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Fourier Transform Infrared Spectra of Freshly Isolated β-Carotene

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The β-carotene from fresh carrots was isolated using column chromatography. The UV spectrum was recorded to ascertain that the separation is complete. An FTIR spectrum recorded in the range 4000–500 cm^{-1} shows the presence of all-*trans*, 7-*cis* and 15-*cis* isomers of β-carotene in the sample. The frequencies of absorption in the spectrum have been identified to the different modes of vibration for the molecule. Another FTIR spectrum of the sample recorded after a week of cold dark storage to identify any changes in the spectrum and to investigate the stability of stucture of β-carotene during storage.

INTRODUCTION

Carotenoids are highly coloured natural pigments found in plants and animals. They are classified under polyene pigments which contain seven to eleven double bonds in their molecular structure. In most carotenoids the central portion of their structure is a long conjugated chain and at the two ends there may be two rings or two open chain structures or one open chain and one ring structure. β-carotene molecule has eleven double bonds nine of which are in conjugation and has β-ionone rings at both ends of the conjugated chain. In nature β-carotene occurs in all green plants often associated with chlorophyll. Carrots are the richest source of β-carotene and contain almost 87% of it.[1, 2]

EXPERIMENTAL

The procedure adopted for the extraction of β-carotene from fresh carrots is the method suggested by Mckone.[3] A weighed sample of smashed carrots is treated with ethanol to remove moisture. The pulp is then filtered using methylene chloride through glass wool and the process of filtration repeated. The filtrate is evaporated by vacuum distillation which results in the carotenoid mixture. The crude carotenoid mixture is chromotographed on a 15 cm column of alumina prepared with ligroin as the solvent. During the eluting procedure many small samples of the yellow β-carotene were collected in test tubes. The solvent was

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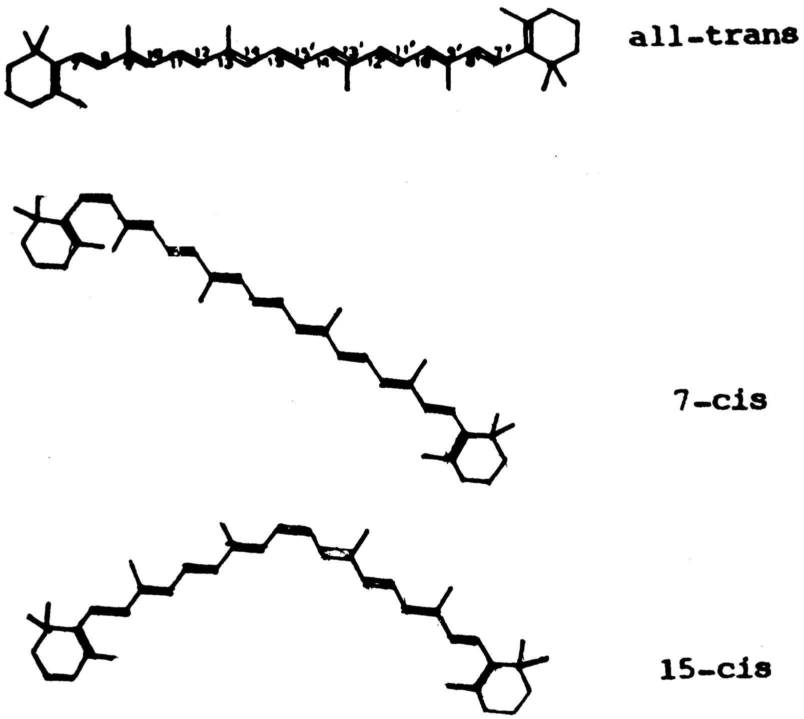

Fig. 1. Structure of the isomers of β-carotene.

evaporated from the extract on a steam bath. UV spectra of several samples collected were recorded to ascertain that the separation is complete. A UV spectrum in the range 700–320 nm recorded on a Shimadzu-160A spectrometer

