OLEORESIN RECOVERY, QUALITY CHARACTERIZATION AND STORAGE STABILITY IN CHILLI (*Capsicum* spp.) GENOTYPES

By

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THESIS

Submitted in partial fulfilment of the requirement
for the degree of

Doctor of Philosophy in Horticulture

Faculty of Agriculture
Kerala Agricultural University

DEPARTMENT OF OLERICULTURE
COLLEGE OF HORTICULTURE
Vellanikkara, Thrissur

1997

DECLARATION

I hereby declare that this thesis entitled **"Oleoresin recovery, quality characterization and storage stability in chilli (*Capsicum* spp.) genotypes"** is a bonafide record of research work done by me during the course of research and that this thesis has not previously formed the basis for the award to me of any degree, diploma, associateship, fellowship or other similar title of any other Univesity or Society.

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ACKNOWLEDGEMENT

I wish to place on record my gratitude and indebtedness to:

DR. M. ABDUL VAHAB, Associate Professor, College of Agriculture, Vellayani and Chairman of my advisory committee for imparting his unceasing guidance, concrete suggestions, constant encouragement and total involvement throughout the course of this study and preparation of this manuscript.

DR. S. RAJAN, Head, Department of Olericulture, DR. P. INDIRA, Assistant Professor, Department of Olericulture, DR. A. AUGUSTINE, Assistant Professor (Biochemistry) and SRI. S. KRISHNAN, esteemed members of my advisory committee for their valuable suggestions and assistance.

SRI. V.K. RAJU for ungrudging, timely, sensible and valuable suggestions.

DR. KESAVACHANDRAN for the help rendered in photographic works

All staff members of my department for their constant support, especially to ANU, PADMA and VIJAYANARAYANAN.

All friends for the co-operation and help received during the course of research.

MR. SURENDRA GOPAL for friendly co-operation and assistance rendered during the biochemical studies.

Parents, Husband and other family members, without whose support, encouragement and blessings, I could never have reached this stage.

Son, ACHU, who was deprived of motherly care during the period of my study.

DR. SIVADAS, Scientist, Spices Board, Kochi; DR. SHANKARIKUTTY, Scientist, Regional Research Laboratory, Thiruvananthapuram, DR. BALAGANGADHARAN, Scientist, ISRO, Thiruvananthapuram, DR. MOLLYKUTTY GEORGE, Central Computer Facility, Kerala Agricultural University for their generous help and valuable suggestions at various times during the research work.

MINI C.

to my parents

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Introduction

INTRODUCTION

Since time immemorial, spices have been considered an indispensable ingredient in culinary art. They are esteemed throughout the world for their ability to impart aroma and flavour to foods and beverages rendering them more palatable and interesting. Spices also form an essential ingredient in the manufacture of high quality cosmetics, premium perfumes and medicines. The quality of spices produced and exported from India has been and continues to be one of the best which earned her the name 'the Home of Spices' (Pruthi, 1993).

Of the five major spices, chilli ranks third, next only to black pepper and cardamom. In India, it is an indispensable spice cum vegetable in every household. Traditionally, the whole or ground chilli was used in the preparation of food products. But the use of this is now limited in food and beverage industries, as they do not lend their full characteristic flavours. Spice extractives, such as oleoresins serve as alternatives to this and provide stability required in product formulation. Chilli oleoresin, which is prepared from dried chilli powder by solvent extraction, represents the complete flavour or true essence.

Oleoresins offer consistency of quality, freedom from microorganisms, uniform dispersion in the product and easy handling and storage. Such attributes give oleoresins tremendous advantage over natural ground spices. The rapid growth in the processing industry in recent years has resulted in all time high demand for chilli oleoresin in both the traditional and modern food products.

Export of spice oils and oleoresins is rising steadily, not only in terms of quantity, but also in foreign exchange earned. The share of these value added products in the spices group has gone up from 8.3 per cent in 1989-'90 to 15.5 per cent in 1991-92 (Anon, 1992). But in the case of chilli oleoresin, India's share is as low as 6 per cent.

The oleoresin from high pungent chilli varieties is used as a counter irritant in lumbago, neuralgia, rheumatic disorders and internally for tonic and carminative action. They are extensively used in products like pain balms and prickly heat powders. Chilli varieties with bright red colour and moderate pungency are used for flavouring food products like hot biscuits, ginger soft drinks and for chewing tobacco.

The initial colour of chilli oleoresin, pungency and their retention vary with extraction procedures. Standardisation of the extraction procedures with respect to solvents and number of washings would help recover maximum oleoresin content from the produce more effectively.

There exists considerable variability in chilli cultivars in respect of yield and quality of oleoresin. By generating detailed information about the oleoresin yield of various chilli species and cultivars, it would be possible to identify such superior varieties and species with high oleoresin yield per unit area. This will fetch more income to the farmers which in turn may help the oleoresin industry also to demand a specific raw material.

Oleoresin recovery and its quality are influenced by the season also. Finding out the best season of cultivation for maximum yield of oleoresin would be helpful for farmers to grow chilli profitably. This may also help in deciding the apt crop rotation in a farming system to maximise net income of the farmers.

The harvesting stage may vary with the purpose for which chilli fruits are used. The content of oleoresin may also vary with the harvesting stage. Fixing the correct maturity stage for maximum oleoresin recovery would be helpful in increasing the yield and quality of oleoresin at lower cost.

The quality attributes of oleoresin viz. pungency and colour may change or deteriorate with the type of containers as well as the period of storage. Studies on container standardisation, quality characterisation and period of storage would suggest the best technique for maximum storage with minimum quality deterioration. This would also help the exporters to select the appropriate containers.

With increase in domestic demand coupled with sizeable international demand, the potential that exists in the country for chilli oleoresin production is required to be exploited.

Considering all these aspects, the present study was taken up with the following objectives:

1. Standardisation of extraction procedures for chilli oleoresin
2. Evaluation of *capsicum* species and cultivars for oleoresin recovery with respect to season and harvest maturity.
3. Studying the influence of harvest maturity on quality of chilli
4. Understanding the quality parameters of oleoresin as affceted by storage.

Review of Literature

REVIEW OF LITERATURE

Of the spice concentrates, capsicum oleoresins distinguished by handsome colour and / or pungency are ranked as the most outstanding. It is the product obtained by solvent extraction of the dried chilli fruits and the subsequent removal of the solvent. It has found increasing industrial use in place of ground chillies and is used in pharmaceuticals, food industry and beverages. Spice oleoresins offer consistency of quality, freedom from microorganism, uniform dispersion in the product and easy handling and storage. The yield and quality of oleoresins depend to a great extent on the crop species, varieties, method of extraction, maturity stages and storage conditions.

The available literature on these aspects relevant to the present study is reviewed under the following heads.

1. Extraction procedures of chilli oleoresin
2. Fruit quality and oleoresin recovery in *Capsicum* species and cultivars
3. Harvest maturity and seasonal influence on quality of chilli
4. Quality changes and storage stability of chilli oleoresin

2.1 Extraction procedures of chilli oleoresin

A closer approximation to the total spice flavour is given by its oleoresin. Selection of proper varieties, conditioning or drying of spice, choice of solvent and condition of extraction are the factors affecting yield and quality of oleoresin.

2.1.1 Solvents and the procedures

The type of solvent and the duration of extraction are important in oleoresin recovery as well as its quality.

Berry (1935) reported that oleoresin of capsicum varied in appearance, solubility and degree of pungency according to the solvents used. Berry and Samways (1937) found that longer extraction was essential for obtaining maximum pungency in the extract. Todd (1959) preferred acetone to other solvents, because of its high purity and nominal price. He described a better method of extraction, where fresh peppers are used instead of dried fruits in the conventional process.

Various methods of extraction of oleoresin from Capsicum have been compared by Ferns (1961). Acetone was considered the best solvent and a laboratory scale counter current apparatus was found to be the best device. By

employing percolation according to the method of the British Pharmaceutical Codex using various solvents, Tandon *et al.*. (1964) found ether as the best solvent for the extraction of capsicum, followed by hexane, chloroform, ethanol and acetone.

Nambudiri *et al.* (1970) adopted batch counter current extraction for chillies. Szabo (1970) asserted that acetone was an excellent solvent for capsaicin and was the most frequently used water miscible solvent.

Mathew *et al.* (1971) compared the efficiency of three different solvents in the extraction of chilli oleoresin. They found that hexane was a poor solvent for capsaicin and alcohol was not efficient to extract the colour. Ethylene dichloride was a good solvent for extraction of chilli oleoresin. Yield of oleoresin was more when fine powders were used. They found that chilli powders of 0.5 and 0.35 mm mesh gave yields of 12% and 16% oleoresin respectively.

Lewis *et al.* (1972) reported that the most commonly employed spice extracting solvent was ethylene dichloride which is immiscible with water and safe against fire hazards. Krishnamurthy and Natarajan (1973) reported acetone as the best solvent for the extraction of pigments by the percolation method. According to Naves (1974), the yield of oleoresin ranged from 4 to 8% with acetone and 4.5 to 11.6% with ethylene dichloride. Salzer (1975) reported that the composition and yield of oleoresins obtained by the extraction of dried

fruits were dependent upon the solvent and the raw material used. The methyl oxide formed in the acetone extracted spices, reacted with sulphur constituents (H_2S) in foods, particularly meat products, resulting in off-odour (Goldenberg and Matheson, 1976).

Shankarikutty *et al.* (1978) investigated the efficiency of six solvents including chloroform, methylene chloride, acetone and ethanol for extracting capsicum. Recovery was 0.12 to 0.22% during a two hour period as compared to a maximum yield of 0.25% when extracted for three hours. Pruthi (1980) suggested that the solvents should be chemically pure and free from any high boiling residues which impart unpleasant odours to the oleoresin. The solvent should have a low boiling range to facilitate complete removal after extraction, but too low boiling point will result in excessive loss of solvent.

Laboratory studies by Rajaraman *et al.* (1981) on oleoresin extraction efficiency of ethyl acetate from pepper, chilli, ginger and turmeric found the solvent as comparable to ethylene dichloride and acetone in yield of extractives and quality. Sigsworth (1987) reported that carbon dioxide extracted oleoresin was superior because of selective extraction of the aromatic constituents rejecting other constituents like waxes, sugars, pigments, proteins etc. Besides, there was no solvent residue problem. Amitkrishna De (1992) found alcohol, acetone and ethyl acetate as the major solvents for extraction of capsaicinoids. The most important essential qualities of capsicum are

pungency, colour and fixed (fatty) oil, which are involved in the manufacture of capsicum extractives (Verghese *et al.* 1992). The crucial factors in extraction of oleoresin are the physical state of subdivision of dried fruits and the nature of solvents used, provided that the fruits have been harvested at an optimum maturity and well dried (Pruthi, 1993). Yao *et al.* (1994) extracted the capsaicin and dihydrocapsaicin using supercritical CO_2 and organic solvents from *C. annuum* and quantified by HPLC.

2.1.2 Detection of solvent residue

The residual solvents in spice oleoresins should be below the permitted level. Methods to evaluate the residual solvents are very important. Todd (1960) reported a technique for evaluating residual solvents in spice oleoresins at levels below 50 ppm with an accuracy of ± 12 ppm. Each solvent (eg. methyl chloride, trichloroethylene, hexane, methanol, isopropanol and acetone) was studied individually and in combination with other solvents. Through gas-chromatography, chlorinated hydrocarbons such as methylene chloride, ethylene dichloride and trichloroethylene were best separated and analysed by Roberts (1968). The samples were extracted with ethanol and the extract was chromatographed on a 150/200 propak a column operated at 160° C and using MCD. Orsi (1970) reported gas chromatographic detection of the acetone content of paprika oleoresin.

Shankaranarayana *et al.* (1991) expressed doubts regarding the safety of the residual organic solvents in the spice extractives. They were of opinion that ethyl alcohol and ethyl acetate are the permitted solvents without any ill effects. The spice extractives like oleoresin should undergo strict quality control during various stages of production. Balakrishnan (1992) found the residual solvent in the product as the most important quality determinant for oleoresin.

2.2 Fruit quality and oleoresin recovery in Capsicum species and Cultivars

High quality oleoresin begins with the selection of proper cultivars. There exists considerable variability in chilli cultivars in respect of yield and quality of oleoresin.

2.2.1 Oleoresin recovery

Mathew *et al.* (1971) analysed the oleoresin yield (%) of some major chillies in the world, which included three varieties from India, four from Africa, two from Japan and one from Bahamas. The yield varied from 8.7 to 16.5%. Lewis (1972) observed distinct differences in quality and yield of oleoresin in varieties of chillies. Mathew and Shankaracharya (1974) observed that Indian chilli could be extracted to produce a product equivalent to oleoresin red pepper, which had limited demand in food industries.

Raina and Teotia (1986) evaluated chillies of Jammu and Kashmir and observed variability in oleoresin recovery. Considerable variation in oleoresin content (9.6 to 18%) using ethylene dichloride as solvent was observed in chillies grown in Himachal Pradesh also (Teotia and Raina, 1986). Tewari (1988) reported that 'Pusa Sadabahar' was a superior quality chilli with 12% capsaicin content in oleoresin compared to 8% of capsaicin in oleoresin of 'Pusa Jwala'.

Pradeepkumar (1990) analysed the oleoresin content of different *Capsicum* species and their interspecific hybrids. The oleoresin content ranged from 18.7% in *C. annuum* to 31.7% in *C. chinense*. *C. frutescens* had oleoresin content of 27.3%. The F_1 hybrids with high oleoresin content were *C. frutescens* x *C. chinense* (35.37%) and *C. annuum* x *C. chinense* (34.4%).

Plant breeding and selection of pepper cultivars (*C. annuum* L.) for paprika and oleoresin were carried out by Navarro and Costa (1991). Along the selection process, several varieties of dark red colour with chlorophyll retainer genes and conventional red colour have been obtained.

Lakshmanachar (1993) reported that varieties with low seed content and free of stalk and calyx were suited for chilli oleoresin production. Pruthi (1993) presented chemical quality attributes of fifteen chilli varieties grown in different regions of India. Oleoresin content of these varieties varied from 6.2 to 12.4% on moisture free basis. Indira *et al* (1994) evaluated twenty five chilli

accessions belonging to *C. annuum, C. chinense* and *C. baccatum* and reported the oleoresin range from 14 to 28%.

2.2.2 Fruit quality

Chilli species and genotypes vary in their fruit quality attributes like capsaicin, capsanthin etc. Berry and Samways (1937) reported that the pungency factor varied among fruits of the cultivars of the same species. Heiser and Smith (1953) reported that the small, thin skinned chilli peppers had the highest capsaicin content. Capsaicin content of several New Pusa varieties of chillies were determined by Deb *et al.* (1963) and found that some of the varieties released for cultivation had a high capsaicin content (1.05 to 1.59%). Balbaa *et al.* (1968) found that the capsaicin content of mature fruit was 50% higher in *C. minimum* than in *C. frutescens*. Davies *et al.* (1970) found a wide variation among cultivars in the total carotenoid contents.

Vinkler (1971) determined the total carotenoids, capsanthin and capsorubin in fruits of *C. annuum* var. *annuum*. He found that capsanthin formed 45 to 80% of the total carotenoids and capsorubin ranged from 6 to 19%. Nagle *et al.* (1979) analysed fifteen chilli cultivars for total pigments, xanthophyll and B-carotene. Significant difference were found between cultivars for objective and subjective colour values. According to Purseglove *et al.* (1981), the species, cultivars or strain grown had a dominant influence

on quality determinant properties of pungency, colour and colour retention. Shankarikutty *et al.* (1982) reported that the species *C. frutescens* had the highest capsaicin content, followed by medium sized chillies belonging to species *C. annuum.*

Govindarajan (1985) reported that the greatest effect on final colour retained and the pungency was the cultivar grown, its intrinsic composition of the colour complex and concentration of the components that contribute the red component. A line designated as 'LCA 206' was selected by Murthy *et al.* (1987) from the *C. annuum* cross G_3 x Huntaka. It showed the bright red colour and good colour retention in storage of Huntaka, combined with the fruit size and yield potential of G_3.

Ferrari *et al.* (1988) tabulated the data on the capsaicin content of 21 cvs. and F_1 hybrids. They suggested difference spectrophotometry and Gibb colorimtery as the recommended methods for capsaicin determination. Narayanan (1988) reported the highest capsaicin content in *C. frutescens.* He found negative association of fruit size with pungency.

The total capsaicinoid content of fruits of *C. frutescens* and *C. annuum* cultivars were determined by Ogbadu *et al.* (1989) spectrophotometrically. *C. frutescens* had 33.7 to 266.5 mg/100g and *C. annuum* cvs. had 0 to 107.2 mg of capsaicinoid per 100g of fruit. Yazawa *et al.* (1989) studied the capsaicinoid

content of hybrids between sweet or pungent cultivars of *C. annuum* and *C. chinense* No.3341.

Pradeepkumar (1990) reported that hybrid *C. annuum* x *C. chinense* was the most promising interspecific hybrid with high values of capsaicin, colour and oleoresin. Improving specific market types for pigment quality should be feasible through plant breeding, since the proportion of colour pigments varied considerably among pepper varieties (Almela *et al.* 1991). Bosland *et al.* (1991) developed a non pungent, high yielding cultivar with high extractable red pigment, known as 'Nu Mex Conquistador'. Capsaicin content in 1018 *C. annuum* varieties were analysed by Pu *et al* (1992). There were 61 varieties with less than 0.01% capsaicin. Pruthi (1993) reported 'Aparna' and 'Pachas Yellow' as the two yellow chilli varieties suitable for capsaicin extraction.

Indira (1994) evaluated twenty paprika types and found them suitable for cultivation during August-January under Kerala condition. Biosynthesis and transformation of carotenoid pigments during ripening of fruits from two Spanish *Capsicum* varieties, 'Bola' and 'Agridulce' were studied by Minguez - Mosquera and Mendez (1994).

2.3 Harvest maturity and seasonal influence on quality of chilli

Pungency and colour are the most important quality parameters of capsicum. The pungent principle in chilli is an integral of atleast 12 analogous vanillylamides, of which capsaicin and dihydrocapsaicin constitute almost 90% of pungency. Colour of chilli is due to the presence of carotenoid pigments. So far 37 pigments have been isolated from chillies, of which capsanthin and capsorubin are important. Geographic location, climate, harvest maturity and processing procedures are the factors influencing the product quality.

2.3.1 Harvest maturity

Cholnoky (1939) on analysing several varieties of paprika for two yearly harvests found that the total colour was higher for the first harvest than for the second. Lease and Lease (1956) reported that the degree of harvest maturity of fruits affected the colour stability of dried ground capsicum. The initial colour of chilli fruits picked at withering stage was superior to that picked at full ripe but succulent.

According to Benedek (1972) the chilli fruits harvested in red and ripe stage continued to live and its colouring matter content increased. He found that mature but still unripe chilli fruits when harvested became red later on, however, it contained less colouring matter than fruits harvested red ripe. Quantitative analysis of the cartenoid pigments of five capsicum cultivars at

four stages of maturation were done by Rahman and Buckle (1980). From immature, mature, half ripened and fully ripened capsicum fruits, upto twelve, twelve, twenty nine and twenty six individual pigments respectively were isolated and identified.

Aczel (1986) determined the carotenoids in unripe, half ripe and ripe fruits. He found that in ripe fruits, the red components comprised > 60% of the total colouring matter, and of this capsanthin formed 53.5%. Nisar Ahmed *et al.* (1987) tabulated the capsaicin content of green, ripe and sun dried fruit of 12 *C. annuum* varieties. They reported that capsaicin content increased in the order green fruit < ripe fruit < sundried fruit.

Changes in the carotenoid pigments of paprika fruits at six stages of ripening were investigated by Deli *et al.* (1992). Thirty four carotenoids were identified from the chromatograms. Dynamic head space, Gas chromatography, Mass spectrometry and sniffing port detection were used by Luning *et al.* (1994) to analyse volatile compounds in *Capsicum* fruits at three ripening stages, such as green, turning and red. Saga and Ogawa (1995) found that carotenoid content increased rapidly between seven and ten weeks after flowering.

2.3.2 Seasonal Influence

Claus (1961) reported that the percentage of capsaicin in the fruits varied greatly depending on species, geographical origin of the sample and climatic condition. Ohta (1962) reported that higher night temperature was responsible for the higher capsaicin content. Laul *et al.* (1970) reported that the total colouring matter of dried chillies showed an increasing trend with the advancement of the season and number of picking. A better understanding of agronomic and environmental factors which affect certain biochemical processes such as capsaicinoid and carotenoid synthesis was warranted (Margoczi *et al.* 1989). Bosland (1993) and Menon (1995) found that harvesting conditions, post harvest operations etc. determined the initial colour in chillies.

However, Van Blaricum and Martin (1951) could not correlate the location in which the chilli was grown or the kind of fertilizers used with difference in colour stability.

2.4 Quality changes and storage stability of chilli oleoresin

Scientific storage is an important stage in the series of operations required for successful and orderly marketing of any product. This is applicable to chillies which are quite sensitive to moisture, storage temperature,

light and air or oxygen. Compared to whole and ground chillies, oleoresins have a long shelf life under ideal conditions. Majority of the literature available on storage studies are confined to whole or ground chillies.

