the oil after the reaction was 18 mE/Kg whereas that of the oil stored under the same conditions was 67 mE/Kg.

From the gel permeation chromatography, the percentage composition of the oil before reaction was glycerides 80%, free fatty acids 15% and after the reaction the glycerides were 50%, free fatty acids 15% and a new compound whose molecular weight was estimated as 1600 was found to an extent of 30%.

The fatty acid composition of the triglycerides before the reaction on percentage basis is: $C_{16:n}$ 14% $C_{18:0}$ 5% $C_{18:1}$ 39% $C_{18:2}$ 41% and the fatty acid composition of triglycerides remaining after reaction was: $C_{16:0}$ 15% $C_{18:0}$ 7.5% $C_{18:1}$ 52% $C_{18:2}$ 20%.

Argentation TLC separates triglycerides depending on the number of double bonds present in the molecule. For sesame oil triglycerides 9 spots fluorescent in UV when sprayed with 2, 6 dichlorofluoresceine were obtained in a solvent system chloroform : methanol (99 : 1 V/V) whereas for the triglycerides remaining after the reaction only 6 spots were obtained.

Hence it is concluded that the highly unsaturated triglycerides of the sesame oil have undergone polymerisation.

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