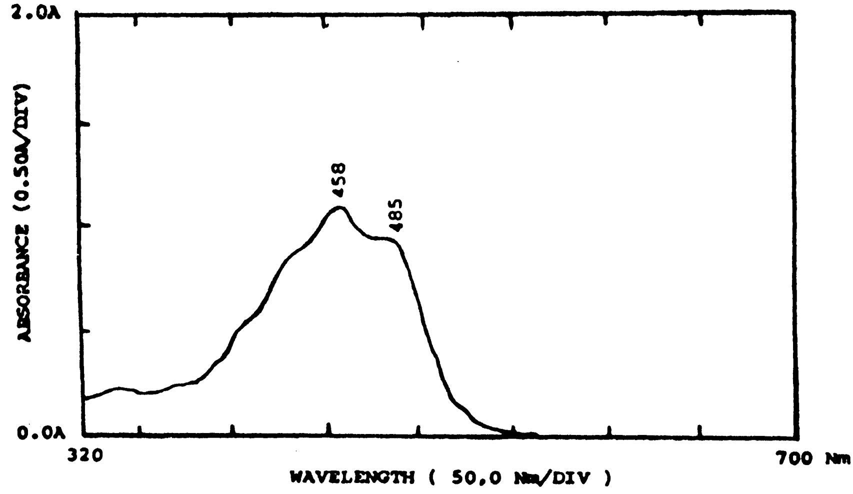

Fig. 2. Ultra-violet spectrum of β-carotene.

is presented Fig. 2. The FTIR spectrum of β-carotene and its storage spectrum recorded in the range 4000–500 cm^{-1} is presented in Figs. 3 and 4 respectively.

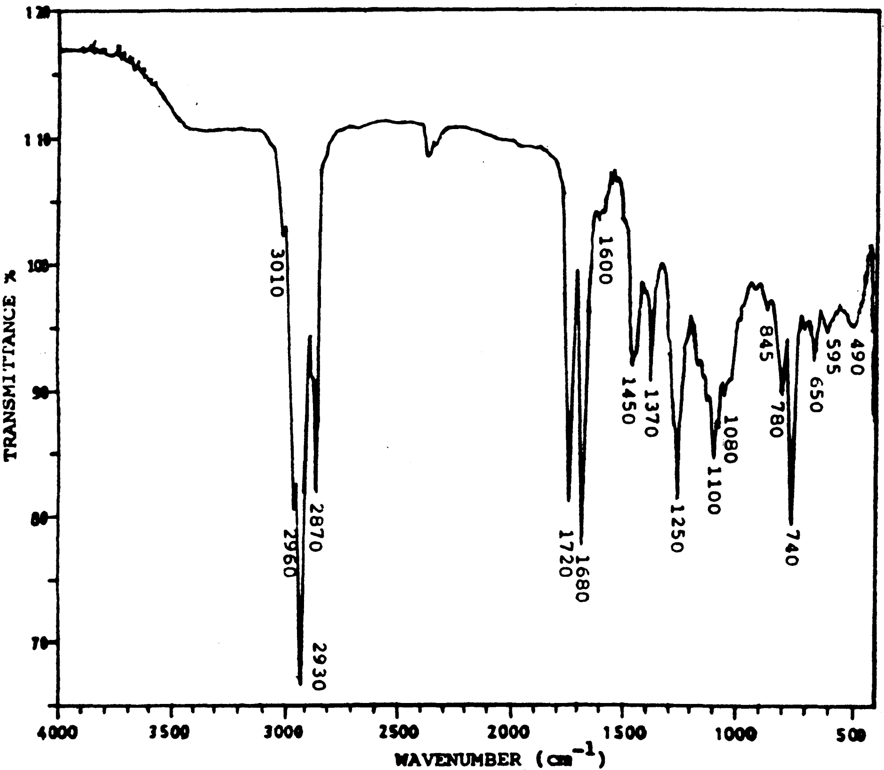

Fig. 3. FT-Infrared spectrum of β-carotene (fresh).