2.4.1 Storage of whole and ground chillies

Van Blaricum and Martin (1951) have tried to correlate the fat content of chillies with colour retention and found that lack of natural anti-oxidants contributed to colour loss on storage. Lease and Lease (1956) found that storage at higher temperature increased the rate of colour destruction. They also observed that the colour retention in peppers was affected by the stage of ripeness at harvest.

Chen and Gujmanis (1968) observed that the chilli powder samples stored with moisture content of 9 to 10% retained better colour.

Natarajan *et al* (1969) reported that treatment given to whole chillies with butylated hydroxy anisole, prophyl gallate or ascorbic acid did not show significant effect on the preservation of colour during storage.

Pruthi (1969) observed bleaching or degradation of colour (Capsanthin and Capsorubin) in Hungarian paprika powder during storage. When the

refrigerated samples were brought to higher temperature, the samples suffered from accelerated colour loss (Krishnamurthy and Natarajan, 1973).

Stringheta *et al.* (1979) reported that each paprika cultivar had varying inherent property to retain colour during storage.

Sumathikutty and Mathew (1983) reported that deterioration of colour in chilli was due to oxidation of carotenoid pigments.

Raina *et al.* (1986) found that the presence of less stable carotenoids and highly autoxidisable fatty acids in paprika might enhance the liability of capsanthin to oxidation.

KoshyJohn (1989) reported that the colour of paprika was reduced considerably when the spice was stored for several months particularly at temperature above 20° C.

Bicas *et al.* (1994) studied the storage stability of carotenoids from new hybrids produced from Hungarian and Spanish paprika cultivars.

2.4.2 Container studies

The primary purpose of a container is to preserve the flavour and keep the product in good condition until it reaches the consumer. The various

materials suitable for storage of spice include paper products, polyethylene flexible films, aluminium foils, glass, tin and timber. Van Blaricum and Martin (1945) and Chen and Gujmanis (1968) found that the type of container in which the dried pepper was stored did not affect colour loss.

Lease and Lease (1956) found that dried ground paprikas stored in stoppered glass bottles at 37°C and 25°C were definitely better in colour retention over a 10 month period than the same peppers stored in kraft paper bags.

Natarajan *et al* (1969) studied the storage behaviour of whole chillies stored in sealed cans over a period of six months. They observed that the stored samples with 11 to 12.9% moisture gave higher colour values than did samples stored with moisture contents below 9%.

Stringheta *et al* (1979) compared the effects of different packaging materials and storage time on the colour of different varieties of paprika. They found that clear glass containers were superior to opaque polythene bags.

KoshyJohn (1988) reported that the storage of dried spice in air tight containers out of sunlight was desirable.

Lownds *et al.* (1994) found that the colour development was cultivar and package dependent and a significant cultivar x package interaction occured at 8 and 20° C.

2.4.3 Storage of oleoresin

Specific studies on the storability of high colour and capsaicinoid in chilli oleoresins are rather nil. Packaging and storage studies conducted by Narayanan *et al.* (1964) revealed the stability of black pepper oleoresin packed in 30 and 125 ml aluminium bottles at different temperature and humidity.

Kanner *et al.* (1978) found poor stability of carotenoids in stored chilli oleoresin as a serious economic problem.

Charazka *et al.* (1981) found that chilli extracts stimulating high pungency are well preserved for nine months at room temperature and for longer periods at 0° C.

Govindarajan *et al.* (1986) reported that with good storage methods, the quality of oleoresin was maintained for fairly long periods.

Balakrishnan (1992) recommended the epoxy (food grade) lined steel drums and high density polythene pails and carboys of internationally

acceptable quality for packing of oleoresin. Aluminium and stainless steel containers were also suggested.

Lakshmanachar (1993) found that spice extractives were stable over long periods of storage with less space.

Sapers (1994) observed extensive fading in oleoresin paprika sample, prepared for colour evaluation after several months of storage at 4°C.

Material and Methods

MATERIALS AND METHODS

The present investigations were carried out in the Department of Olericulture, College of Horticulture, Kerala Agricultural University, Vellanikkara during the period from 1993-1996. The experimental field is located at an altitude of 22.5M above M.S.L and is in between 10° 32″N and 76° 16″E longitude. The area experiences a warm humid tropical climate.

The study was undertaken on the following aspects.

1. Standardisation of extraction procedures for chilli oleoresin.
2. Evaluation of Capsicum species and cultivars for oleoresin.
3. Influence of harvest maturity on quality of chilli.
4. Quality changes and storage stability of chilli oleoresin.

3.1 Standardisation of extraction procedures of chilli oleoresin

3.1.1 Experimental materials

The chilli variety, Ujwala (plate. 1) released from the Kerala Agricultural University, was used for the study.

The variety belongs to *C. annuum* L. which was developed by single plant selection of an open pollinated material introduced from Japan. It is a

Plate 1. UJWALA

high yielding variety with clustured (9-10 fruits/cluster), pungent long fruits which are dark green at green stage and turning deep red on ripening. It is a medium duration variety resistant to bacterial wilt and free from viral leaf curl under field condition. Due to high pungency and colour, fruits of Ujwala have great demand in Oleoresin industry.

3.1.2 Experimental methods

Crop husbandry

Uniform and healthy seedlings of the above variety were transplanted in an area of 80 m^2. All the management practices were given as per Kerala Agricultural University Package of Practice Recommendations (KAU, 1993).

Oleoresin Extraction

Oleoresin was extracted by Soxhlet extraction methods (Langenau, 1959) using following six solvents.

S_1 - Acetone

S_2 - Ethyl alcohol

S_3 - Dichloroethane

S_4 - Hexane

S_5 - Benzene

S_6 - Ethyl acetate

General properties of the extraction solvents are given in Table 1.

Table 1 General properties of the solvents

Solvents	Chemical formula	BP (°C)	Solubility in water (g/100 ml at 25°C)	Flash point (°C)	MP	FP	Cost/ litre (Rs.)	Remarks
Acetone	CH_3 CO CH_3	56.1 - 56.5	∞	-18	-94°C	1°F (-17°C)	132	Flammable liquid
Ethyl alcohol	C_2H_5 OH	78.4	∞	13	-130°C	48°F (8°C)	610	Edible solvent
Dichloroethane	CH_3 CH Cl_2	83	0.9		-35°C	60°F (15°C)	346	Cancer suspect agent
Hexane	$CH_3(CH_2)_4CH_3$	66-71	insoluble	-26	-95°C	-10°F (-23°C)	1320	Flammable liquid
Benzene	C_6H_6	79.1	0.7	-11	6.8°	12°F (-11°C)	240	Cancer suspect agent, Flammable liquid
Ethyl acetate	$CH_3CO_2C_2H_5$	77-77.2	7.4	-4	-84°C	26°F (-3°C)	214	Flammable liquid

BP - Boiling Point FP - Freezing Point MP - Melting Point

Red ripe fruits were harvested, destalked, dried in hot air oven at 50° C for 2 hours to 6% moisture level and powdered to 100 mesh sieve.

Five gram chilli powder was weighed, packed in filter paper and placed in soxhlet apparatus. 200 ml solvent was taken in a round bottom flask of the apparatus and heated in water bath. The temperature was maintained at boiling point of the solvents. Extraction with each solvent was repeated five times, thus each extraction serving as replications. After complete extraction, the solvent was evaporated to dryness by vacuum.

3.1.3 Observations

The following observations were recorded from the experiment.

3.1.3.1 Oleoresin yield (%)

Yield of oleoresin from 5 g chilli powder was found and converted to percentage.

3.1.3.2 Siphoning number

Number of Siphoning required for complete extraction of 5 g sample was recorded.

3.1.3.3 Siphoning time (mts)

Time required for each siphoning was noted in minutes.

3.1.3.4 Extraction efficiency of solvents for colour value

Colour extraction efficiency of solvents was determined as per EOA (1975). One gram oleoresin was weighed accurately and dissolved in 100 ml of acetone. Of this stock solution, 1 ml was diluted to 100 ml in a volumetric flask with acetone. Using tongsten lamp source and acetone as a blank, the absorbance of this 0.01% solution was taken at 458 nm using a spectronic 20 spectrophotometer. The absorbance value was multiplied by 61000 (an empirical factor worked out to relate to data from the colour matching method) to obtain the Nesslerimetric colour value.

3.1.3.5 Extraction efficiency of solvents for β-carotene

The total colour value was expressed in terms of β carotene using the equation:

$$\beta \text{ carotene } (mg/g) = \frac{E \times 100}{196 \times L \times W} \text{ where,}$$

E = Optical density

L = Path length in centimeter (usually 1 cm)

W= Weight in grams of the sample per milli litre of the final extract.

Factor 196 was for average mixture of carotene in food sample.

3.1.3.6 Extraction efficiency of solvents for Capsanthin - Spectrophotometric method

The red colour of chillies is mainly due to carotenoid pigments, which are nearly 37 in number. Capsanthin is the major pigment, constituting about 35% of the total pigments (Curl, 1962).

Extraction efficiency of solvents for capsanthin was determined by Hungarian standard method (Benedek, 1958).

Sample extraction

0.05g oleoresin was weighed and transferred to a glass stoppered flask with 1 gm anhydrous sodium sulphate. The pigments were extracted by shaking with 50 ml benzene for 30 minutes in the dark. The extract collected were made upto 100 ml in a volumetric flask.

Calibration of Instrument

A standard solution was made with 1.35 g potassium dichromate, dissolved in 20 ml sulphuric acid (5%). The absorbance of this solution measured in 1 cm. cuvette against 5% sulphuric acid at 477 nm should be 0.315. The instrument calibration factor, 'f' was calculated by

$f = 0.315/e$ where,

e = absorbance measured for the above standard solution.

Measurement

The absorbance of the diluted oleoresin was measured at 477 nm against the solvent benzene. Pigment content was calculated by the formula,

$$\text{Pigment content, } F = \frac{e x f}{1826 \times b \times c} \times 10^5 \quad \text{where,}$$

F = total pigment in gram/kilogram dry matter as capsanthin

e = absorbance at 477 nm

f = calibration factor of instrument

b = weight of sample taken

c = dry matter content of sample

1826= extinction coefficient of 1% (g/v) benzene solution of capsanthin at 477 nm.

10^5 = conversion factor to calculate pigment per kilogram dry matter.

3.1.3.7 Extraction efficiency of solvents for capsanthin (Thin layer chromatographic method)

The capsanthin content of oleoresin was separated by Thin layer chromatogrpaphy (Ikan, 1969). The special advantages of TLC compared to other methods include versatility, speed and sensitivity.

0.1 g oleoresin was weighed and 6 ml ether was added to it. To this diluted mixture, 1 ml, 30% methanolic potassium hydroxide was added and stirred for 8 hours using a magnetic stirrer. The ether layer separated was collected, washed by shaking with 0.5 ml water several times in a separating

funnel and concentrated to 0.2 ml by vacuum system. This was diluted with 0.6 ml petroleum ether and kept in a refrigerator for 24 hours, for separating the crystals of capsanthin. The ether extract was spotted on TLC plate using petroleum ether and acetone in 9:1 ratio (Preparation of TLC plate is given in Appendix - 1).

3.1.3.8 Extraction efficiency of solvents for pungency (Thin layer chromatographic method)

The pungency of chilli is due to capsaicin and traces of allied compounds (Sumathykutty and Mathew, 1983).

The capsaicin and allied compounds can be separated from other constituents by TLC on silica gel. The pungent principle can react with Folin - Dennis reagent to give a blue complex, which can be estimated colourimetrically (Mathew *et al* 1971). (Preparation of Folin - Dennis reagent is given in Appendix - 2).

0.05 g oleoresin was weighed and mixed with 0.1 ml acetone. 5 µl of this solution was spotted on TLC plate and developed with developing solvent (80 ml benzene : 5 ml methanol). The plate was exposed for about 30 minutes to free it of the solvent and lightly sprayed with Folin - Dennis reagent. The clear blue capsaicin spot was marked with a stainless steel spatula in the form of a circle enclosing the spot as well as an area of 0.25 cm beyond the spot.

The silica gel containing capsaicin in the marked area was scooped and transferred to a test tube. 3.5 ml of distilled water and 0.5 ml Folin - Dennis reagent were added into the test tube and mixed well. After 3 minutes, one ml of 25% aqous sodium carbonate solution was added, mixed thoroughly and set aside for one hour.

The blank was prepared using silica gel layers from the blank area in the plate and the optical density was read in spectrophotometer at 725 nm. The amount of capsaicin in the spot was determined from the curve prepared using standard capsaicin. (Preparation of standard capsaicin curve is given in Appendix - 3).

3.1.3.9 Extraction efficiency of solvents for pungency - Scoville Heat Units

For the assay of pungency, the dilution method of Scoville prescribed by American Spice Trade Association (ASTA, 1981) was followed. Fundamentally, this is based on the response of human tasters to a sample diluted to its threshold of detection. The reciprocal of the dilution is called the Scoville Heat Units (IS: 8104-1976).

Heat Units of oleoresins prepared using different solvents were calculated based on the equation,

Scoville Units = ppm capsaicin x 15

3.1.3.10 Solvent residue

The most important quality determinant for oleoresin is the residual solvent in the product (Balakrishnan, 1992). Gas chromatographic analysis was followed for the determination of residual solvent.

Sample extraction

The following chemicals were added to oleoresin samples along with 100 ml distilled water in a 250 ml flask.

* Toluene solution containing 2500 ppm benzene (1 ml)
* Sodium sulphate (10 g)
* Ammonium sulphate (10g)

The flask was connected to a volatile oil apparatus for oils heavier than water and collected 15 ml of distillate. 15 g of potassium carbonate was added to the distillate, cooled while shaking and allowed the two phases to separate. The upper layer was used for injection.

Analysis

Column - Carbowax 20 M 10% 100°C
Flow rate - (He) 40 ml/minute
Detector - TCD
Injection temperature - 200°C

Calculation

Standard solvent was injected into Gas chromatograph and the peaks were noted. Based on the retention time of solvent, the residue in oleoresin sample was calculated from the graph.

3.2 Evaluation of Capsicum species and cultivars for oleoresin recovery

3.2.1 Influence of season

3.2.1.1 Experimental materials

The influence of season on oleoresin yield was studied in the following nine chilli genotypes.

V_1 - *Capsicum annuum* (Gundu) - CA 653

V_2 - *Capsicum annuum* (Pendulous) - Arka Lohit

V_3 - *Capsicum annuum* (Cluster) - Ujwala

V_4 - *Capsicum annuum* (Paprika) - KTPL - 19

V_5 - *Capsicum chinense* - CA 640

V_6 - *Capsicum chinense* - CA 645

V_7 - *Capsicum frutescens* - CA 671

V_8 - *Capsicum frutescens* - CA 648

V_9 - *Capsicum baccatum* - CA 670

3.2.1.2 Experimental methods

All these genotypes were evaluated in three seasons viz. S1 - Summer (January - March); S2 - Rainy (May - July) and S3 - Winter (September - November). The experiment was laid out in Randomized Block Design with 27 treatments and 3 replications. The plot size was 3.6 x 2.7 m^2 with 48 plants spaced at 45 cm x 45 cm. All the operations were carried out as per Package of Practice Recommendations of Kerala Agricultural University (KAU, 1993). From all plots, 25 plants were randomly marked as observation plants.

3.2.1.3 Observations recorded

Days to flower

Number of days from sowing to flowering of 50% of plants was noted and average recorded.

Days to fruitset

Number of days from sowing to fruitsetting of 50% of the plants was observed and average recorded.

Days to harvest

Number of days from sowing to first harvest in 50% of plants was observed and average worked out.

Fruits/plant

Fruits from all harvests were counted, average worked out and expressed as fruits/plant.

Fruit yield/plant (g)

Fruits from all harvests were weighed and the average expressed in grams.

Oleoresin yield (%)

Oleoresin was extracted by solvent extraction method using ethyl acetate, which was the solvent standardised and expressed as percentage.

3.2.2 Influence of harvest maturity

3.2.2.1 Experimental materials

Fruits were harvested at three different stages of maturity from the nine chilli genotypes, raised during the three seasons (Plate 2). The three maturity stages were:

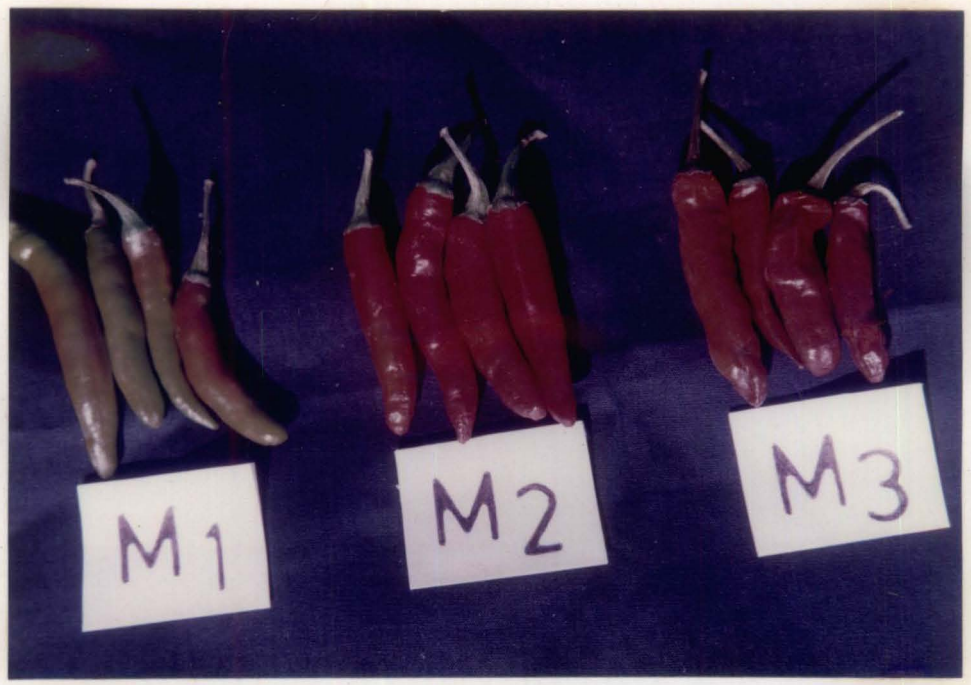


Plate 2 : stages of harvest maturity

M1 - Stage when mature fruit just starts changing its colour to intermediate stage.

M2 - Stage when the fruit becomes fully ripe, but firm and succulent in nature.

M3 - Stage when the fully ripe fruit has become shrivelled in appearance.

Colour changes of the fruits of different genotypes used in the study, to judge the above maturity stages are shown in Table 2.

The experiment formed two factorial RBD, one with 9 genotypes and 3 seasons and other with 9 genotypes and 3 maturity stages each with 3 replications.

3.2.2.2 Observations recorded

Fruits/Plant

Fruits from all harvests were counted, average worked out and expressed as fruits/plant.

Fruit yield/plant (g)

Fruits from all harvests were weighed, average worked out and expressed in grams.

Table 2 Colour changes of fruits in different genotypes

Genotypes		Mature	Intermediate	Ripe
C. annuum	CA 653	Green	Brown	Red
	Arka Lohit	Green	Brown	Red
	Ujwala	Green	Orange red	Red
	KTPL-19	Green	Orange red	Red
C. chinense	CA 640	Green	Orange	Reddish orange
	CA 645	Green	Orange	Reddish orange
C. frutescenes	CA 671	Green	Orange	Red
	CA 648	Green	Orange red	Red
C. baccatum	CA 670	Green	Orange	Red

Oleoresin yield (%)

Oleoresin was extracted by solvent extraction method using ethyl acetate and expressed as percentage.

3.3 Influence of harvest maturity on quality of chilli

The nine chilli genotypes grown during summer season were harvested at three maturity stages as in previous case to form 27 treatments comprising of 9 genotypes and 3 maturity stages of a Factorial CRD with 3 replications. Dried fruits were powdered and evaluated for the following characters.

3.3.1 Colour value - (E.O.A method) : as in previous case.

3.3.2 Pungency (Folin - Dennis method) : as in previous case.

3.3.3 Total sugar (Anthrone method)

Chilli powder (100 mg) was hydrolysed by keeping in a boiling water bath for 3 hours with 5 ml, 2.5 N hydrochloric acid and cooled to room temperature. This was neutralised with sodium carbonate and made up the volume to 100 ml. The supernatent was collected, 0.5 ml taken and made up the volume to 1 ml using distilled water. 4 ml anthrone reagent was added, heated for 8 minutes, cooled and the absorbance was read at 630 nm. From the standard graph prepared (Preparation of standard curve for sugar is given in Appendix - 4) the total sugar present in the sample was calculated and expressed as percentage.

3.4 Quality and storage stability of chilli oleoresin

The oleoresin extracted from the variety 'Ujwala' using the standardised solvent viz. ethyl acetate was subjected to storage study at laboratory level. The treatments included four types of containers viz. B1 - PVC jar with plastic cap; B2 - Polythene jar with plastic cap; B3 - PET jar with plastic cap; B4 - amber coloured glass bottles with plastic cap (Plate 3) and two conditions viz. open which was kept in open room in sunlight; and dark, where the bottles were covered with black paper.

