A Rapid Color Reaction of Crude Sesame Oil

The new color reaction of sesame oil (*Sesamum indicum*) described by Rao et al. (1) shows that the addition of trichloroacetic acid solution in chloroform to 50% ethereal solution of the oil gives initially a pale blue color and then sky blue and gradually turns to green in a few hours. The reaction for the color formation as explained by the authors is due to methylenedioxyphenoxy group, a sesame oil constituent, by a free radical mechanism in the presence of peroxides, normally formed in oil by autoxidation.

It was much earlier observed by the present author that the addition of concentrated HCl to sesame oil, especially to older samples, gives a dark green color. This reaction was found to be very rapid presumably because the concentrated HCl is stronger than trichloroacetic acid and hence no blue color as intermediate stage was formed. This green color formed immediately after addition of HCl persisted for a long time.

The test is performed as follows: 1 ml of concentrated HCl is added to 1–2 ml of a 50% petroleum ether solution of sesame oil (v/v) and shaken thoroughly. A green color immediately appears which is more pronounced in older samples probably because of higher peroxide content of these samples.

Several trials were made with fresh sesame oil prepared from pure seeds as well as raw sesame oil purchased from market. A comparative study of the two tests confirms that the present test gives a sharp change of color to green and the other test, with trichloroacetic acid, takes a longer time, i.e., several hours to complete the reaction.

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A Simple Method for Preparation of Methyl *trans*-10, *cis*-12 Octadecadienoate

Abstract

A simple crystallization procedure is described for the preparation of *trans*-10,*cis*-12 octadecadienoate from methyl esters of alkali-isomerized linoleic acid.

Conjugated isomers of methyl linoleate are useful model compounds to study the mechanism of hydrogenation. *Trans*-10,*cis*-12 isomer of methyl linoleate has been separated from methyl esters of alkali-isomerized linoleic acid by a series of crystallizations (1). A simple two-step crystallization procedure gives improved yields of *trans*-10,*cis*-12 octadecadienoate from methyl esters of alkali-isomerized linoleic acid.

Fifty grams of methyl linoleate was added to 100 g of distilled ethylene glycol and 26 g of potassium hydroxide which had been heated under nitrogen to 180 C. Heating was continued for 30 min and the isomerized acids were recovered from the reaction mixture in the usual manner. The acids were esterified with methanol and sulfuric acid catalyst. In 556 ml acetone (12.5 ml/g) 44.5 g of distilled methyl esters ($a_{233\,m\mu} = 91.6$; $\frac{a_{10.2\,\mu}}{a_{10.6\,\mu}} = 1.18$) were dissolved and the solution was cooled to −57 to −59 C. The crystalline fraction was redissolved in 200 ml acetone and recrystallized at the same temperature to obtain 16.7 g ($a_{233\,\mu} = 96.2$; $\frac{a_{10.2\,\mu}}{a_{10.6\,\mu}} = 1.30$) of methyl *trans*-10,*cis*-12 octadecadienoate. Gas liquid chromatography indicated that there were traces of the *cis*,*cis* isomer as an impurity and that the *trans*, *trans* isomer was not present. The yield of the product corresponds to 37.5% of the starting material. About 75% recovery has been accomplished by this procedure since the starting material contained 50% of the *trans*-10,*cis*-12 isomer. Reductive ozonolysis (2) followed by gas liquid chromatography indicated 1–2% of $\Delta^{9,11}$ isomer as an impurity. Reduction of a similarly prepared material with potassium azodicarboxylate (3) followed by periodate-permanganate oxidation (4) of the *cis* and *trans* monoenes showed that the double bond at the 10,11-position had *trans* configuration while the 12,13 double bond possessed *cis* configuration.

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Cationic Fatty Acid Derivatives Aid Retention of Starch and Pigment Color in Cellulosic Pulp

Abstract

Cellulosic pulp in water suspension treated with 0.06% selected cationic fatty acid derivatives at pH 4.5 retained up to 99% of gelatinized corn starch and a pigment color.

Inorganic acid and acetic salts of amines and quaternary ammonium compounds derived from fatty acids attach themselves to polysaccharides by complex formation or by physical adsorption (1–3). Several of these compounds have been patented for use as retention aids for starch (4–6) and other anionic additives (4,6,7) in paper manufacture. In a screening program on retention agents for starch in cellulosic pulp we evaluated a number of fatty acid-derived cationic products including some of those stated to be of value for this purpose. We compared the capacities of the most effective of these compounds for retaining gelatinized corn starch and also a pigment color in bleached softwood sulfate pulp at pH 4.5.

The compounds were the quaternary ammonium salts: (A) carbethoxymethyldimethyloctadecyl ammonium chloride synthesized from Armeen DM18D (Armour Industrial Chemical Company, Chicago) (8); (B) the amide and (C) the hydrazide, both prepared from the carbethoxy compound (6,8); (D) hexadecyltrimethylammonium chloride (Arquad 16–50, Armour); and (E) an amine salt, tallow-1,3-propylene diamine (Duomeen T, Armour) diacetate (4,5).

Stock dispersions of corn starch and cationic agent used to evaluate starch and pigment retention were prepared by heating mixtures of 1.5% starch with 0.015% and with 0.03% of cationic agents in water at 95 C for 30 min. Portions of the dispersions were added to 1.2% cellulosic pulp slurries in water with continuous stirring to obtain the desired addition levels. Pulp mixtures were then adjusted to pH 4.5 with 0.5 N sulfuric acid and stirred 5 min longer before filtering by suction on coarse fritted-glass funnels. Suitable portions of the filtrates were analyzed for starch by the method of Browning et al. (9). Starch retained in pulps was calculated by difference.

Cationic efficiencies of the fatty compounds for pigment color were determined by the spectrophotometric method of Mehltretter et al. (10) and reported as the percentages of anionic Halopont blue RNM pigment color (E.I. du Pont de Nemours and Co., Inc.) retained by the treated cellulosic pulp at pH 4.5.

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TABLE I

Retention of Starch and Pigment Color by Cellulosic Pulp[a] Containing Various Cationic Fatty Acid Derivatives

Cationic agent	Addition level,[b] %		Additive retained,[c] %	
	Starch	Cationic agent	Starch	Pigment color[d]
A	3	0.03	84	....
	3	0.06	95	....
	1	0.01	88	....
	0	0.06	....	91
B	3	0.03	84	....
	3	0.06	96	....
	1	0.01	88	....
	0	0.06	....	92
C	3	0.03	83	....
	3	0.06	96	....
	0	0.06	....	90
D	3	0.03	92	....
	3	0.06	99	....
	1	0.01	94	....
	0	0.06	....	92
E	3	0.06	99	99
Control	3	0	50	....
	1	0	60	....
	0	0	....	6

[a] Consistency of cellulosic pulp slurry was 1.2%.
[b] Dry pulp basis.
[c] .... indicates that no starch or pigment color was added and therefore no analyses were made for retention.
[d] Addition level (dry pulp basis) 0.5% where pigment color was used.

The data (Table I) show an extremely high retention of starch and anionic pigment color in cellulosic pulp by addition of only 0.01% to 0.06% (dry pulp basis) of the cationic fatty acid derivatives. These compounds are expected to find application under practical papermaking conditions.

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