RESULTS AND DISCUSSION

UV spectrum

The ultra violet spectrum of β-carotene recorded shows absorption maximum at 485 and 458 nm. The theoretical calculation of λ_{max} for β-carotene using the Fieser-Kuhn rules is 453.3 nm. The UV spectra of all-*trans*, 7-*cis* and 15-*cis* configurations of β-carotene show absorption maximum at 456 nm and 484 nm.[4]

FTIR spectum

The structure of stretched (all-*trans*), terminal bent (7-*cis*) and central bent (15-*cis*) configurations of β-carotene is shown in Fig. 1. The molecule has β-ionone rings at both ends of the conjugated chain with 4 methyl groups on the polyene backbone and 6 methyl groups on the ionone rings. The β-carotene molecule gives the most stable *cis-trans* configuration among the carotenoids and therefore most suitable for structural analysis. Since the two halves of the molecule have identical substructures which may cause same type of vibrations and leads to considerable coupling and mixing of vibrations. The methyl groups

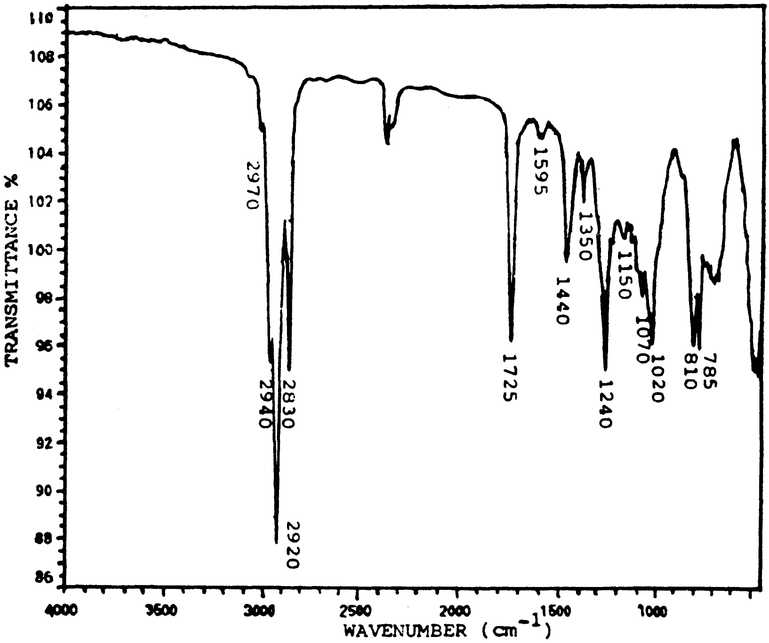

Fig. 4. FT-Infrared spectrum of β-carotene (stored).

also interfere with the above couplings and mixings. The modes of vibration for the molecule can be classified as skeletal and peripheral modes and local and extended modes. The skeletal modes are the vibrations of the polyene backbone which include coupled C═C—C stretchings and C═C—C and C—C—C skeletal deformations. The C—H bendings will have in-plane and out-of-plane modes. The peripheral modes are the vibrations of the methyl groups and the β-ionone rings. If a vibration is confined to atoms within two methyl groups it is called a local mode and those which extend beyond the two methyl groups is called an extended mode. The extended modes are produced through long range coupling and mixing. There is considerable coupling between C═C and C—C stretchings with C—H in-plane bendings and between C—H out-of-plane vibrations with C═C torsions. So it is difficult to make a straightforward assignment of the frequencies present in the spectrum of the molecule. The vibrations belonging to the methyl groups can be identified while the vibrations belonging to the β-ionone rings are extremely weak in the infrared spectrum. β-carotene has two halves which correspond to the structure of retinal whose vibrational analysis[5, 6] is useful in the interpretation of the spectrum of β-carotene.