The experiment formed a Factorial CRD with 8 treatments and 4 replications. Storage was done at room temperature for 12 months from August, 1995 to July, 1996.

3.4.1 Observations recorded

3.4.1.1 Colour value (E.O.A. method) : as in previous case.

3.4.1.2 Pungency (Folin - Dennis method) : as in previous study.

The observations were recorded at the time of storage, one month after storage and then at four month intervals.

3.5 Statistical analysis

Appropriate statistical analyses were carried out using standard packages MSTATC and SPAR1 available at the Computer Centre of the College of Horticulture, Vellanikkara.

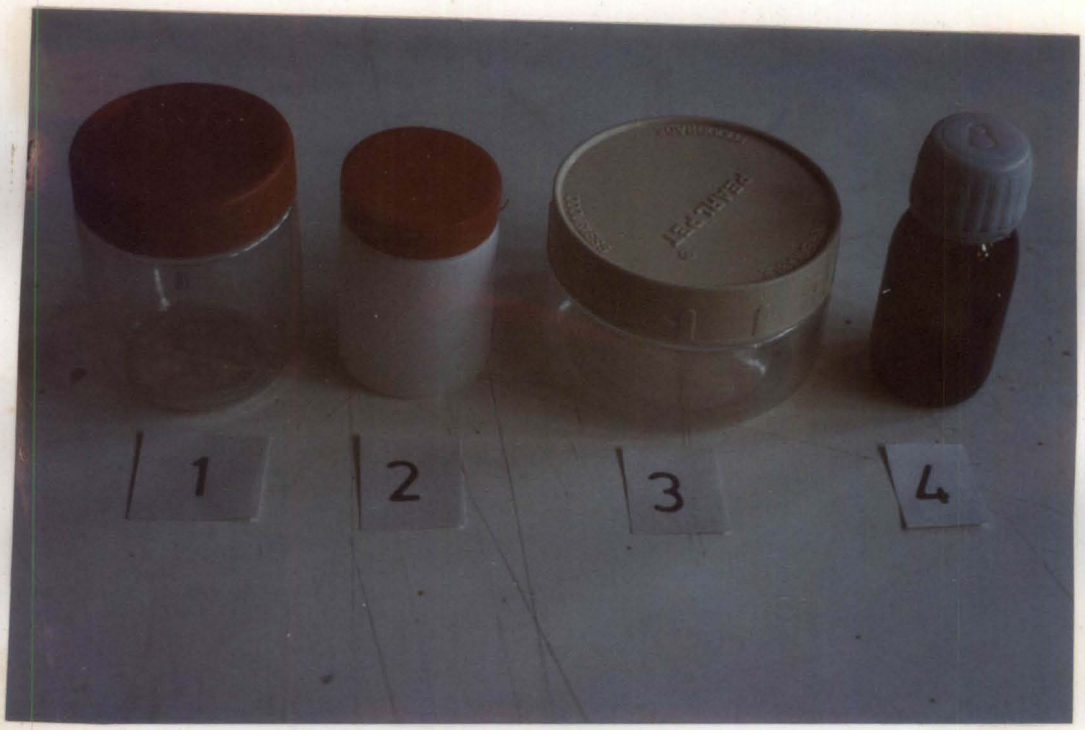


Plate 3 : Types of containers

Results

RESULTS

Data recorded in the present study were analysed and results are presented under the following heads.

1. Standardisation of extraction procedures for chilli oleoresin
2. Evaluation of *Capsicum* species and cultivars for oleoresin recovery.
3. Influence of harvest maturity on quality of chilli
4. Quality changes and storage stability of chilli oleoresin

4.1 Standardisation of extraction procedures of chilli oleoresin

General analysis of variance showed significant differences among the solvents for all the characters studied. The solvents differed significantly for oleoresin yield, siphoning number, siphoning time and extraction efficiency for colour and pungency (Table 3).

4.1.1 Oleoresin yield

The yield of oleoresin ranged from 10.6% to 37.2% with an average of 16.89%. Highest yield was extracted by ethyl alcohol (37.2%) and the lowest by dichloroethane (10.6%).

Table 3 Efficiency of solvents for oleoresin extraction

Solvents	Oleoresin yield (%)	Siphoning No.	Siphoning time (mts)	Capsanthin (g/kg)	Colour value	Capsaicin (mg/g)
Acetone	14.6	11.2	25.2	4.661	22404 (10.017)	68.4 (4.226)
Ethyl alcohol	37.2	22.2	40.2	1.468	1091 (6.995)	31.1 (3.437)
Dichloro ethane	10.6	12.4	21.2	1.247	4134 (8.327)	67.69 (4.215)
Hexane	11.4	12.4	27.0	6.836	12407 (9.426)	49.89 (3.910)
Benzene	14.6	11.0	18.4	2.424	10178 (9.228)	58.32 (4.066)
Ethyl acetate	12.9	9.2	24.4	8.184	16865 (9.733)	75.79 (4.328)
CD (P=0.05)	2.54	2.01	1.01	0.02	0.46	0.006

(Values in paranthesis are the transformed values)

4.1.2 Siphoning number

Siphoning number ranged from 9.2 to 22.2 with an average of 13.06. Ethyl acetate took the minimum number of siphoning (9.2) which was on par with acetone (11.2) and benzene (11.0). Ethyl alcohol took the maximum siphoning number (22.2) for extraction.

4.1.3 Siphoning time(min)

Siphoning time ranged from 18.4 minutes for benzene to 40.2 minutes for ethyl alcohol with an average of 25 minutes.

4.1.4 Extraction efficiency of solvents for colour value

Acetone had the maximum (22404 Nesslerimetric colour value) efficiency for colour value. Ethyl acetate (16865) was on par with acetone in extraction efficiency. The lowest colour value (1091) was measured for ethyl alcohol.

4.1.5 Extraction efficiency of solvents for β carotene (mg/g)

The total colour value was expressed in terms of β carotene also (Table 4). The β carotene content ranged from 91.3 to 1873.4 mg/g, the highest content was recorded by acetone and the lowest by ethyl alcohol.

4.1.6 Extraction efficiency of solvents for capsanthin (Spectrophotometric method)

Capsanthin content of oleoresin samples ranged from 1.247 to 8.184 g/kg (Table 3). Oleoresin extracted using ethyl acetate had maximum (8.184 g/kg) capsanthin content, followed by hexane (6.836). Acetone could extract only 4.661 g/kg of capsanthin. Minimum capsanthin content was in oleoresin extracted using dichloro ethane (1.247 g/kg).

4.1.7 Extraction efficiency of solvents for capsanthin (TLC method)

Thin layer chromatographic method of separation and determination of capsanthin from oleoresin was done and Rf values are presented (Table 5). Capsanthin, the major colour pigment appeared as a pink spot with an Rf value around 0.8 (Plate 4). Maximum number of spots (6) was seen in chilli oleoresin sample extracted using ethyl acetate.

4.1.8 Extraction efficiency of solvents for capsaicin (TLC method)

Thin layer chromatographic method was followed for separating capsaicin from oleoresin samples and capsaicin appeared as blue spot with an Rf value about 0.15 (Plate 5). Capsaicin content of oleoresin samples extracted using different solvents ranged from 31.1 to 75.79 mg/g (Table 3). Ethyl acetate showed maximum efficiency (75.79) for capsaicin extraction, followed by acetone (68.4 mg/g). Oleoresin prepared using ethyl alcohol had the minimum (31.1 mg/g) capsaicin content.

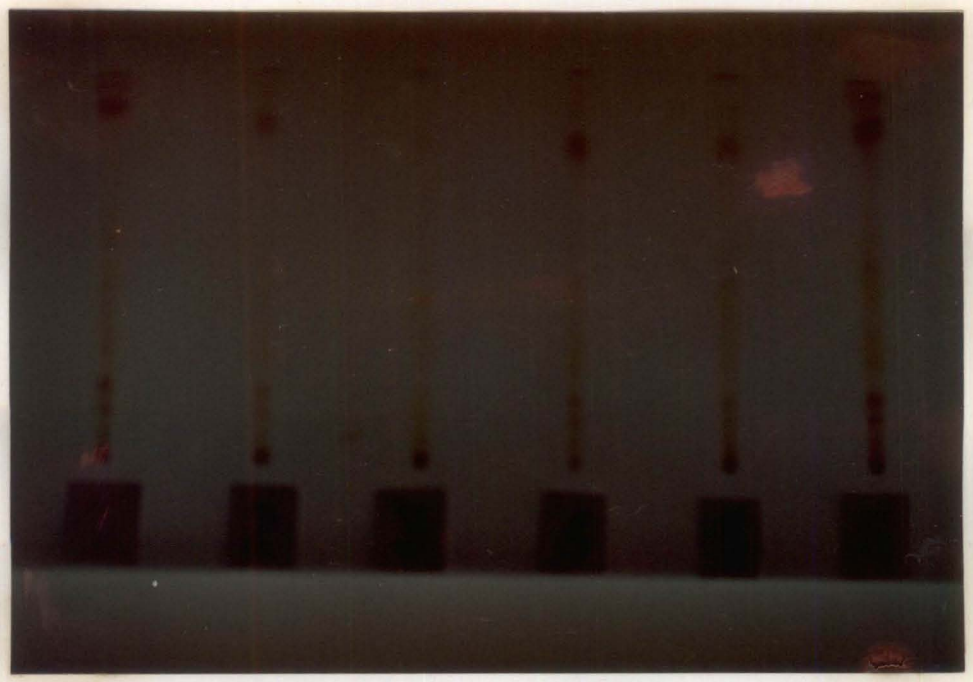


Plate 4 : Thin layer chromatographic separation of capsanthin

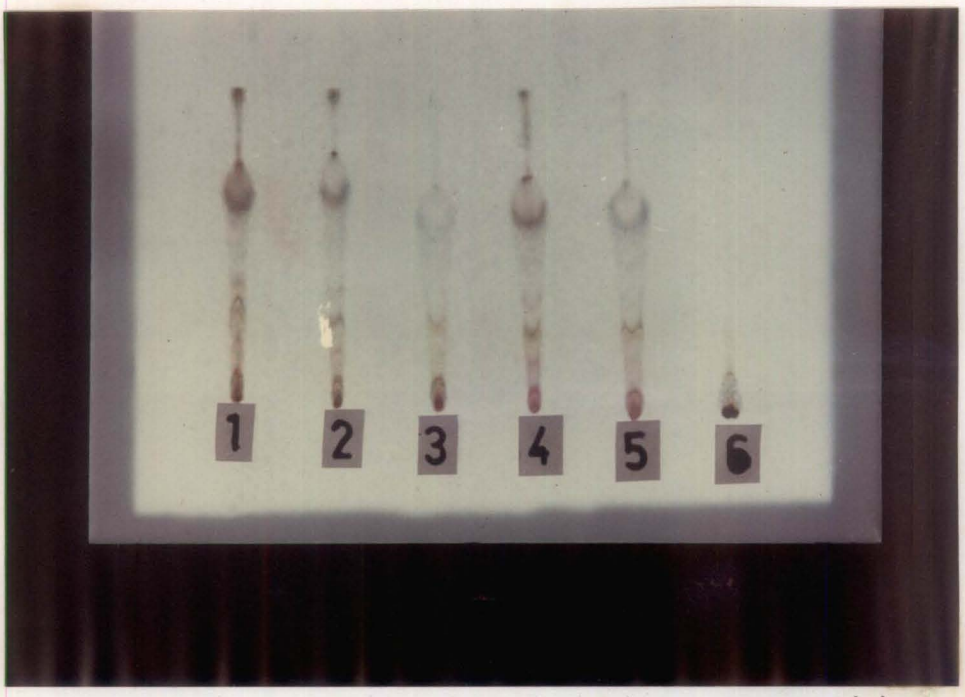


Plate 5 : Thin layer chromatographic separation of capsaicin

Table 4 Mean performance of solvents for extraction

Solvents	β carotene (mg/g)	Scoville units
Acetone	1873.4	1026630
Ethyl alcohol	91.3	466620
Dichloro ethane	345.9	1015260
Hexane	1644.4	748560
Benzene	851.0	874590
Ethyl acetate	1410.7	11365580

Table 5 Results of chromatographic studies

Solvents	Rf values	
	Capsaicin	Capsanthin
Acetone	0.157	0.96
Ethyl alcohol	0.133	0.88
Dichloro ethane	0.184	0.85
Hexane	0.171	0.82
Benzene	0.150	0.82
Ethyl acetate	0.158	0.88

4.1.9 Extraction efficiency of solvents for pungency - Scoville Heat Units

Scoville Heat unit was calculated from the capsaicin content of oleoresin and presented in Table 4. Maximum heat unit was observed with oleoresin having maximum capsaicin content and vice-versa. Scoville unit of oleoresins prepared using ethyl acetate and ethyl alcohol were 11,365,80 and 4,666,20, respectively.

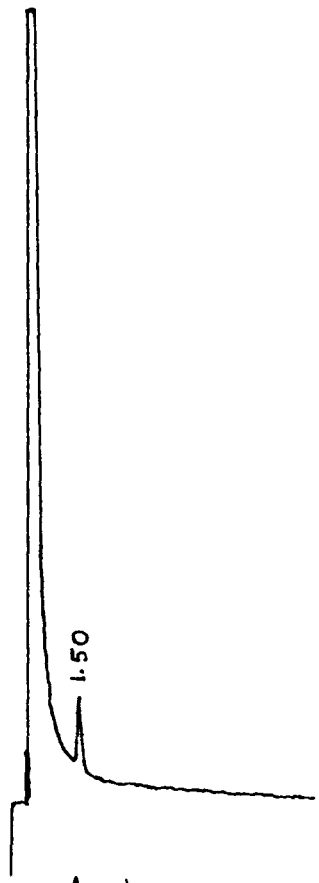
4.1.10 Solvent residue

Standard solvent peaks were noted from the graphs obtained from gas chromatograph and retention time was calculated. Based on the retention time, the solvent residue in oleoresin sample was measured (Table 6) and depicted in Fig (1, 2 & 3).

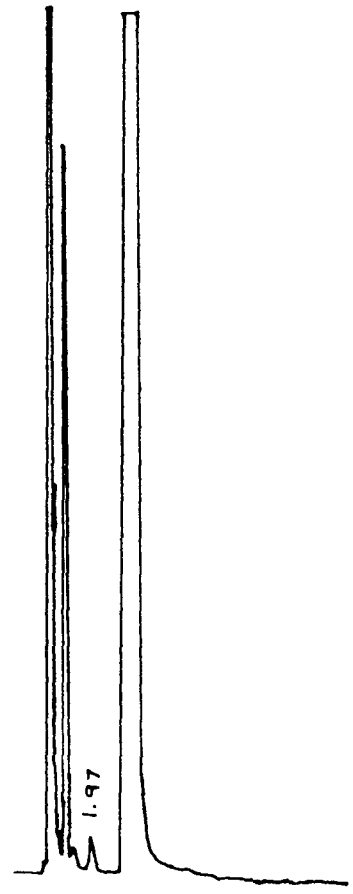
In the case of oleoresin yield, ethyl alcohol was superior, but considering the extraction efficiency of the solvent for quality determinants viz. colour and pungency, it was inferior.

Acetone was a better solvent for extraction of colour and regarding yield, it was second to ethyl alcohol. Though hexane appeared to be a good solvent for extraction of colour, it was lower in yield, compared to ethyl acetate.

FIG. 1 GC-CHROMATOGRAM OF SOLVENT RESIDUE IN OLEORESIN

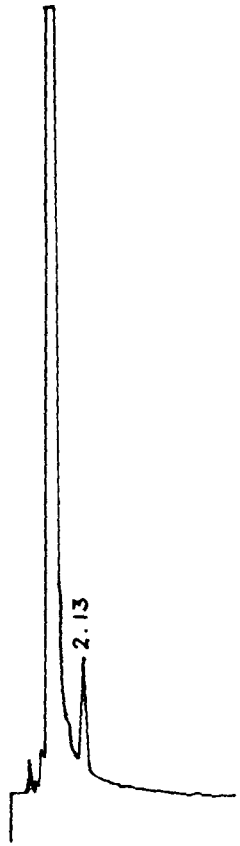


Acetone
Conc : 0.1188



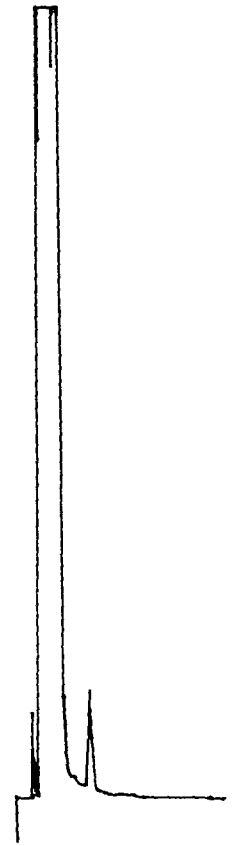
Ethyl alcohol
Conc: 0.0309

Fig: 2 GC - CHROMATOGRAM Of SOLVENT RESIDUE IN OLEORESIN



Dichloro ethane

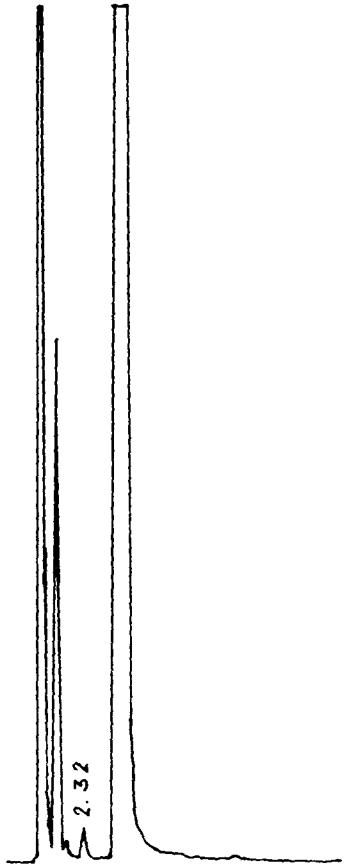
Conc: 0.2602



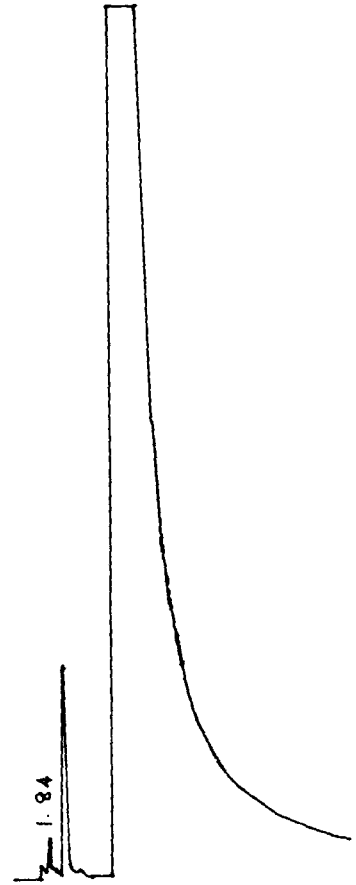
Hexane

Conc: 0.2485

FIG. 3 GC - CHROMATOGRAM OF SOLVENT RESIDUE IN OLEORESIN



Benzene
Conc: 0.127



Ethyl acetate
Conc: 0.0516

Table 6 Solvent residue in oleoresin samples

Solvents	Retention time	Concentration in g sample
Acetone	1.50	0.1188
Ethyl alcohol	1.97	0.0309
Dichloro ethane	2.13	0.2602
Hexane	1.22	0.2485
Benzene	2.32	0.1273
Ethyl acetate	1.84	0.0516

Ethyl acetate is the solvent having higher extraction efficiency for pungency and for colour extraction, it was on par with acetone. Ethyl acetate was the fastest in extraction and was comparable to acetone in yield. Above all, ethyl acetate is safe and the quality of oleoresin will not be affected by the presence of traces of solvents.

Considering all these aspects, ethyl acetate was standardised as the best solvent for extraction of chilli oleoresin for further studies of this work.

4.2 Evaluation of capsicum species and cultivars for oleoresin recovery

General analysis of variance showed significant difference among cultivars for all the characters studied.

4.2.1 Effect of season

Pooled analysis of variance over seasons (Table 7) showed that season x cultivar interaction was significant for all the characters viz. days to flower, days to fruitset, days to harvest, fruits/plant, fruit yield/plant and oleoresin yield (%). Mean performance of cultivars for these characters over season are presented in Tables 8 and 9.

Table 7 Pooled analysis of variance for different characters over seasons

Sources of variation	df	Mean squares					
		days to flower	days to fruitset	days to harvest	fruits/ plant	fruit yield/ plant	oleoresin yield
Season	2	1.03	1.20	0.58	34.16**	123.30**	1.59*
Cultivar	8	1.22*	3.44	0.86*	22.74**	30.09*	1.34**
Interaction (season x genotype)	16	0.36*	1.51*	0.28*	3.15*	9.26*	0.31*
Pooled error	48	0.09	0.03	0.08	0.04	0.06	0.51

* Significant at 5% level

** Significant at 1% level

4.2.1.1 Days to flower

Significant differences was observed among genotypes for days to first flowering during all the three seasons. During summer CA 653 was the earliest to flower, which took 79 days to first flowering and CA 640 the late which took 104.67 days. Ujwala was the earliest (54.67 days) in rainy season and CA 645 observed as late (123.67 days). Arka Lohit was the earliest in winter (80 days) and CA 645 the late (127.67 days). Considering all the seasons, Ujwala was the earliest (75.78 days) to flower and CA 645 lagged behind all other cultivars (118.33 days).

There was significant difference among seasons for days to flower. The genotypes were generally late in winter (104.26 days). No significant difference was noticed for the days taken to first flowering between summer and rainy seasons.

4.2.1.2 Days to fruitset

During summer season CA 653 was the earliest to set fruits (96 days) and CA 640 late (124 days). Ujwala was the earliest to set fruits during rainy (71.67 days) and winter (94.67 days) seasons. During rainy season CA 671 was the late (140 days) and CA 653 during winter (141.67). In general, Ujwala was the earliest genotype (91.11 days) to set fruits and CA 645 was the late (132.11 days). With regard to season, genotypes took more days to fruitset in winter (118.2 days) than in rainy (112.48 days) and summer (109.6 days).

4.2.1.3 Days to harvest

Ujwala was the earliest to harvest during summer and rainy seasons (113.67 and 121.33 days). CA 640 was the late (168.69 days) during summer and CA 653 (168 days) during rainy season. During winter KTPL-19 was the earliest (126 days) and CA 653 the late (205.33 days). In general, CA 653 took maximum (171.11) and KTPL-19 minimum days (130.78) to harvest irrespective of season.

With regard to season, the genotypes took more days to harvest during winter (158.51) than in summer (151.18 days) and rainy (146.17) seasons.

4.2.1.4 Fruits/Plant

Pooled analysis of variance indicated significant differences for fruits/plant among genotypes and also among seasons. Ujwala had maximum number of fruits/plant during summer and rainy seasons (68.67 and 29.67) and CA 670 the minimum (2.77 and 1.19). During winter, Arka Lohit produced more number of fruits/plant (23.43) followed by Ujwala (22.33). CA 670 was the lowest in fruits (1.29) per plant during winter.

In general, Ujwala was the best genotype for production of fruits (40.22), followed by Arka Lohit (29.0). CA 670 had the lowest fruits (1.83) per plant.

Table 8 Mean performance of cultivars for days to flower, fruitset and harvest

Genotype	Days to flower				Days to fruitset				Days to harvest			
	Summer	Rainy	Winter	Mean	Summer	Rainy	Winter	Mean	Summer	Rainy	Winter	Mean
CA 653	79.00 (8.88)	93.00 (9.64)	103.67 (10.18)	91.89 (9.57)	96.00 (9.80)	136.67 (11.69)	141.67 (11.90)	124.78 (11.13)	140.00 (11.83)	168.00 (12.96)	205.33 (14.33)	171.11 (13.04)
Arka Lohit	93.00 (9.64)	90.00 (9.49)	80.00 (8.93)	87.67 (9.35)	103.00 (10.15)	110.00 (10.48)	98.00 (9.89)	103.67 (10.17)	140.67 (11.86)	145.67 (12.07)	147.67 (12.15)	144.67 (12.03)
Ujwala	84.00 (9.17)	54.67 (7.35)	88.67 (9.41)	75.78 (8.64)	107.00 (10.34)	71.67 (8.46)	94.67 (9.73)	91.11 (9.51)	134.67 (11.60)	121.33 (11.01)	146.00 (12.08)	134.00 (11.56)
KTPL-19	95.00 (9.75)	78.67 (8.90)	95.67 (9.78)	89.78 (9.48)	107.67 (10.38)	88.67 (9.42)	106.67 (10.33)	101.00 (10.04)	140.00 (11.83)	126.33 (11.24)	126.00 (11.23)	130.78 (11.43)
CA 640	104.67 (10.21)	87.33 (9.35)	127.33 (11.28)	106.44 (10.28)	124.00 (11.14)	102.33 (10.12)	136.67 (11.69)	121.00 (10.98)	168.69 (12.99)	144.67 (12.03)	169.67 (13.03)	161.00 (12.68)
CA 645	103.67 (10.17)	123.67 (11.12)	127.67 (11.3)	118.33 (10.86)	116.67 (10.80)	139.00 (11.78)	140.67 (11.86)	132.11 (11.48)	158.00 (12.56)	167.33 (12.94)	168.00 (12.29)	164.44 (12.60)
CA 671	90.00 (9.49)	113.00 (10.63)	110.00 (10.49)	104.33 (10.20)	102.00 (10.10)	140.00 (11.83)	123.67 (11.12)	121.89 (11.02)	156.67 (12.51)	158.00 (12.57)	159.00 (12.61)	157.89 (12.56)
CA 648	97.00 (9,85)	94.00 (9.70)	103.67 (10.18)	98.22 (9.91)	115.33 (10.74)	115.67 (10.75)	113.33 (10.64)	114.78 (10.71)	161.00 (12.76)	142.33 (11.93)	165.33 (12.86)	156.89 (12.52)
CA 670	103.00 (10.15)	92.00 (9.59)	101.67 (10.08)	98.89 (9.94)	117.00 (10.82)	108.33 (10.41)	110.67 (10.68)	112.00 (10.63)	159.00 (12.61)	144.33 (12.01)	162.00 (12.73)	155.11(12.45)
Mean	94.37 (9.71)	91.80 (9.58)	104.26 (10.18)	96.81 (9.83)	109.60 (10.47)	112.48 (10.60)	118.20 (10.87)	113.40 (10.65)	151.18 (12.29)	146.17 (12.09)	158.51 (12.59)	151.95 (12.33)
CD (5%) Genotypes	0.60				1.23				0.53			
Season	0.35				0.71				0.31			
Interactiion	0.50				0.28				0.46			

Values in paranthesis show transformed values

On an average chilli genotypes produced maximum fruits per plant (26.27) during summer and minimum during rainy season (7.63).

4.2.1.5 Fruit yield/plant (g)

Fruit yield per plant was maximum (197.84 g) for CA 645 and minimum for CA 648 (5.34 g) during summer season. During rainy and winter season, Ujwala had maximum yield per plant (33.4 and 33.97 g). CA 671 had lowest yield (2.37 g) during rainy season and CA 648 (3.76 g) during winter. In general, CA 645 had maximum fruit yield/plant (74.69 g) followed by Ujwala (52.79 g). The lowest fruit yield was observed in CA 648 (4.49 g) irrespective of season.

Chilli genotypes produced maximum yield (58.63 g) per plant during summer season and minimum (10.31 g) during rainy season.

4.2.1.6 Oleoresin yield (%)

Significant difference was noticed among genotypes and seasons for oleoresin yield. CA 670 gave maximum percentage of oleoresin (30.4%) during summer and Ujwala, the minimum (13.33%). During rainy season,

Table 9 Mean performance of cultivars for yield of fruits

Genotype	Fruits per plant				Fruit yield per plant				Fruit yield per hectare (kg) *			
	Summer	Rainy	Winter	Mean	Summer	Rainy	Winter	Mean	Summer	Rainy	Winter	Mean
CA 653	5.00 (2.23)	3.03 (1.74)	6.73 (2.55)	4.92 (2.17)	12.70 (3.56)	3.33 (1.82)	10.85 (3.24)	8.96 (2.87)	627.20	164.4	535.80	442.50
Arka Lohit	51.67 (7.19)	11.90 (3.45)	23.43 (4.84)	29.00 (5.16)	81.67 (9.04)	11.98 (3.46)	26.43 (5.14)	40.03 (5.88)	4033.10	591.6	1305.2	1976.8
Ujwala	68.67 (8.29)	29.67 (5.45)	22.33 (4.72)	40.22 (6.15)	91.00 (9.54)	33.40 (5.78)	33.97 (5.82)	52.79 (7.05)	4493.80	1649.4	1677.5	2606.9
KTPL-19	11.33 (3.36)	4.97 (2.24)	4.77 (2.18)	7.02 (2.59)	40.90 (6.40)	14.23 (3.77)	9.38 (3.06)	21.50 (4.41)	2019.80	702.7	463.2	1061.7
CA 640	8.33 (2.88)	3.03 (1.72)	4.03 (2.01)	5.13 (2.2)	41.8 (6.46)	3.29 (1.80)	10.11 (3.18)	14.60 (3.82)	2064.20	162.5	499.3	908.6
CA 645	53.0 (7.28)	5.33 (2.31)	5.57 (2.36)	21.30 (3.98)	197.84 (14.06)	10.76 (3.28)	15.47 (3.93)	74.69 (7.09)	9769.9	531.4	764.0	3688.4
CA 671	30.0 (5.46)	5.10 (2.26)	5.04 (2.24)	13.38 (3.32)	34.80 (5.87)	2.37 (1.53)	4.33 (2.08)	13.83 (3.16)	1718.5	117.0	213.8	683.0
CA 648	5.63 (2.37)	4.23 (2.06)	4.03 (2.01)	4.63 (2.14)	5.34 (2.31)	4.37 (2.09)	3.76 (1.94)	4.49 (2.11)	263.7	215.8	185.7	221.7
CA 670	2.77 (1.66)	1.43 (1.19)	1.29 (1.13)	1.83 (1.33)	21.61 (4.65)	5.07 (2.97)	7.70 (2.77)	12.79 (3.46)	1067.2	250.4	380.2	631.6
Mean	26.27 (4.52)	7.63 (2.49)	8.58 (2.67)	14.16 (3.76)	58.63 (6.88)	10.31 (2.94)	13.55 (3.48)	27.50 (5.24)	2895.3	509.1	669.1	1358.0
CD (5%) Genotypes	1.80				3.04				* Calculated values			
Season	1.02				1.80							
Interactiion	0.33				0.40							

Values in paranthesis show transformed values

maximum percentage of oleoresin was produced by Arka Lohit (32.43) followed by KTPL-19 (25.87). KTPL-19 had the highest oleoresin percentage (37.83) during winter, followed by Arka Lohit (37). CA 671 was poor in oleoresin during rainy and winter seasons (9.2 and 13.63%). In general, Arka Lohit and KTPL-19 were on par (33.14% and 30.12%) in oleoresin content. CA 671 was the poorest oleoresin yielder (13.17%) irrespective of season. With regard to seasons, the genotypes grown during winter season had maximum oleoresin (27.24%) followed by summer (23.76%) and rainy seasons (19.13%). Considering the yield and oleoresin together, summer is the best season for total oleoresin yield per unit area (Table 10).

Correlation studies

The association between oleoresin yield and its components were also studied at genotypic level and the results are presented in Table 11.

Among the component characters, only fruits/plant had positive and significant genotypic correlation with oleoresin yield (r_g=0.26). The study revealed that the association of attributes of earliness viz. days to flower, fruitset and harvest with oleoresin yield were negative. Days to fruitset had highest negative correlation with oleoresin yield. The correlation of fruit yield/plant with yield of oleoresin was not significant, though positive (0.047).

Table 10 Mean performance of cultivars for oleoresin yield

Genotype	Yield of oleoresin (%)				Yield of oleoresin per hectare (kg) *			
	Summer	Rainy	Winter	Mean	Summer	Rainy	Winter	Mean
CA 653	28.10 (5.30)	15.77 (3.97)	15.20 (3.82)	16.96 (4.31)	37.00	5.60	17.30	15.85
Arka Lohit	30.00 (5.42)	32.43 (5.68)	37.00 (6.06)	33.14 (5.72)	272.20	43.55	109.10	147.6
Ujwala	13.33 (3.60)	19.97 (4.47)	36.67 (6.03)	23.82 (4.70)	121.60	67.80	125.50	126.60
KTPL-19	26.67 (4.99)	25.87 (5.08)	37.83 (6.10)	30.12 (5.39)	107.70	38.20	35.80	64.90
CA 640	23.33 (4.81)	17.33 (4.16)	29.57 (5.43)	23.41 (4.80)	96.80	5.90	30.00	42.90
CA 645	20.00 (4.37)	10.00 (3.16)	22.33 (4.72)	17.44 (4.08)	392.70	11.16	35.00	129.75
CA 671	16.67 (4.03)	9.20 (3.02)	13.63 (3.67)	13.17 (3.58)	63.00	2.40	6.40	19.80
CA 648	25.37 (5.02)	18.43 (4.32)	24.60 (4.96)	22.93 (4.76)	14.70	9.10	14.00	12.60
CA 670	30.40 (5.47)	22.77 (4.77)	28.40 (5.33)	27.19 (5.19)	61.30	10.90	20.50	29.14
Mean	23.76 (4.87)	19.13 (4.37)	27.24 (5.22)	23.38 (4.89)	141.80	19.60	38.70	65.60
CD (5%) Genotypes	0.56				* Calculated Values			
Season	0.32							
Interactiion	1.85							

Values in paranthesis show transformed values

Table 11 Genotypic correlation coefficient (r_g) among different parameters

Characters	Days to fruitset	Days to harvest	Fruits/ Plant	Fruit yield/ plant	Oleoresin yield
Days to flower	0.876**	0.733**	-0.535**	0.400*	-0.616**
Days to fruitset		0.990**	-0.744**	0.016	-0.802**
Days to harvest			-0.625**	-0.350**	-0.664**
Fruits/ plant				0.844**	0.260*
Fruit yield/ plant					0.047

* Significant at 5% level

** Significant at 1% level

Inter-correlation among different characters

Days to flower had significant and positive association with days to first fruitset (r_g=0.876), days to first harvest (r_g=0.733) and fruit yield/plant (r_g=0.4). Days to fruitset had a significant and positive association with days to first harvest (r_g=1.08) and negative association with fruits/plant (r_g=-0.744). Days to harvest had significant negative association with fruits/plant (r_g=-0.625) and fruit yield/plant (r_g=0.844).

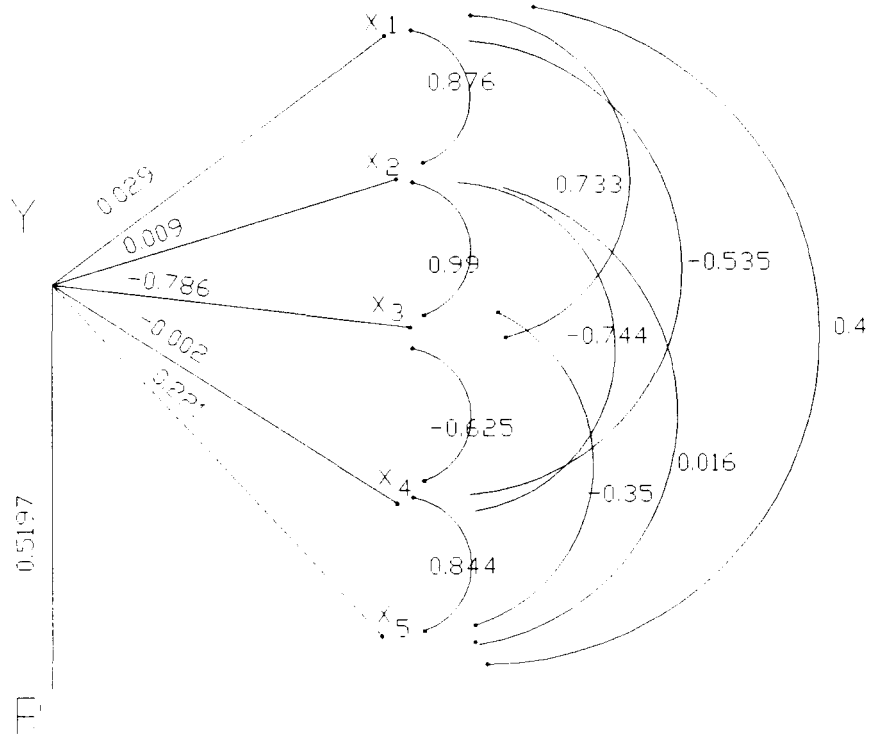
Path coefficient analysis

The direct and indirect contribution of the characters on yield of oleoresin was found out by partitioning the correlation between yield and component characters into direct and indirect effects (Table 12). All the parameters were selected for path coefficient analysis (Fig. 4).

Days to flower exhibited the highest positive direct effect on yield of oleoresin (0.029). This was followed by days to fruitset (0.009). The other two characters namely days to harvest and fruits per plant exhibited negative direct effect on yield of oleoresin (-0.786 and -0.022 respectively).

Days to flower had a positive and indirect effect on yield of oleoresin through days to fruitset and fruits per plant (0.008 & 0.012). The indirect effect of days to flower through days to harvest (-0.576) was negative and significant.

Fig. 4 Path diagram showing direct and indirect effects.



P – Residual effect

X_1 – Days to flower

X_2 – Days to fruitset

X_3 – Days to harvest

X_4 – Fruits per plant

X_5 – Fruit Yield per plant

Y – Oleoresin yield

Days to fruitset had a positive indirect effect on yield of oleoresin through days to flower (0.025) and fruits/plant (0.016). But this character had a significant and negative effect on oleoresin yield through days to harvest (-0.848). Days to harvest had low, positive effect on yield of oleoresin through days to flower (0.021), days to fruitset (0.010) and fruits/plant (0.013). Fruits/plant had a low and negative effect on yield of oleoresin through days to flower (-0.015) and days to fruitset (-0.007). But fruits/plant had a high positive effect on yield of oleoresin through days to harvest (0.491).

Phenotypic stability

Stability parameters like regression coefficient b(i) and deviation from regression $S^2d(i)$ for days to flowering, fruitset, days to harvest, fruits per plant, fruit weight/plant and yield of oleoresin were worked out as per Eberhart and Russel (1966) and are presented in Table 13 and 14.

Based on the grand mean performance, the cultivar Ujwala was the earliest to flower and fruit. Ujwala took only 75.78 days to flower and 91.11 days to fruitset. Ujwala did not show much difference in days to harvest (134 days) from KTPL-19, which was the earliest to harvest (130.78 days). CA 645 took maximum days to flower (118.33), fruitset (132.11) and harvest (164.44).

Arka Lohit and KTPL-19 were stable with regard to days to flowerset . CA 640 was an above average stable genotype.

Table 12 Direct and indirect effect of characters on oleoresin yield

Characters	Days to flower	Days to fruitset	Days to harvest	Fruit/plant	Fruit yield/ plant
Days to flower	0.029	0.008	-0.576	0.012	-0.089
Days to fruitset	0.025	0.009	-0.848	0.016	-0.004
Days to harvest	0.021	0.010	-0.786	0.013	0.077
Fruits/ plant	-0.015	-0.007	0.491	-0.022	-0.187
Fruit yield/ plant	0.011	0.002	0.275	-0.018	-0.221

The underlined diagonal values indicate direct effect

Residual : 0.5197

Considering the regression coefficient approximately equal to unity [b(i)→ 1] and deviation from regression, not significantly different from zero [$S^2d(i)\rightarrow 0$] *C. baccatum* and CA 645 were stable genotypes with regard to days to harvest. CA 653 was an above average stable genotype with regard to days to fruitset and harvest.

Arka Lohit, Ujwala and CA 645 were above average stable genotypes for fruits per plant. Considering the grand mean, Ujwala had maximum fruits per plant and *C. baccatum* produced the lowest number of fruits/plant.

Considering the grand mean, CA 645 was had highest fruit yield per plant (74.69 g), followed by Ujwala (52.79 g). Arka Lohit and Ujwala were stable for fruit yield. Considering the grand mean, Arka Lohit had the highest oleoresin recovery (33.14) and CA 671 had the lowest (13.17). Regarding oleoresin recovery, KTPL-19 was stable. Ujwala and CA 645 were above average stable genotypes as indicated by high b(i) values.

4.2.2 Effect of harvest maturity

Pooled analysis of variance over different harvest maturity have been done (Table 15 & 16). Mean performance of genotypes for the characters over three different harvest maturity are presented in Table 17, 18 and 19.