In the spectum of β-carotene the key infrared bands at 1250 cm^{-1} and 740 cm^{-1} are characteristic of 7-*cis* configuration and the IR band at 780 cm^{-1} is characteristic of 15-*cis* configuration isomers being present in the extraction from fresh carrots. All isomers specifically *cis-isomer* of β-carotene give two characteristic coupled C═C—C stretchings at 1720 cm^{-1} and 1680 cm^{-1} in the IR spectrum. The C—H in-plane bendings coupled with C═C and C—C stretchings are in the region 1350–1100 cm^{-1}. A series of small absorption peaks can be seen centred at about 1100 cm^{-1} due to C—H in-plane bendings. The key infrared bands at 780 cm^{-1} and 740 cm^{-1} corresponding to the 15-*cis* and 7-*cis* isomers respectively are assigned to the C—H out-of-plane waggings. The weak absorption at 845 cm^{-1} also belongs to this mode of vibration. The strong absorption at 2930 cm^{-1} is assigned to —C—H stretchings[7] and the weak absorption at 3010 cm^{-1} is due to ═C—H stretchings. The molecule has methyl groups attached to carbon atoms which give rise to symmetric and asymmetric stretching and bending modes classified as the peripheral modes. The CH_3 symmetric and asymmetric stretches are always in the constant region of 3000–2800 cm^{-1} and has been assigned to 2870 cm^{-1} and 2960 cm^{-1} respectively. The CH_3 symmetric deformation occurs at 1370 cm^{-1} and asymmetric deformation at 1450 cm^{-1}. The methyl rocking vibration though not consistent due to its interaction with all other modes is assigned to the frequency at 1080 cm^{-1}. [8–10] The C—CH_3 stretching vibration coincides with the key infrared band of 7-*cis* isomer to give the strong absorption at 1250 cm^{-1}. The peripheral modes corresponding to the β-ionone rings give rise to bands around 1450, 1360 and 1250 cm^{-1} coinciding with the group vibrations of the methyl groups. Some of the bands above 1550 cm^{-1} in isomeric β-carotene may be due to β-ionone rings; the band at 1600 cm^{-1} is ascribable to this mode. Since the sample contains *cis-trans* isomers of β-carotene many of the bands due to the isomers independently have now collectively been obscured from the spectrum resulting in rather a simplified spectrum.

Ramana and Singh in their studies[11] have shown that β-carotene preserved better in low temperature dark storage than in shelf storage. They have shown that the amount of β-carotene preserved was found to decrease with storage time. The FTIR spectrum of β carotene recorded after a week of cold dark storage shows that the intensities of the various absorptions are reduced and exhibit slight frequency shift. There appears only one of the two bands due to coupled C═C—C stretching vibration at 1725 cm^{-1}. The C—H in-plane bending is a series of small absorptions at around 1150 cm^{-1} and of weak intensity compared to its occurrence as a strong intensity band in the previous spectrum. There appears a very sharp absorption at 810 cm^{-1} due to C—H out-of-plane waggings. The methyl rocking vibration has several strong absorptions at 1020 cm^{-1} and 1070 cm^{-1}. The characteristic band at 740 cm^{-1} due to 7-*cis* isomer of β-carotene is absent while the other band at 1240 cm^{-1} is seen in the spectrum. The characteristic band due to the 15-*cis* isomer is seen to occur at 785 cm^{-1}. The spectrum shows some structural degradation of the β-carotene molecule with prolonged cold dark storage.

Conclusion

The UV spectrum establishes the extraction of β-carotene from fresh carrots. In spite of the vibrations being complex for the molecule the various absorptions in the spectrum have been assigned to the different modes of vibration. The FTIR spectrum recorded after a week of cold dark storage show differences confirming structural degradation of the β-carotene molecule. This may be biologically important in view of fresh β-carotene being a major source in the synthesis of vitamin A, the deficiency of which leads to affectation of vision.

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