Table 13 Stability parameters for days to flower, fruitset and harvest

Genotype	Days to flower			Days to fruitset			Days to harvest		
	Mean	b(i)	$S^2d(i)$	Mean	b(i)	$S^2d(i)$	Mean	b(i)	$S^2d(i)$
CA 653	91.89	1.32	148.46	124.78	4.55	447.11	171.11	3.18	1025.70
Arka Lohit	87.67	0.95	7.10	103.67	-0.83	41.06	155.67	0.22	12.88
Ujwala	75.78	2.10	290.33	91.11	-0.53	627.13	134.00	1.61	10.39
KTPL-19	89.78	1.00	90.38	101.00	0.42	216.71	130.78	-0.23	114.00
CA 640	106.44	2.96	41.51	121.00	2.21	409.05	161.00	1.47	153.51
CA 645	118.33	0.91	250.86	132.11	2.36	137.48	164.44	1.19	50.86
CA 671	104.33	0.39	291.21	121.89	1.62	619.55	157.89	0.09	-5.83
CA 648	98.22	0.75	-7.50	114.78	-0.26	-4.57	156.89	1.38	103.63
CA 670	98.89	0.52	40.39	112.00	-0.54	23.60	155.11	1.08	43.30
Mean	96.82			113.59			152.88		

Table 14 Stability parameters for yield of fruits and oleoresin

Genotype	Fruit/plant			Fruit yield/plant			Yield of oleoresin		
	Mean	b(i)	$S^2d(i)$	Mean	b(i)	$S^2d(i)$	Mean	b(i)	$S^2d(i)$
CA 653	4.92	0.01	6.04	8.96	0.13	23.68	19.69	0.08	89.09
Arka Lohit	29.00	1.89	46.76	40.03	1.35	49.27	33.14	0.50	-0.18
Ujwala	40.22	2.33	44.83	52.79	1.22	4.15	23.82	1.87	156.04
KTPL-19	7.02	0.35	-0.63	21.50	0.62	21.86	30.12	1.40	7.08
CA 640	5.13	0.27	-0.49	18.40	0.76	7.90	23.41	1.50	16.36
CA 645	21.30	2.61	1.75	74.69	3.95	31.25	17.44	1.55	-11.19
CA 671	13.38	1.37	0.16	13.83	0.67	-1.62	13.17	0.60	-0.86
CA 648	4.63	0.08	-0.73	4.49	0.03	-1.40	22.93	0.75	10.04
CA 670	1.83	0.08	-0.75	12.79	0.28	0.95	27.19	0.74	-4.07
Mean	14.16			27.50			23.38		

Table 15 Pooled analysis of variance for different characters over season

Sources of variation	df	Mean Squares								
		Fruit per plant			Fruit yield per plant			Oleoresin yield		
		M1	M2	M3	M1	M2	M3	M1	M2	M3
Genotypes	8	7.26**	7.22**	7.40**	7.33**	27.99*	5.94**	8.71**	5.74**	1.13
Season	2	10.24**	8.31**	9.04**	44.50**	110.43**	17.60**	4.04	8.49	0.97
Interaction (Genotypes x Season)	16	0.65*	0.95*	0.57*	1.69*	8.46*	0.79*	1.50	0.81	0.61*
Pooled error	48	0.03	0.59	0.02	0.11	0.04	0.02	1.18	0.59	0.61

* Significant at 5% level

** Significant at 1% level

Table 16 Pooled analysis of variance for different characters over maturity

Sources of variation	df	Mean Squares								
		Fruit per plant			Fruit yield per plant			Oleoresin yield		
		Summer	Rainy	Winter	Summer	Rainy	Winter	Summer	Rainy	Winter
Genotypes	8	45.62**			20.65**	5.02**	4.44**	1.41	3.49*	2.17*
Season	2	0.17			5.63**	0.50	0.28*	0.74	5.39*	0.13
Interaction (Genotypes x Season)	16	0.41*	Not Significant		1.26*	0.21*	0.06*	0.98*	1.19	0.61*
Pooled error	48	0.06			0.12	0.02	0.03	0.67	1.02	0.69

* Significant at 5% level

** Significant at 1% level

4.2.2.1 Fruits/plant

When fruits were harvested at turning stage (M1) during summer, Arka Lohit produced maximum fruits per plant (59.2) followed by Ujwala (51.9). During rainy season, Ujwala produced maximum fruits/plant (31) followed by Arka Lohit (12.7). During winter season, Arka Lohit and Ujwala were on par in fruits/plant (25.7 and 23.6). CA 670 had lowest fruits per plant in all the three seasons (2.5, 1.41 and 1.21).

When harvested at full ripe stage (M2), Arka Lohit, Ujwala and CA 645 produced maximum fruits per plant (59.3, 55.8 and 41.7) during summer. During rainy and winter seasons, Ujwala had maximum fruits per plant (30.3 and 26.3). CA 670 produced minimum fruits per plant in all the three seasons (2.53, 1.41 and 1.1) when harvested at full ripe stage.

When harvesting was delayed to withering stage (M3), Arka Lohit produced maximum (58.6) fruits per plant, followed by Ujwala (52.6) during summer. During rainy and winter seasons, Ujwala produced maximum fruits per plant (31.3 and 26.3) followed by Arka Lohit. CA 670 had least fruits per plant during all the three seasons.

In general, Ujwala and Arka Lohit were superior for fruits per plant, when harvested at turning, full ripe or withering stage, irrespective of seasons. CA 670 was lowest in fruits/plant.

In general, Capsicum genotypes had more fruits per plant during summer followed by winter and rainy seasons irrespective of stages of harvest maturity.

During summer and winter cultivars showed significant difference in fruits/plant, between three maturity stages.

Further analysis revealed that season x maturity interaction was non-significant. Considering genotype, season and stages of harvest maturity, the maximum fruits/plant was observed in Arka Lohit during summer when harvesting was done at full ripe stage which is on par with the other two stages of harvest of the same variety and also with Ujwala harvested at full ripe stage in the same season.

4.2.2.2 Fruit yield/plant (g)

When harvested at turning stage, CA 645 had highest (129.2 g) yield per plant, followed by Arka Lohit (96.4) during summer. Ujwala produced highest fruit weight (31 g) followed by Arka Lohit (12.7 g) during rainy

Table 17 Mean performance of cultivars at different harvest maturity for yield of fruit

Genotype	Fruits per plant											
	Summer				Rainy				Winter			
	M1	M2	M3	Mean	M1	M2	M3	Mean	M1	M2	M3	Mean
CA 653	13.70 (3.70)	5.00 (2.21)	8.53 (2.91)	9.08 (2.94)	3.06 (1.74)	3.03 (1.74)	3.00 (1.74)	3.03 (1.74)	6.50 (2.53)	5.67 (2.36)	5.97 (2.43)	6.05 (2.44)
Arka Lohit	59.20 (7.69)	59.30 (7.70)	58.60 (7.65)	59.03 (7.68)	12.70 (3.56)	12.40 (3.51)	11.83 (3.44)	12.31 (3.50)	23.60 (4.85)	21.97 (4.68)	22.40 (4.70)	22.70 (4.74)
Ujwala	51.90 (7.20)	55.80 (7.46)	52.60 (7.25)	53.40 (7.30)	31.00 (5.56)	30.30 (5.50)	31.30 (5.59)	30.90 (5.55)	25.70 (5.06)	26.30 (5.12)	26.30 (5.12)	26.10 (5.10)
KTPL-19	9.40 (3.06)	9.10 (3.01)	10.40 (3.21)	9.63 (3.09)	4.97 (2.22)	5.03 (2.24)	5.00 (2.23)	5.00 (2.23)	5.03 (2.24)	5.17 (2.27)	5.03 (2.24)	5.08 (2.77)
CA 640	5.33 (2.30)	10.00 (3.16)	5.03 (2.24)	6.73 (2.56)	2.10 (1.44)	3.37 (1.82)	2.03 (1.42)	2.50 (1.56)	3.63 (1.90)	3.77 (1.94)	3.37 (1.83)	3.53 (2.37)
CA 645	36.80 (6.05)	41.70 (6.45)	30.20 (5.47)	36.20 (5.99)	5.03 (2.24)	5.20 (2.27)	5.03 (2.24)	5.09 (2.25)	5.03 (2.24)	5.00 (2.24)	5.10 (2.25)	5.04 (2.77)
CA 671	29.20 (5.38)	30.50 (5.50)	27.70 (5.25)	29.10 (5.38)	5.20 (2.27)	5.11 (2.25)	5.07 (2.25)	5.13 (2.26)	5.13 (2.26)	4.97 (2.23)	5.10 (2.25)	5.17 (2.25)
CA 648	8.00 (2.81)	6.40 (2.52)	8.30 (2.86)	7.57 (2.73)	4.13 (2.03)	4.23 (2.05)	4.03 (2.00)	4.13 (2.03)	4.07 (2.01)	4.21 (2.05)	4.33 (2.08)	4.20 (2.04)
CA 670	2.50 (1.57)	2.53 (1.58)	2.60 (1.60)	2.54 (1.58)	1.41 (1.17)	1.42 (1.17)	1.10 (1.04)	1.31 (1.13)	1.21 (1.09)	1.10 (1.04)	1.30 (0.97)	1.20 (1.04)
Mean	24.00 (4.42)	24.28 (4.40)	22.66 (4.27)	23.71 (4.36)	6.10 (2.47)	6.25 (2.50)	5.95 (2.44)	6.10 (2.47)	7.24 (2.69)	10.04 (3.17)	7.02 (2.65)	8.10 (2.85)

Values in paranthesis show transformed values

CD (P=0.05) for comparison of (1) seasonal means = 0.05 (2) maturity means = 0.05 (3) Varietal means = 0.09
two way combinations (1) season x genotype = 0.15 (2) maturity x genotypes = 0.15
three way combinations = 0.27

season. CA 670 had the lowest (1.41) yield during rainy season. During winter, Ujwala and Arka Lohit were on par for yield per plant.

When harvested at full ripe stage, CA 645 produced maximum yield per plant (162.7 g) followed by Arka Lohit (104.3 g) during summer. During rainy and winter seasons, Ujwala produced the highest fruit yield/plant (32.4 and 39.8 g). CA 671 produced lowest (2.95 g) fruit yield per plant during rainy season. CA 648 had the lowest fruit yield during summer and winter seasons (5.06 and 3.91 g) at full ripe stage.

At withering stage, Arka Lohit recorded the highest yield per plant (76.8 g) followed by Ujwala (60.9 g) during summer season. During rainy season, Ujwala produced the highest yield (32.7 g) followed by KTPL-19 (14.16 g). CA 671 and CA 640 were lowest in yield per plant during rainy season (1.96 and 1.95 g). During winter seasons, Ujwala and Arka Lohit did not differ significantly in fruit yield per plant (22.7 and 22.64 g). CA 648 yielded lowest during summer and winter seasons (4.19 and 3.44 g). In general, Ujwala and Arka Lohit were high yielders, irrespective of seasons, when fruits were harvested at withering stage.

Considering all the seasons, yield was maximum in summer, followed by winter and rainy seasons, irrespective of stages of harvest.

During summer and winter seasons cultivars did not differ significantly in yield/plant when harvested at turning or full ripe stage, but yield was lowest at withering stage.

Plant yield was maximum when harvested at full ripe stage, moderate at withering stage and least at turning stage.

The interaction between season x maturity was significant for yield/plant. Highest fruit yield was observed in summer season when fruits were harvested at turning and full ripe stage. The interaction of genotypes with season and stages of harvest maturity was also significant. Considering genotypes in relation to season and maturity stages, yield was maximum (162.7 g) in CA 645 during summer, when harvested at full ripe stage.

4.2.2.3 Oleoresin yield (%)

The cultivars belonging to *C. annuum* had high oleoresin yield when fruits were harvested at turning stage. CA 670 had the lowest oleoresin yield. At full ripe stage, Arka Lohit and KTPL-19 had highest oleoresin recovery irrespective of seasons.

When harvesting was delayed to withering stage, Arka Lohit, CA 670, CA 645 and Ujwala were higher in oleoresin recovery.

Table 18 Mean performance of cultivars at different harvest maturity for yield of fruit

Genotype	Fruit yield per plant											
	Summer				Rainy				Winter			
	M1	M2	M3	Mean	M1	M2	M3	Mean	M1	M2	M3	Mean
CA 653	36.90 (6.07)	12.90 (3.59)	17.80 (4.22)	22.50 (4.63)	3.06 (1.75)	3.37 (1.83)	3.13 (1.77)	3.19 (1.78)	10.16 (3.19)	9.03 (2.99)	8.30 (2.87)	9.16 (3.02)
Arka Lohit	96.40 (9.80)	104.30 (10.21)	76.80 (8.76)	92.50 (9.60)	12.70 (3.56)	11.90 (3.45)	10.32 (3.21)	11.64 (3.41)	27.99 (5.29)	24.20 (4.92)	22.64 (4.76)	24.94 (4.99)
Ujwala	73.90 (8.59)	74.30 (8.61)	60.90 (7.81)	69.70 (8.34)	31.00 (5.57)	32.40 (5.69)	32.70 (5.72)	32.03 (5.66)	29.20 (5.41)	39.80 (6.31)	22.70 (4.77)	20.80 (5.50)
KTPL-19	36.23 (6.02)	34.50 (5.87)	28.70 (5.36)	33.14 (5.75)	4.97 (2.23)	14.40 (3.79)	14.16 (3.76)	11.17 (3.26)	10.48 (3.24)	10.10 (3.17)	8.73 (2.95)	9.77 (3.12)
CA 640	25.97 (5.09)	45.60 (6.75)	19.50 (4.42)	30.36 (5.42)	2.10 (1.45)	3.31 (1.80)	1.95 (1.39)	2.45 (1.55)	9.45 (3.07)	9.58 (3.09)	7.75 (2.78)	8.93 (2.98)
CA 645	129.20 (11.36)	162.70 (12.75)	41.60 (6.45)	111.20 (10.19)	5.03 (2.24)	10.13 (3.18)	9.59 (3.10)	8.25 (2.84)	14.60 (3.82)	13.97 (3.74)	13.70 (3.70)	14.09 (3.75)
CA 671	44.60 (6.58)	35.60 (5.94)	27.70 (5.26)	35.97 (5.93)	5.20 (2.28)	2.95 (1.71)	1.96 (1.40)	3.37 (1.8)	4.71 (2.17)	4.29 (2.07)	3.72 (1.93)	4.24 (2.05)
CA 648	8.80 (2.99)	5.06 (2.25)	4.19 (2.04)	6.02 (2.43)	4.13 (2.03)	4.46 (2.11)	3.26 (1.80)	3.95 (1.98)	4.31 (2.07)	3.91 (1.98))	3.44 (1.85)	3.89 (1.97)
CA 670	16.10 (3.98)	15.97 (4.00)	12.99 (3.59)	14.99 (3.86)	1.41 (1.17)	8.83 (2.95)	5.57 (2.36)	5.27 (2.16)	7.79 (2.79)	6.63 (2.57)	6.68 (2.58)	7.03 (2.64)
Mean	52.01 6.72)	54.55 (6.66)	32.23 (5.32)	46.26 (6.20)	7.73 (2.48)	10.19 (2.95)	10.19 (2.72)	9.04 (2.72)	13.18 (3.45)	13.50 (3.43)	10.85 (3.13)	12.51 (3.34)

Values in paranthesis show transformed values

CD (P=0.05) for comparison of (1) seasonal means = 0.13 (2) maturity means = 0.13 (3) Varietal mean = 0.22

two way combinations (1) season x genotype = 0.39 (2) maturity x genotypes = 0.39 (3) season x maturity = 0.22

three way combinations = 0.67

In general, cultivars had high oleoresin during summer, when fruits were harvested at turning stage (26.4 %). But, when harvesting was delayed to fullripe or withering stage, highest oleoresin yield (26.5% and 26.2%) were observed during winter season.

During summer season, KTPL-19, Arka Lohit and CA 648 had high oleoresin recovery (34.7, 32.5 and 25.6%) irrespective of harvest maturity. During rainy season, Arka Lohit was the highest (35.5%) in oleoresin yield. During winter, Ujwala, KTPL-19 and Arka Lohit were high yielders of oleoresin (42.43, 35.8 and 32.4%).

During summer and winter seasons, the cultivars were on par in oleoresin yield, when the harvest maturity was fixed at turning, full ripe or withering stage. But in rainy season, oleoresin recovery was high in fruits at withering stage.

The interaction of season x stages of harvest was significant for percentage of oleoresin recovery. Maximum recovery (26.52%) was in winter season harvested at full ripe, withering or turning stage, which is on par with withering stage of rainy and turning and full ripe stage of summer seasons.

Table 19 Mean performance of cultivars at different harvest maturity for yield of oleoresin

Genotype	Yield of Oleoresin (%)											
	Summer				Rainy				Winter			
	M1	M2	M3	Mean	M1	M2	M3	Mean	M1	M2	M3	Mean
CA 653	30.00 (5.34)	13.00 (3.57)	16.30 (4.03)	19.80 (4.31)	23.60 (4.85)	12.60 (3.43)	20.80 (4.54)	19.00 (4.27)	30.20 (5.41)	14.20 (3.72)	16.60 (4.06)	20.30 (4.40)
Arka Lohit	30.00 (5.42)	37.30 (6.06)	30.30 (5.53)	32.50 (5.67)	38.80 (6.20)	32.10 (5.65)	35.50 (5.91)	35.50 (5.92)	29.90 (5.41)	37.70 (6.11)	29.60 (5.43)	32.40 (5.65)
Ujwala	50.00 (7.04)	13.60 (3.63)	10.00 (3.16)	24.50 (4.61)	13.90 (3.73)	20.20 (5.59)	19.00 (4.31)	17.70 (4.17)	35.30 (5.86)	35.30 (5.86)	56.70 (7.48)	42.43 (6.41)
KTPL-19	43.30 (6.51)	39.50 (6.24)	21.30 (4.61)	34.70 (5.79)	17.60 (4.13)	19.10 (4.11)	18.90 (4.34)	18.53 (4.19)	42.90 (6.47)	41.70 (6.38)	22.90 (4.79)	35.80 (4.35)
CA 640	20.00 (4.37)	23.30 (4.80)	13.30 (3.63)	18.90 (4.27)	16.40 (3.93)	17.40 (4.16)	25.50 (4.49)	19.80 (4.19)	20.70 (4.46)	24.40 (4.94)	13.80 (3.70)	12.70 (3.56)
CA 645	16.70 (3.93)	20.00 (4.37)	19.00 (3.56)	18.60 (3.98)	18.60 (4.18)	9.97 (3.15)	22.10 (4.69)	16.90 (4.01)	18.30 (4.17)	21.30 (4.61)	19.80 (4.36)	19.80 (4.38)
CA 671	15.00 (3.83)	16.20 (4.01)	16.70 (4.03)	15.96 (3.96)	18.80 (4.17)	9.30 (3.03)	20.10 (4.48)	16.10 (3.89)	14.60 (3.8)	14.30 (3.77)	18.20 (4.21)	15.70 (3.93)
CA 648	23.30 (4.70)	25.40 (5.01)	28.00 (5.28)	25.60 (5.01)	18.30 (4.18)	15.10 (3.75)	26.80 (5.16)	20.10 (4.36)	23.80 (4.75)	24.60 (4.95)	29.10 (5.38)	25.80 (5.02)
CA 670	9.60 (3.02)	25.00 (4.83)	28.30 (5.28)	20.97 (4.38)	6.20 (2.37)	16.03 (3.79)	29.80 (5.43)	17.34 (3.86)	9.50 (2.96)	25.20 (5.01)	29.30 (5.39)	21.30 (4.45)
Mean	26.40 (4.90)	23.70 (4.72)	20.35 (4.35)	23.50 (4.85)	19.10 (4.19)	16.90 (3.95)	24.30 (4.82)	20.10 (4.48)	25.02 (4.81)	26.52 (5.04)	26.20 (4.98)	25.10 (5.01)

Values in paranthesis show transformed values

CD (P=0.05) for comparison of (1) seasonal means = 0.28 (2) Varietal mean = 0.48

two way combinations (1) season x genotype = 0.83 (2) maturity x genotypes = 0.83 (3) season x maturity = 0.48

4.3 Influence of harvest maturity on quality of chilli

When the genotypes were analysed for quality parameters like colour, pungency and total sugar content over three harvest maturity stages, significant differences were observed for all those characters. Mean performance of different genotypes at the three maturity stages genotypes are presented in Table 20.

4.3.1 Colour value

When the harvesting was done at turning stage, CA 653 produced highest colour value (732) followed by Ujwala (691.33). KTPL-19 exhibited highest colour value at full ripe and withering stage (1687.67 and 3131.17). CA 645 produced the lowest colour value during the above two maturity stages (333.5 and 501). On an average, KTPL-19 exhibited highest (1809.61) colour value, followed by Ujwala (1511.44). CA 640 and CA 645 were poor in colour values (356.61 and 356.78).

In general, all the chilli genotypes exhibited highest colour values at withering stage, moderate at full ripe stage and lowest at turning stage.

4.3.2 Pungency

When the fruits were harvested at turning stage, CA 645 was the most pungent (21.39 mg/g capsaicin) genotype which was on par with CA 640,

Ujwala, Arka Lohit and CA 648. KTPL-19 was the least pungent (9.5 mg/g) genotype. When harvested at full ripe stage, Ujwala was highly pungent (24.4 mg/g) followed by KTPL-19 (24.27 mg/g). At withering stage, CA 645 had highest pungency (24.59 mg/g), closely followed by Ujwala (24.5 mg/g), which were on par with all other genotypes, except CA 653. Considering all stages, CA 640 was the most pungent (22.37 mg/g) genotype and CA 670, the least (11.99 mg/g). In general, chilli fruits were least pungent when harvested at turning stage (14.7 mg/g) and the pungency showed an increasing trend as the harvesting was progressed from turning to full ripe and withering stage. But the fruits harvested at full ripe and withering stages were on par in pungency in the case of Ujwala and KTPL-19.

4.3.3 Total sugars (%)

When the chilli fruits were harvested at turning stage, CA 653 had maximum sugar content (18.89%) followed by CA 640 (14.51%), CA 671 had lowest sugar content (9.64%) at turning stage. When the fruits were harvested at full ripe stage the sugar content of cultivars ranged from 11.84% to 16.15%. The cultivars did not differ significantly for sugar content at withering stage. In general, whatever be the stages of harvest, CA 653 was the highest (14.1%) in total sugar content, followed by CA 670 (13.3 %). CA 648 had minimum sugar content (11.1%) in fruits.

On analysing the cultivars for total sugar content at three different maturity stages, no significant difference was observed between turning and full ripe stage, chilli fruits at withering stage was low in (11.03%) in sugar content.

Table 20 Mean performance of cultivars for quality parameters

Genotype	Colour value				Pungency (mg/g)				Total sugar (%)			
	turning stage	full ripe	withering stage	Mean	turning stage	full ripe	withering stage	Mean	turning stage	full ripe	withering stage	Mean
CA 653	732.00	1057.33	1429.17	1072.83	10.04	13.54	16.56	13.38	18.89	12.63	10.70	14.10
Arka Lohit	569.33	1077.67	1992.67	1213.20	16.44	18.25	19.14	17.94	14.35	12.20	11.01	12.50
Ujwala	691.33	1433.50	2409.50	1511.44	15.17	24.40	24.50	21.35	12.21	11.84	10.17	11.40
KTPL-19	610.00	1687.67	3131.17	1809..61	9.50	24.27	18.20	17.31	13.21	12.67	11.27	12.40
CA 640	155.83	355.83	558.17	356.61	20.86	21.87	24.38	22.37	14.51	14.10	11.10	13.20
CA 645	233.83	335.50	501.00	356.78	21.39	20.51	24.59	22.16	10.80	13.77	10.65	11.74
CA 671	274.40	549.00	1250.50	691.30	13.39	18.04	18.64	16.70	9.64	16.15	11.24	12.30
CA 648	404.67	711.67	976.00	697.40	14.67	15.85	19.20	16.57	10.04	12.48	10.63	11.10
CA 670	484.07	1207.70	1582.83	1091.53	10.88	4.22	20.88	11.99	13.21	14.16	12.52	13.30
Mean	461.72	935.10	1536.78	977.86	14.70	17.90	20.70	17.75	12.99	13.33	11.03	12.45

4.4 Quality changes and storage stability of chilli oleoresin

Storage study was carried out at laboratory level using the oleoresin extracted from the variety Ujwala by ethyl acetate. When the oleoresin stored in different containers and kept in two different contrasting conditions (open and dark) were analysed for colour value and pungency, the samples showed significant differences for those values with changes in containers and storage conditions.

4.4.1 Colour value

A general reduction in colour value ranging from 57% in amber coloured bottles to 65% in PVC containers was noticed even one month after storage. Oleoresin stored in amber coloured bottles was superior in colour value (7220.88) while the PVC bottle was poor in colour retention.

At four months after storage, a general reduction of 63.6 to 68.6 % was noticed in colour values. However, no significant difference was noticed between bottles regarding colour value. Oleoresin stored in PET jars had better colour value (6146.05) compared to other containers.

General reduction in colour values ranged from 65.5 to 72.9% and 58.2 to 83.6% at six and eight month after storage. PET jar was better in retaining the colour value (5824.81 and 7041.69) at six and eight month after storage. Oleoresin stored in PVC jar was poor in colour value (4566.65 and 2768.94)

compared to samples stored in other bottles At twelve months after storage, a general reduction of 72.9 to 90.6 % was noticed in colour values. Amber coloured glass bottle was better in preserving the colour value (4574.6) compared to other bottles (Fig 5).

Oleoresin stored in dark was higher in colour value compared to samples kept in open condition irrespective of bottles. Still a reduction in colour values ranged from 58.8% in dark to 64.4% in open storage within a month of storage. The percentage reduction in colour value was lower from four months of storage onwards. Changes in colour values during storage are presented in Table 21 and depicted in Fig. 6.

4.4.2 Capsaicin (mg/g)

General reduction in capsaicin content ranged from 33.4% in PVC to 59.9% in PET jar within a month of storage. The comparison of bottles for retention of capsaicin content showed that, the capsaicin content was highest (50.44 mg/g) in samples stored in PVC and lowest (30.36 mg/g) in oleoresin kept in PET jar. From four month of storage onwards, the capsaicin content showed a gradual increase. The percentage reduction ranged from 32.8% in PVC to 12.3% in PET at four month after storage and 21.5 in PVC to 1.5% in polythene containers at six months after storage. At eight months of storage, the percentage reduction in capsaicin content was only 3.2% in amber coloured bottles, but in all other containers, capsaicin content has exceeded

Fig.5 Effect of bottles on colour value during storage

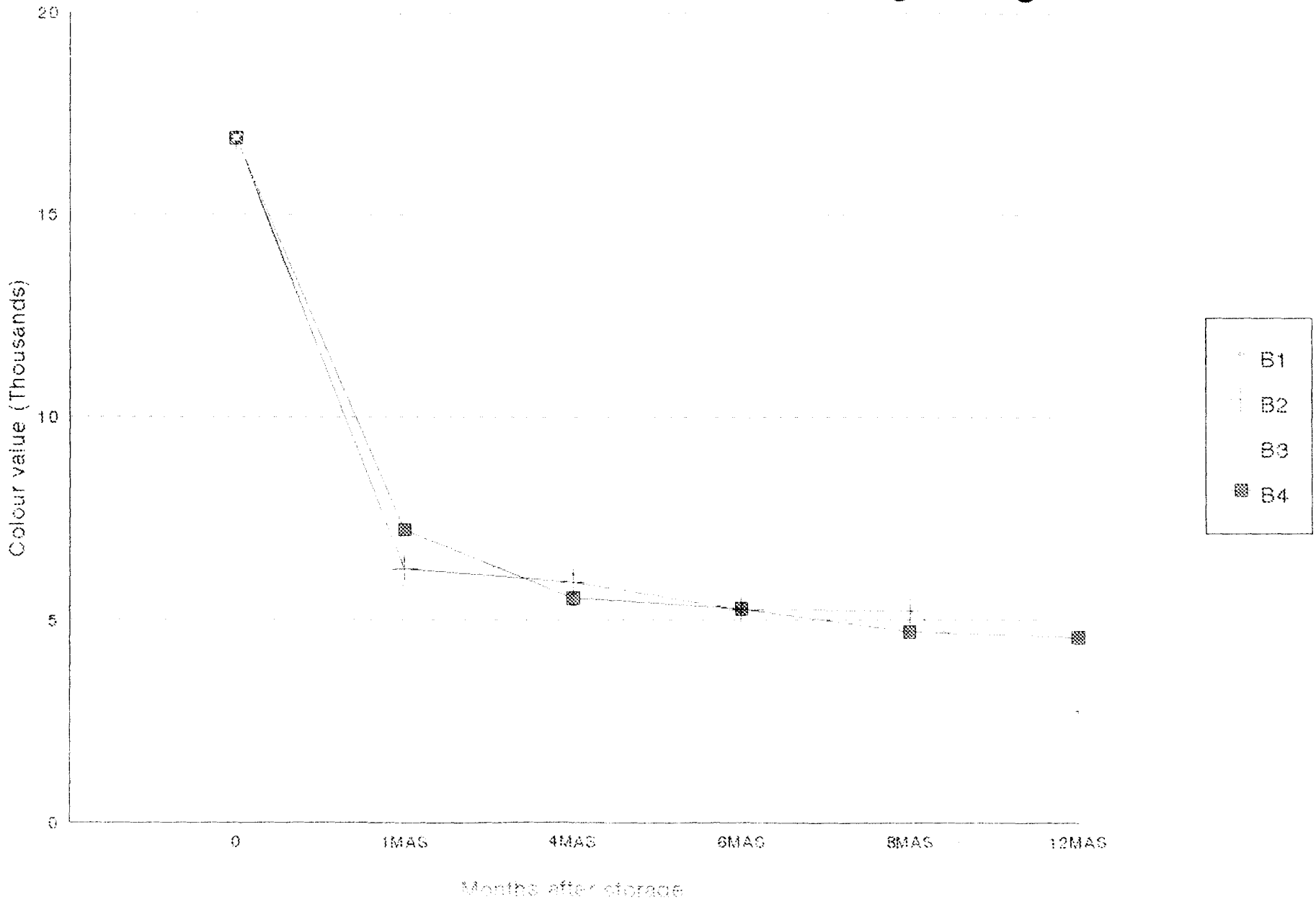


Fig.6 Changes in colour value during storage

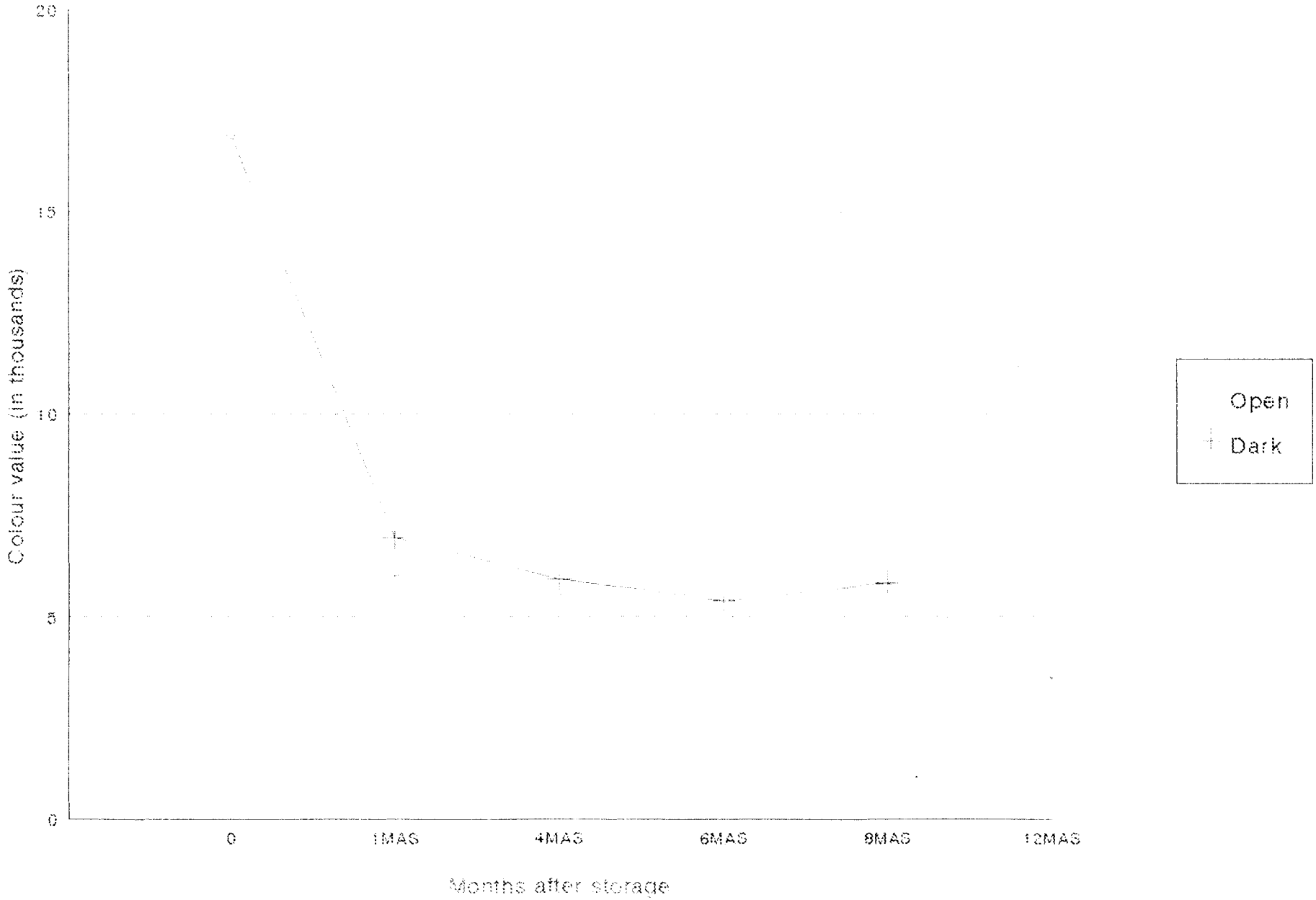


Table 21 Effect of storage on colour value

One month after storage					
	B1	B2	B3	B4	Mean
Open	5197.88 (69.2)	5923.38 (64.9)	6243.88 (63.0)	6633.76 (6.07)	5999.72 (64.4)
Dark	6625.75 (60.7)	6606.88 (60.8)	6744.88 (60.0)	7808.0 (53.7)	6946.38 (58.8)
Mean	5911.81 (65.0)	6265.13 (62.9)	6494.38 (61.5)	7220.88 (57.0)	6473.05 (61.6)
4 month after storage					
Open	5097.23 (69.8)	5196.5 (69.2)	6115.23 (63.7)	5704.6 (66.2)	5528.39 (67.2)
Dark	5508.5 (67.3)	6691.58 (60.3)	6176.88 (63.4)	5414.68 (67.9)	5947.91 (64.7)
Mean	5302.86 (68.6)	5944.04 (64.8)	6146.05 (63.6)	5559.64 (67.0)	5739.15 (65.9)
6 month after storage					
Open	4276.0 (74.6)	4926.0 (70.8)	5588.88 (66.9)	5475.8 (67.5)	5066.67 (70.0
Dark	4857.3 (71.2)	5587.3 (66.9)	6060.75 (64.1)	5096.63 (69.8)	5400.5 (67.9)
Mean	4566.65 (72.9)	5256.63 (68.8)	5824.81 (65.5)	5286.21 (68.7)	5233.6 (69.0)
8 month after storage					
Open	1387.75 (91.8)	4650.75 (72.4)	5322.25 (68.4)	4796.13 (71.6)	4039.22 (76.0)
Dark	4150.13 (75.4)	5806.88 (65.6)	8761.13 (48.1)	4628.38 (72.6)	5836.63 (65.4)
Mean	2768.94 (83.6)	5228.81 (69.0)	7041.69 (58.2)	4712.25 (72.1)	4937.92 (70.7)
12 month after storage					
Open	1285.75 (92.4)	2562.0 (84.8)	3461.75 (79.5)	4551.0 (73.1)	2965.13 (82.5)
Dark	1906.25 (88.7)	2943.25 (82.6)	4529.25 (73.2)	4598.2 (72.8)	3494.24 (79.3)
Mean	1596.0 (90.6)	2752.63 (83.7)	3995.5 (76.4)	4574.6 (72.9)	3229.68 (80.9)

Values in paranthesis indicates % decrease from the initial value

the initial value. At four month after storage, oleoresin stored in PET jar had highest pungency (66.41 mg/g). Oleoresin stored in polythene jar had highest pungency at six and eight months of storage (74.6 and 87.33 mg/g).

When oleoresin samples stored in different containers were analysed for capsaicin content at twelve months after storage, the pungency was found decreased. Pungency was better preserved in PET jars (55.55) and samples stored in polythene jar had lowest capsaicin (41.66) content.

A reduction in capsaicin content was 32.9% in dark and 54.7% in open at one month after storage. At four month after storage, reduction ranged from 22.1% in dark to 27.1% in open storage. Till four month after storage, capsaicin content was higher in dark compared to open storage. But during six, eight and twelve months after storage, open storage was better in keeping a high pungency level in oleoresin. The capsaicin content was 70.28 mg/g in open and 62.67 in dark at six month after storage and the content was increased to 82.74 mg/g in open and 76.73 in dark at eight months after storage. At twelve months after storage, the capsaicin content was 38.01 mg/g in dark and 56.78 mg/g in open condition. Changes in capsaicin content during storage is presented in Table 22 and depicted in Fig. 7 and 8

Table 22 Effect of storage on capsaicin (mg/g)

One month after storage					
	B1	B2	B3	B4	Mean
Open	36.33 (-52)	43.83 (-42.1)	23.33 (-69.2)	34.16 (-54.0)	34.41 (-54.7)
Dark	64.55 (-14.8)	54.11 (-28.6)	37.39 (-50.6)	46.99 (-38.0)	50.76 (-32.9)
Mean	50.44 (-33.4)	48.97 (-35.3)	30.36 (-59.9)	40.58 (-46.4)	42.59 (-43.8)
4 month after storage					
Open	45.66 (-39.7)	47.83 (-36.9)	67.72 (-10.6)	59.66 (-21.2)	55.22 (-27.1)
Dark	56.21 (-25.8)	56.1 (-25.9)	65.1 (-14.1)	58.55 (-22.7)	58.99 (-22.1)
Mean	50.94 (-32.8)	51.96 (-31.4)	66.41 (-12.3)	59.1 (-22.0)	57.1 (-24.6)
6 month after storage					
Open	62.61 (-17.3)	84.12 (11.05)	55.45 (-26.8)	78.96 (4.2)	70.28 (-7.2)
Dark	56.33 (-25.7)	65.08 (-14.1)	67.43 (-10.9)	61.84 (-18.4)	62.68 (-17.3)
Mean	59.47 (-21.5)	74.6 (-1.5)	61.44 (-18.9)	70.4 (-7.1)	66.48 (-12.2)
8 month after storage					
Open	67.77 (-10.5)	97.46 (28.7)	88.16 (16.3)	77.55 (2.4)	82.74 (9.2)
Dark	85.99 (13.5)	77.16 (1.9)	74.66 (-1.4)	69.11 (-8.8)	76.73 (1.3)
Mean	76.88 (1.5)	87.33 (15.3)	81.41 (7.5)	73.33 (-3.2)	79.74 (5.3)
12 month after storage					
Open	52.89 (-30)	48.44 (-35.9)	50.44 (-33.2)	75.33 (-0.3)	56.78 (-24.8)
Dark	42.88 (-43.3)	34.88 (-53.8)	38.66 (-48.8)	35.77 (-52.6)	38.01 (-49.7)
Mean	47.88 (-36.6)	41.66 (-44.9)	44.55 (-41.0)	55.55 (-26.5)	47.4 (-37.3)

Values in paranthesis indicates % decrease from the initial value

Fig. 7 Effect of bottles on capsaicin during storage

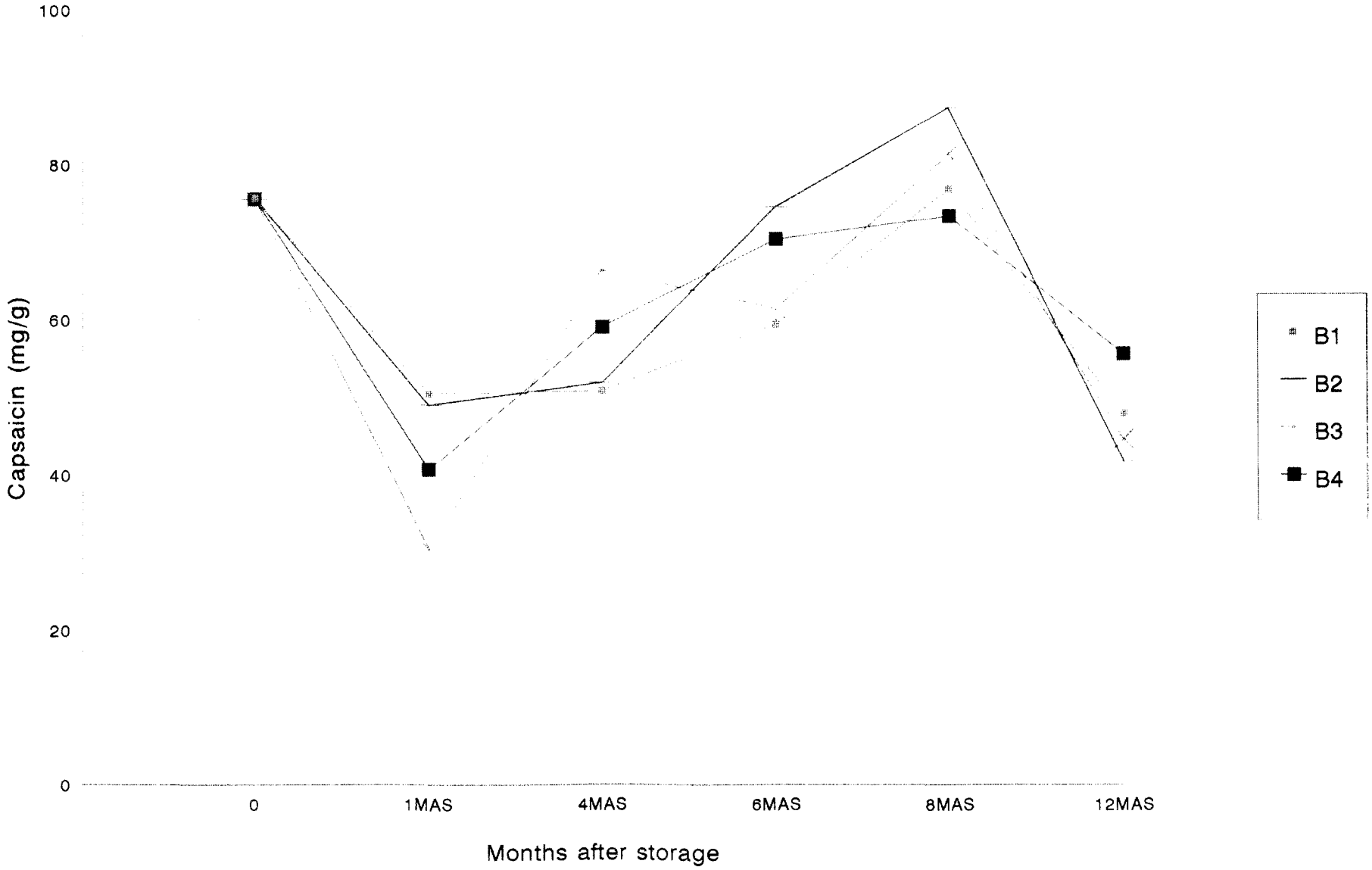
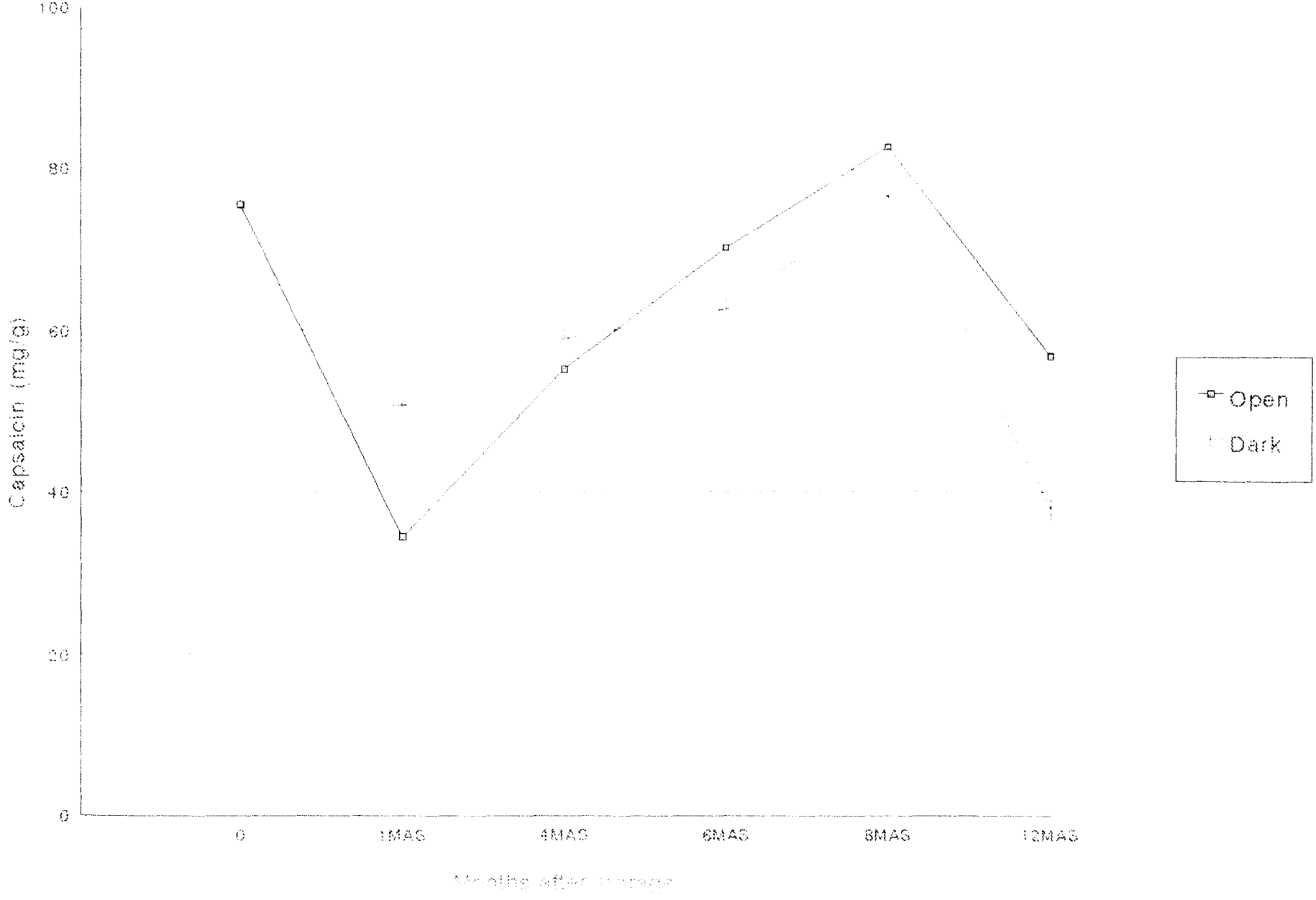


Fig.8 Changes in capsaicin content during storage



Discussion

DISCUSSION

India is one of the major chilli producing countries in the world. Chilli is an indispensable spice cum vegetable in every homestead. It is the only source of capsanthin and capsaicin, the colour and pungent principles respectively. Chilli oleoresin is fastly gaining momentum in the export market, replacing the dry chilli powder.

Oleoresin is prepared from dried chilli by extraction with a volatile solvent. The initial colour, pungency and their retention vary with extraction procedures. Standardisation of the extraction procedures with respect to solvents and number of siphoning would be helpful in recovering maximum oleoresin content from the produce.

The yield and quality of oleoresin vary with species, cultivars, seasons and stages of harvest maturity. The quality attributes of oleoresin viz. pungency and colour may change or deteriorate with type of containers as well as the storage conditions.

Identification of the species/cultivars with maximum oleoresin content and evolving a suitable method of storage would be an important contribution to the oleoresin industry and export. Under the circumstances, the present investigations were carried out with the objective of standardising the oleoresin extraction procedures, identifying *capsicum* species/cultivars for maximum yield of oleoresin with respect to season and harvest maturity stage, studying

the influence of harvest maturity on chilli quality and understanding the quality parameters of oleoresin as affected by storage.

5.1 Standardisation of extraction procedures for chilli oleoresin

Six solvents viz. acetone, ethyl alcohol, dichloro ethane, hexane, benzene and ethyl acetate were compared for the oleoresin extraction efficiency on the variety Ujwala in respect of oleoresin yield, siphoning number, colour value, pungency and solvent residue.

The solvents showed significant difference for all the characters studied, indicating variation in their efficiency of oleoresin extraction (Fig 9). The highest oleoresin yield (37.2%) was recorded by ethyl alcohol. Though the extraction efficiency of ethyl alcohol was about 2.5 times more compared to other solvents, it was not preferred as it took maximum siphoning number and time, indicating the slow extraction capacity of the solvent. The extraction efficiency of the solvents (Table 23) indicated that ethyl alcohol could extract only very low levels of the colour value and capsaicin as reported by Mathew *et al.* (1971). The higher yield of oleoresin could be due to non-flavour substances like resins, gums etc. extracted by ethyl alcohol which is in agreement with the findings of Rajaraman *et al.* (1981).

Acetone and benzene were second in oleoresin yield. As benzene is a cancer suspect agent, it was not considered for standardisation. Acetone was

Table 23 Comparative efficiency of solvents for oleoresin extraction

Solvents	Colour value	β carotene (mg/g)	Capsanthin (g/kg)	Capsaicin (mg/g)	Scoville Heat Unit
Acetone	22404	1873.4	4.661	68.4	1026630
Ethyl alcohol	1091	91.3	1.468	31.1	466620
Dichloro ethane	4134	3459	1.247	67.69	1015260
Hexane	12407	1644.4	6.836	49.89	748560
Benzene	10178	851.0	2.424	58.32	874590
Ethyl acetate	16865	1410.7	8.184	75.79	11365580

Fig.9a EFFICIENCY OF SOLVENTS FOR OLEORESIN EXTRACTION - (Oleoresin yield %)

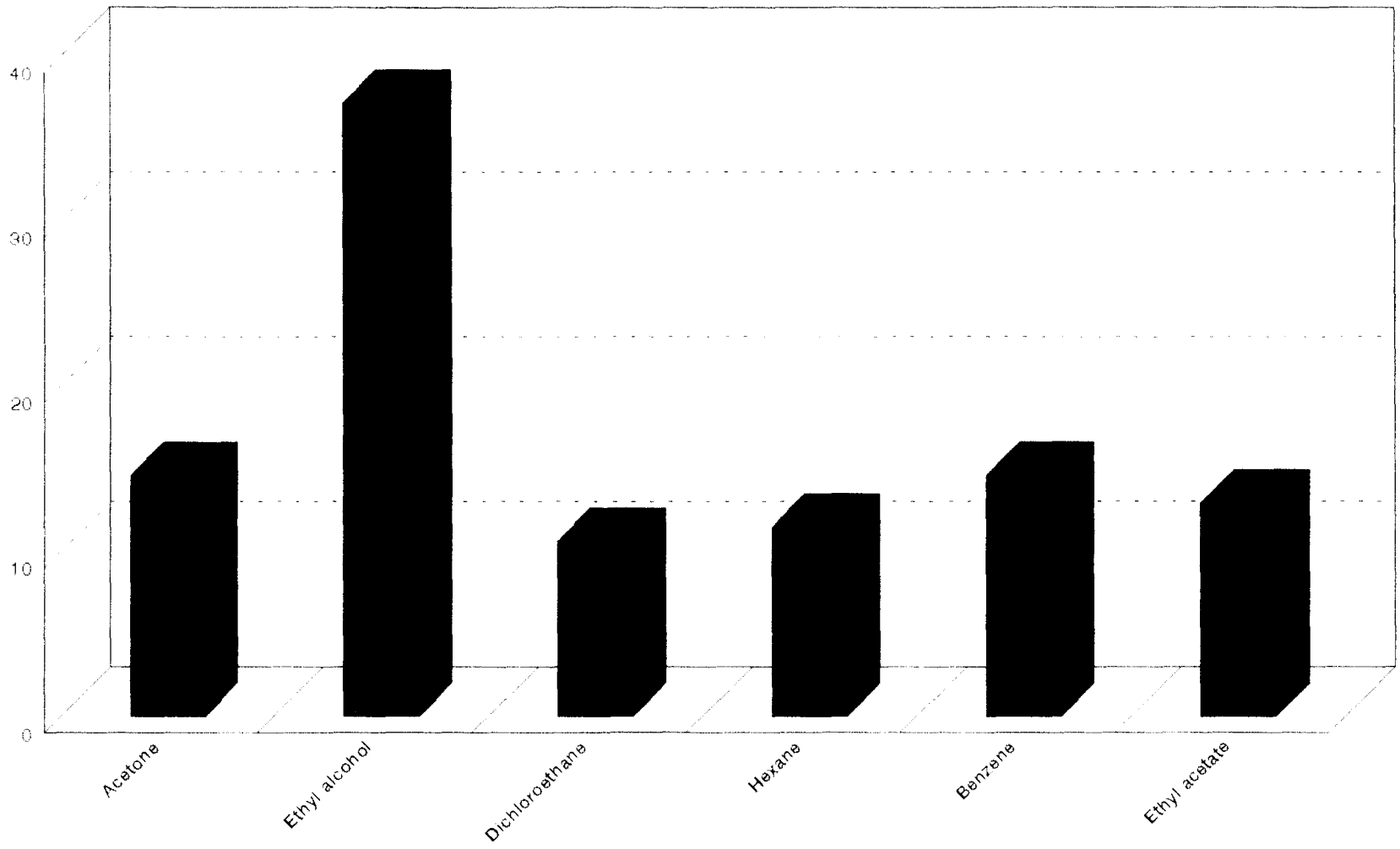


Fig.9b EFFICIENCY OF SOLVENTS FOR OLEORESIN EXTRACTION - (Capsanthin (g/kg)

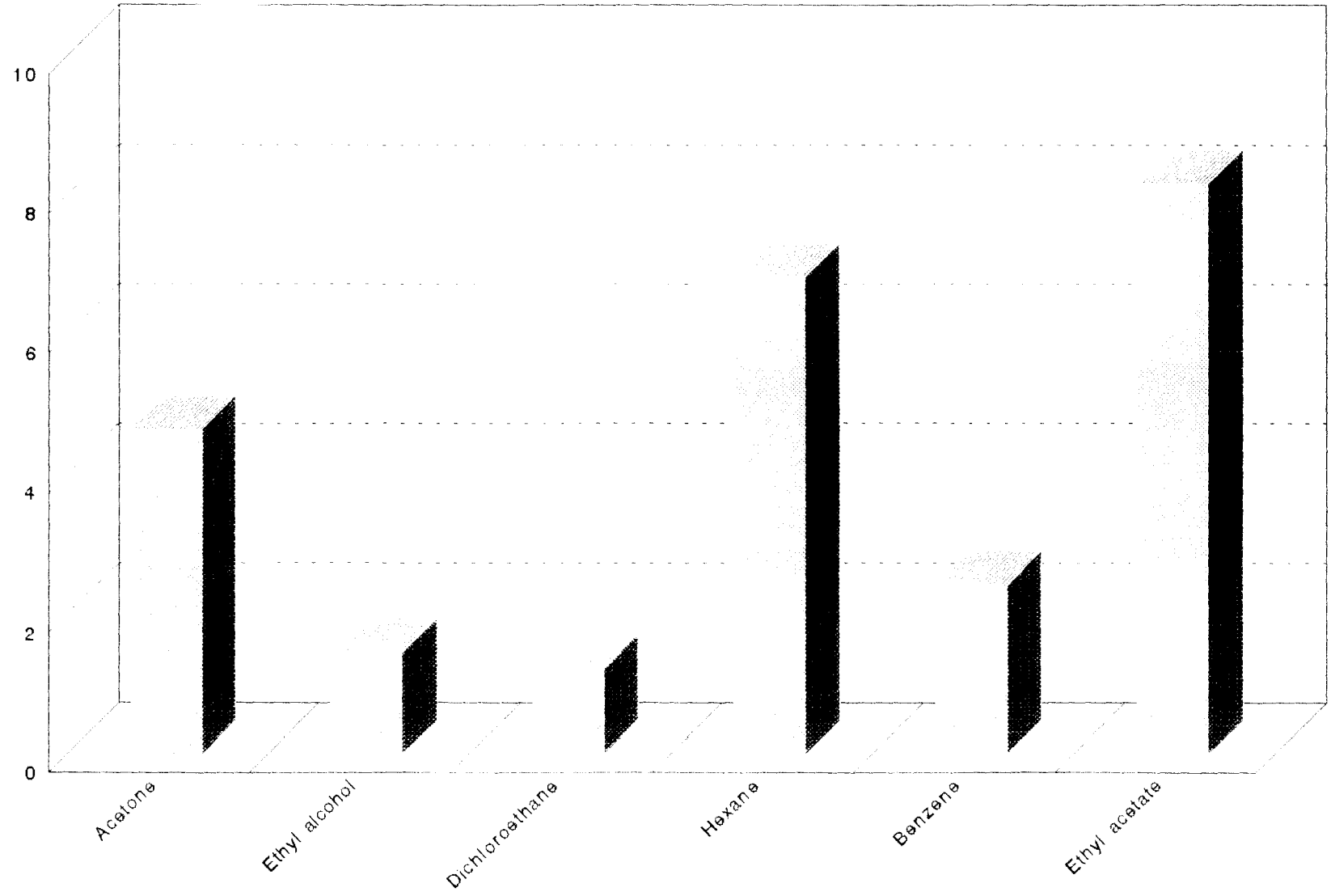
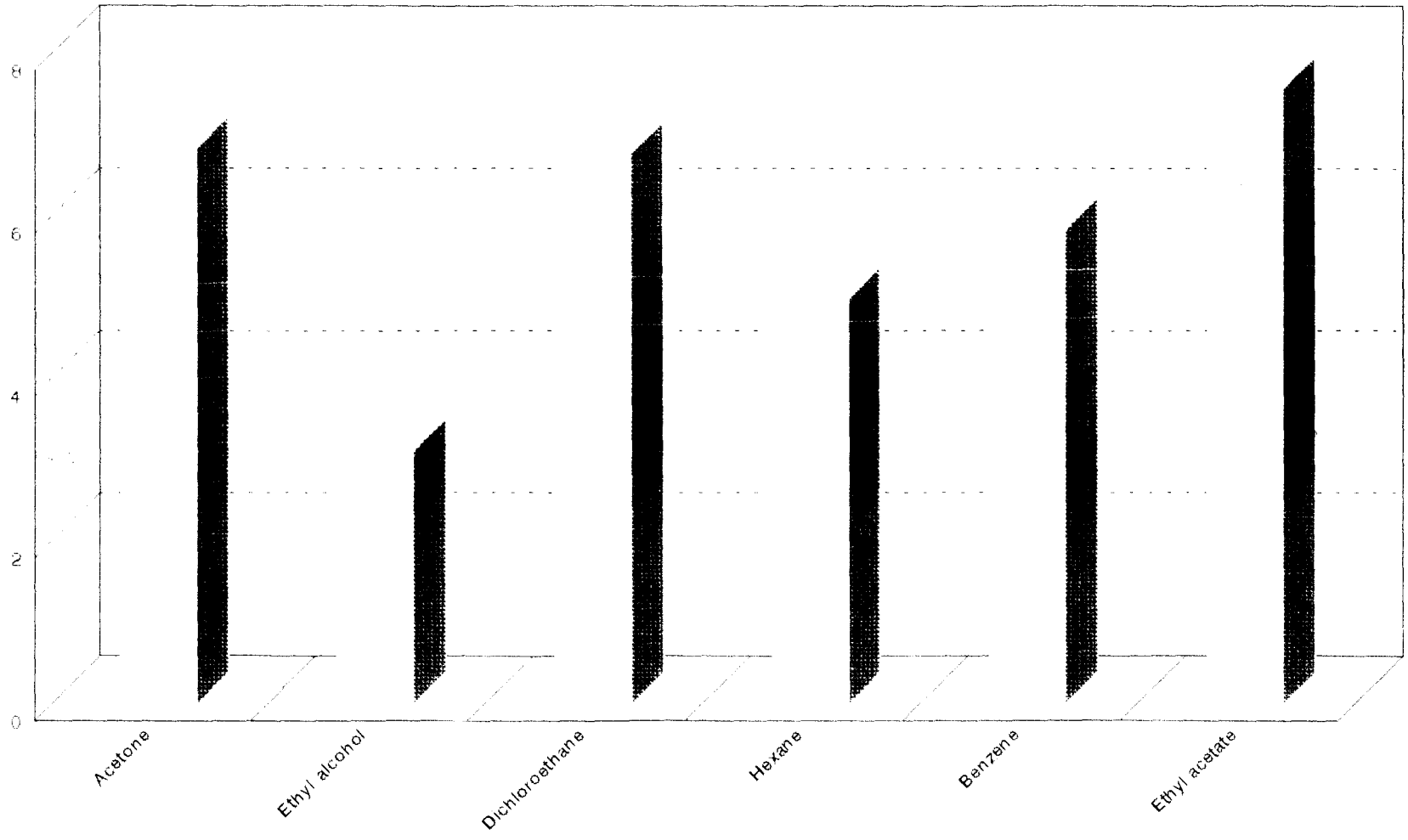


Fig.9c EFFICIENCY OF SOLVENTS FOR OLEORESIN EXTRACTION - (Capsaicin %)



first (22404 Nesslerimetric value) in colour and second in capsaicin extraction. Acetone could extract 68.4 mg/g of capsaicin, indicating the excellentefficiency of solvent for capsaicin. The findings of Szabo (1970) and Rajaraman *et al.* (1981) support the present study.

Ethyl acetate was on par with acetone and benzene in oleoresin yield and the extraction is faster compared to acetone. The colour extraction efficiency of ethyl acetate was very high (16865 colour value) which is second to acetone. In addition, ethyl acetate extracted the highest (75.79 mg/g) capsaicin also. According to Rajaraman *et al.* (1981), ethyl acetate has shown a superiority in extraction of pepper and ginger.

While the solvent residue limit for acetone is only 2 ppm, the limit permitted in ethyl acetate is as high as 250 ppm. Simple drying agents can purify the ethyl acetate unlike in the case of ethyl alcohol and acetone, where rectification is required. The odour characteristics of ethyl acetate are described as mild, fruity and agreeable. So the quality of the oleoresin will not be affected by the presence of traces of solvents and hydrolysed products like ethyl alcohol and acetic acid. Doubts are raised in recent years, about the use of chlorinated solvents like dichloroethane. Considering all these aspects,ethyl acetate was standardised as the best solvent for chilli oleoresin extraction.

5.2 Evaluation of capsicum species and cultivars for oleoresin recovery

Nine chilli genotypes were evaluated in three seasons viz. summer, rainy and winter and at three maturity stages. The correlation between oleoresin yield and its components, inter correlation among different characters and the direct and indirect contributions of these characters on oleoresin yield were also worked out.

The results revealed significant differences for all the characters studied. This observed variability is attributed to genetic as well as environmental influence. Significant season x genotype interaction for all the characters was observed.

Considering all the seasons, the variety 'Ujwala' was earliest in flowering and fruitset. Ujwala was on par with Arka Lohit, KTPL-19, CA 648 and CA 670 in fruitset. The varieties were generally late in winter. Though winter in Kerala is not as distinct as in other temperate regions, the days are comparatively longer and warmer during summer months. The carbohydrate accumulation will be generally more during longer days of the summer and less amount will be utilized in respiration during short nights. Consequently, more amount of carbohydrate will be available for growth and development under the warm humid conditions of Kerala, favouring the flowering and fruitset of tropical crops like chilli. The results reported by Balbaa *et al.* (1968) and Rylski (1972) provide evidence for the faster rate of growth and development in summer plants than in winter plants. With respect

to number of fruits also, Ujwala ranked first. In rainy and winter season, fruit yield was also high in Ujwala, though second in summer.

As far as oleoresin recovery is concerned, Arka Lohit was the first, which was only third in fruit yield. Considering yield and oleoresin content, Arka Lohit could be ranked first for oleoresin yield per unit area per plant. The total oleoresin yield in Ujwala showed third in ranking considering three seasons which is next to Arka Lohit and CA 645. Excluding summer, Ujwala is exceedingly superior in fruit and total oleoresin yield per plant. From several observations made in the Kerala Agricultural University, it was found that the performance of Ujwala is poor in extreme summer which could be attributed to its lower rank in three seasons in the present study.

With respect to yield/plant, genotypes were generally superior in summer. As far as oleoresin content is concerned, genotypes were high in winter. Considering yield and oleoresin together, summer is the best season for total oleoresin yield per plant. The poor oleoresin content in summer is compensated by the higher yield. In Opium poppy, Waller and Nowacki (1978) pointed out that the highest alkaloid level was found under climatic conditions of water stress.

Studies on the association between oleoresin yield and its components at genotypic level revealed significant correlation of number of fruits per plant with oleoresin yield. When the number of fruits increases, size is seen reduced. In small sized fruits, the percentage of constituents will be higher than that of

large sized fruits. This is true with many other chemical constituents in vegetable crops. In bittergourd, constituents like protein, fat, minerals, carbohydrate, calcium and iron are much higher in small sized fruits compared to large sized fruits (Gopalan *et al.* 1982).

The attributes of earliness had a negative correlation with oleoresin yield. This indicates that late varieties are rich sources of oleoresin. This means that late varieties are better for oleoresin constituents. The late variety may get more time for the accumulation of chemical constituents by metabolic inter conversion than the early varieties. The association between fruit yield and oleoresin recovery was negligible. The highest yielding genotype, CA 645 was the second from the last, supporting the present findings. This suggests that increase in yield need not result in an increase in oleoresin content.

Studies on path analysis revealed that days to flowering had maximum direct effect on yield of oleoresin followed by days to fruitset. The other two characters viz. days to harvest and fruits/plant had negative effect on yield of oleoresin.

Based on stability parameters like regression coefficient, b(i) and deviation from regression, Ujwala was the earliest to flower and to fruit and for fruits per plant and second in fruit yield per plant. The stable genotypes for fruit yield were Arka Lohit and Ujwala. For oleoresin recovery, Ujwala and CA 645 were the genotypes suitable for better management. For the last few years, Ujwala has been found to be a high yielding and responsive variety to

better inputs under Kerala condition. Arka Lohit is an improved variety recommended for better management from the IIHR, Bangalore.

Influence of three stages of harvest maturity in three seasons, on the fruit and oleoresin yield was also observed in the nine genotypes of chilli. Pooled analysis showed significant differences among the genotypes for all the characters.

Number of fruits per plant was influenced by stages of harvest maturity. The promising genotypes for number of fruits were Ujwala and Arka Lohit for harvesting at all the three stages of maturity. These two varieties were released based on their superiority in performance over the last many years from Kerala Agricultural University, Vellanikkara and Indian Institue of Horticultural Research, Bangalore, respectively.

The yield of fruits per plant was also affected by stages of harvest maturity. Yield was maximum at full ripe (M2) which is on par with turning stage (M1) and was the lowest at withering stage (M3). The fruit attains its maximum size and weight at full ripe stage and thereafter shows a decline due to shrinkage and consequent weight loss.

Oleoresin recovery was not affected by stages of maturity. However, the interaction between stages of maturity and season was significant. Till turning stage, metabolic pathway was similar to that of any other plants. Because of the increasing trend of carbohydrate and other organic compound

accumulation, the formation of oleoresin might have continued during the full ripe and withering stage.

5.3 Influence of harvest maturity on quality of chilli

When the genotypes were analysed, they exhibited highest colour value at withering stage, moderate at full ripe and lowest at turning stage. The result of Lease and Lease (1956) and Benedek (1972) supports the present study. The darker and more flaming red chilli is considered as higher grade, which fetches higher price. The deepening of chilli colour at withering stage was ascribed to drying, which is accomplished by the subsequent formation of colouring matter. The pigment content increases in relation to the increase in dry matter as reported by Benedek (1972).

The present finding that chilli fruits were least pungent at turning stage, moderate at full ripe and highest at withering stage, was in agreement with Balbaa *et al.* (1968) and NisarAhmed *et al* . (1987) who reported that the capsaicinoid content increased with fruit maturation in relation to increase in dry matter content.

On analysing the cultivars for total sugar content at different maturity, it was seen that the chilli fruits harvested at withering stage was low in sugar content (11.03%) compared to fruits harvested at turning or full ripe stage. The decrease of the sugar content observed during the withering period can be interpreted by the decomposition of sugars into organic acids, as a part of

which gets utilised in respiration and other part serves on a basic material for cartenoid synthesis. The experiment also conforms the theory that the carotenoid synthesis occurs at the charge of sugar content, increasing the colour with advancement of maturity. The findings of Benedek (1972) that the sugar content decreases during after-ripening period confirms the present result.

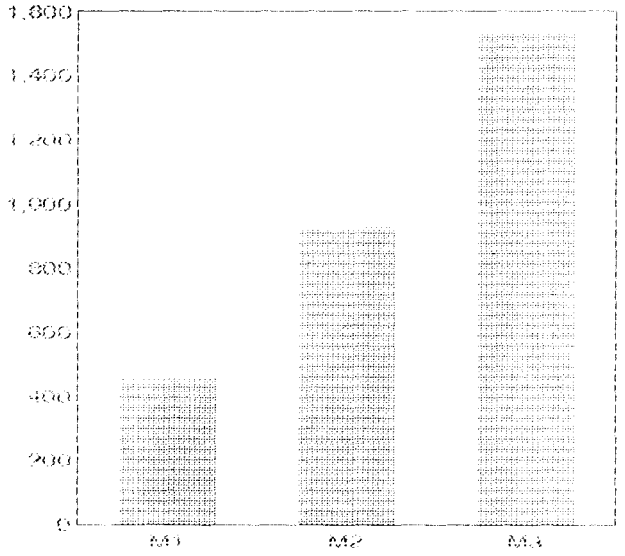
Pungency increases moderately with decrease of sugar, where as the colour increase is rapid. Sugar is degraded to organic acids and finally pigment formation is more during withering stage. This degradation and interconversion of carbohydrate in chillies are independent of plant growth and development.

Among the genotypes evaluated for colour, KTPL-19 had the highest value (1809.61). This genotype is a paprika type developed at IARI Regional Station, Katrain, which is generally high in colour value and low in pungency. This is in agreement with the findings of Joshi *et al.* (1993). However, under Vellanikara condition, this variety was moderately pungent (17.31 mg/g capsaicin). The percentage of capsaicin in fruits depends on climatic conditions, which could be reason for pungent behaviours under the warm humid conditions of Kerala. In addition, fruits in summer are also reported to possess higher pungency according to Balbaa *et al.* (1968).

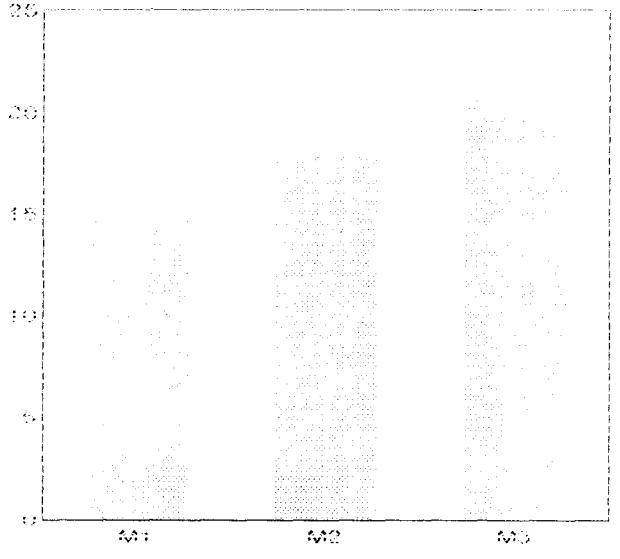
In the present study, genotypes under *C. annuum* were high and those under *C. chinense* were low in colour value. The findings of Indira *et al.* (1994) revealed a similar result. However, the results of Pradeepkumar (1990)

Harvest Maturity On Quality Of Chilli

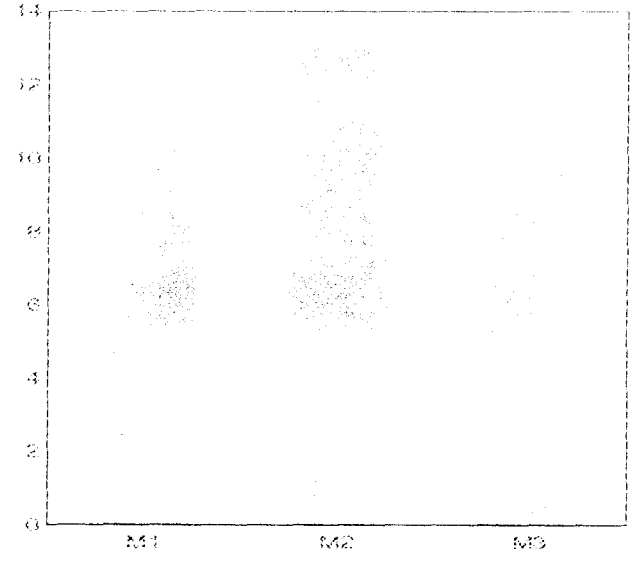
Colour Value



Pungency (mg/g)



Total Sugar %



Comparison of different containers in retaining colour value showed no definite pattern. But significant difference was noticed among samples, stored in different containers. Van Blaricom and Martin (1945) and Chen and Gujmanis (1968) could also find that the type of container did not affect colour loss. PET jar was comparatively better in retaining a higher colour value compared to other bottles.

Oleoresin stored in dark was higher in colour value compared to samples kept in open condition. Sunlight exhibited pronounced effect in bleaching the colour and brings about discolouration of red pigments. The reports of Krishnamurthy and Natarajan (1973) supports the present findings. It can be concluded that for better storage, oleoresin samples should be kept in dark. If one is interested in colour value alone, storage in any containers is not recommended.

Analysis of samples at different periods in varying containers showed that the capsaicin content of oleoresin samples was reduced to about 45 to 67% within one month. After that a gradual increase was noticed in capsaicin content. Eventhough there was no such report available in chillies, Lorenz (1951) could give a result which is very close to our observation. He found that there was considerable conversion of starch to sugar in summer squash during the first few days of storage. As in the case of colour value, containers did not influence the capsaicin content in any definite manner. But polythene bottles (B2) were superior in preserving a higher pungency value, followed by

PET jar, after eight months of storage. After that, the capsaicin content of the oleoresin samples kept in all the containers started decreasing. At twelve months after storage the polythene jar was the poorest in retaining the capsaicin content, which was considered as comparatively best at eight month after storage. The reduction in capsacin content of oleoresin samples kept for storage starts after eight months but the details regarding the exact period at which the reduction starts need further investigaton.

Reduction in capsaicin content of oleoresin was higher in open condition, irrespective of bottles upto one month. Capsaicin content was higher in dark compared to open at fourth month, it equalled at 4 1/2 months and afterwards it exceeded the capsaicin content in dark (Fig. 8). The remarkable effect of light on capsaicinoid synthesis could be due to the activation of enzyme systems involved in biosynthesis of capsaicinoid. This is in confirmity with the results of Govindarajan (1985).

At eight months after storage, the capsaicin content in oleoresin has exceeded the initial pungency value at the time of storage. It is clear that the chilli oleoresin stimulating high pungency are well preserved during storage due to lower fat content. The reports of Charazka *et al.* (1981) supports the present findings. So for preserving the pungency alone, storage of oleoresin in polythene containers under open condition is recommended, till eight months.

Summary

SUMMARY

The present investigation "Oleoresin recovery, quality characterisation and storage stability in chilli (*Capsicum spp.*) genotypes" was conducted at the Department of Olericulture, College of Horticulture, Vellanikkara during 1993-1996. The objectives were to standardise the chilli oleoresin extraction procedures using different solvents; to evaluate *capsicum* species and cultivars for yield of oleoresin with respect to season and stages of harvest maturity; to study the effect of harvest maturity on chilli quality and to understand the quality parameters of oleoresin as affected by storage.

Chilli oleoresin was extracted from the variety 'Ujwala' by soxhlet extraction methods using six different solvents and the procedure was standardised, based on the efficiency of solvents.

Nine chilli genotypes were evaluated in three seasons viz. summer, rainy and winter and at three different harvest maturity. Evaluation was done for earliness, yield of fruits and oleoresin.

The nine chilli genotypes grown during summer season were harvested at three maturity stages and evaluated for quality parameters like colour, pungency and total sugar content.

The oleoresin extracted from the variety 'Ujwala' using the standardised solvent was subjected to a storage study under laboratory conditions and changes during storage was recorded for an year.

The results of the present investigation are summarised as below:

1. Among the solvents tried viz. acetone, ethyl alcohol, dichloro ethane, benzene, hexane and ethyl acetate, ethyl acetate was the fastest in chilli oleoresin extraction which was standardised as the best solvent for chilli oleoresin extraction.
2. Though highest oleoresin yield was extracted by ethyl alcohol, it took maximum siphoning time and number indicating its low extraction efficiency.
3. Acetone had the maximum colour extraction efficiency (22404 Nesslerimetric colour value) while ethyl alcohol had the minimum (1091).
4. Ethyl acetate was most efficient for capsaicin extraction (75.79 mg/g)
5. Thin layer chromatographic separation of capsanthin and capsaicin showed maximum number of spots in oleoresin samples extracted using ethyl acetate.
6. Solvent residues calculated using Gas Liquid Chromatography showed the lowest concentration in samples extracted using ethyl alcohol (0.0309) followed by ethyl acetate (0.0516).
7. Among the nine genotypes, Ujwala was the earliest to flower and fruitset and CA 645 was the late.

8. Seasonal influence on maturity showed that the genotypes were generally late in winter.
9. Ujwala was the best genotype for fruits per plant, followed by Arka Lohit. CA 670, a *C. baccatum* type had the lowest fruits per plant.
10. Chilli genotypes produced maximum fruits during summer season and minimum during winter.
11. CA 645 had maximum fruit yield per plant followed by Ujwala. Lowest fruit yield was observed in CA 648, irrespective of seasons.
12. Chilli genotypes gave maximum yield during summer and minimum during rainy seasons.
13. Considering the fruit yield and oleoresin content, Arka Lohit had maximum oleoresin yield per unit area per plant.
14. Ujwala was superior in fruit and total oleoresin yield per plant except in summer.
15. Genotypes were high in oleoresin content during winter. But considering the fruit yield and oleoresin content together, summer is the best season for total oleoresin yield per plant.
16. Maximum number of fruits per plant was observed in Arka Lohit during summer, when harvesting was done at full ripe stage, which is on par with the other two stages of harvest of the same variety and also with Ujwala harvested at full ripe stage in the same season.

17. Plant yield was maximum when harvested at full ripe stage, moderate at withering stage and least at turning stage.
18. Interaction between season x maturity was significant for fruit yield per plant. Highest fruit yield was observed in summer season when fruits were harvested at turning and full ripe stage.
19. Considering the genotypes in relation to season and harvest maturity, fruit yield was maximum (162.7g) in CA 645 during summer, when harvested at full ripe stage.
20. Cultivars belonging to *C. annuum* had high oleoresin yield when harvested at turning stage.
21. At full ripe stage, Arka Lohit and KTPL-19 had highest oleoresin recovery.
22. Arka Lohit, CA 670, CA 645 and Ujwala were having high oleoresin recovery, when harvested at withering stage.
23. Cultivars had high oleoresin content during summer, when fruits were harvested at turning stage (26.4%). When harvesting was delayed to full ripe or withering stage, highest oleoresin recovery ((26.5% and 26.2%) were noticed during winter season.
24. During summer and winter seasons, genotypes were on par in oleoresin yield when harvest maturity was fixed at full ripe or withering stage. But in rainy season, oleoresin recovery was high in fruits harvested at withering stage.
25. The attributes of earliness had a negative correlation with oleoresin yield indicating that late varieties are rich sources of oleoresin.
26. Increase in yield did not result in an increase in oleoresin content.

27. Stability analysis revealed Ujwala and CA 645 as the best genotypes for oleoresin recovery under better management. The stable genotypes for fruit yield were Arka Lohit and Ujwala.
28. Maturiy studies showed that the colour and pungency increased with fruit maturity.
29. Chilli fruits harvested at withering stage was low in sugar content compared to fruits harvested at turning or full ripe stage.
30. KTPL-19, a paprika type had the highest colour value. Genotypes under *C. annuum* were high and those under *C. chinense* were low in colour value.
31. The capsaicin content of the genotypes varied from 1.1 to 2.2 % indicating its suitability for oleoresin required for Pharamaceutical industry.
32. The genotypes under *C. chinense* such as CA 640 and CA 645 were highest in pungency
33. The variety Ujwala was moderate for colour and pungency values.
34. The less pungent genotypes are characterised by high sugar content indicating the inverse relationship between sugar content and capsaicin
35. The storage studies revealed a drastic reduction in colour value in oleoresin samples within one month of storage, but the rate of reduction was decreased after one month.
36. Studies on the containers showed that the types of container did not influence the colour loss.

37. Studies on storage condition showed that the oleoresin stored in dark was higher in colour value compared to samples kept in open.
38. Capsaicin content was reduced to 33% to 60% within one month, after which it gradually increased till eight months, again decreased at twelve months of storage.
39. Containers did not influence the capsaicin content in any definite manner.
40. Influence of light on capsaicin content showed that it was higher in dark than in open at fourth month and equal at 4 ½ months and it exceeded in light, indicating the remarkable effect of light on capsaicinoid synthesis.
41. Delaying the harvest to withering stage and storing the oleoresin in polythene containers for eight months under open condition can be recommended for increased pungency.
42. Storage of oleoresin is not advisable for preservation of colour value.

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Appendices

APPENDIX - 1

Preparation of Thin Layer Chromatography (TLC) Plates

* Mix 30 g silica gel containing calcium sulphate as binder.
* Mix with 60 ml distilled water.
* Pour the slurry into a TLC spreader adjusted to a thickness of 150 μm and spread over 5 to 6 glass plates of 20 x 20 cm.
* Air dry the plates for 4 to 5 hours
* Activate by drying in an oven at 100 to 105°C for 30 minutes and store in a desiccator.

OLEORESIN RECOVERY, QUALITY CHARACTERIZATION AND STORAGE STABILITY IN CHILLI (*Capsicum* spp.) GENOTYPES

By

MINI C.

ABSTRACT OF A THESIS

Submitted in partial fulfilment of the requirement
for the degree of

Doctor of Philosophy in Horticulture

Faculty of Agriculture
Kerala Agricultural University

DEPARTMENT OF OLERICULTURE
COLLEGE OF HORTICULTURE
Vellanikkara, Thrissur

1997

ABSTRACT

An investigation on "oleoresin recovery, quality characterisation and storage stability in chilli (*Capsicum* spp.) genotypes" was undertaken in the department of Olericulture, College of Horticulture, Vellanikkara during 1993-96 with the objective of standardising the solvent for chilli oleoresin extraction, identifying *capsicum* species or cultivars for maximum oleoresin yield with respect to season and stage of harvest and understanding the quality parameters of oleoresin as affected by storage.

Among the six solvents tried viz. acetone, ethyl alcohol, dichloro ethane, hexane, benzene and ethyl acetate, ethyl acetate was standardised as the best solvent in respect of time and efficiency of extraction.

Evaluation of nine genotypes for oleoresin under three different seasons at three stages of harvest maturity identified Arka Lohit as the highest yielder of oleoresin. Genotypes were higher in oleoresin content during winter. Considering the fruit yield and oleoresin recovery together, summer was the best season for total oleoresin yield. The genotypes were on par in oleoresin yield in summer and winter when fruits were harvested at full ripe or withering stage. During rainy season, oleoresin content was maximum at withering stage.

The colour and pungency increased with fruit maturity. The capsaicin content of the genotypes varied from 1.1 % to 2.2 % indicating its suitability for oleoresin required for pharmaceutical industry. Colour value was highest in the paprika type, KTPL-19.

The storage studies of oleoresin revealed that the type of container did not influence the colour loss. The oleoresin stored in dark was higher in colour value compared to samples kept in open. For capsaicin, open condition of storage was better than dark storage. Delaying the harvest to withering stage and storing the oleoresin in polythene containers for eight months under open conditions can be recommended for increased pungency. Storage is not advisable for colour retention.