

Comparison of the Efficacy of Micro and Nano Emulsions of Caraway Essential Oil on Quality of Red Chili Fruits(*Capsicum annuum*L.) during Cold Storage

T. F. A. El-Moghazy

Medicinal and Aromatic Plants Res. Dept., Hort. Res. Instit., Agric. Res. Center, Giza, Egypt

E-mail: drtamereimo051@gmail.com

Abstract- The essential oils (EOs) are good substitutes to antimicrobial during postharvest and can also maintain the sensory acceptability when applied to fresh fruits, the micro and nanoemulsions (ME and NE) are the most suitable and studied thanks to the ease of handling, cheap and safe. The aim of this study was to investigate the effect of ME and NE of Caraway EO on the quality parameters of red chili fruits(*Capsicum annuum*L.) under cold storage in 2019 and 2020 seasons. The fruits were immersed in water(control) unpackaged, polypropylene package (PP), ME at (0.05 and 0.1%) and NE at (0.05 and 0.1%) of caraway oil in PP. After immersing for 2 min in treatments, fruits were air dried for half hour at room temperature, then stored for 32 days at 7° C and 90–95% R.H. and assessed 8 days to determine the changes in fruit quality characteristics during storage. The weight loss %, decay %, fruit firmness, total soluble solids, anthocyanin content, total acidity %, and capsaicin content of red chili fruits during storage were investigated. Results showed that, all of tested ME and NE of EO application significantly decreased weight loss, decay percentage and increased fruits storage period. Moreover, ME and NE of Caraway EO, positively, affected postharvest quality properties including fruit firmness, total soluble solids, titratable acidity, anthocyanin content and capsaicin content. It was observed that NE of EO at 0.1 and 0.05% from Caraway gave the best efficacy on weight loss, decay, firmness, total soluble solids, titratable acidity, anthocyanin content and capsaicin content than ME. Generally, NE at 0.1% PP, NE at 0.05% PP and ME at 0.1% PP treatments were the best among all treatments during the storage period.

Keywords: Red Chili Fruits (*Capsicum annuum* L.), Essential Oils, Micro and Nanoemulsions, Caraway, Fruit Quality, Packing and Cold Storage, volatile oils content and composition

INTRODUCTION

Green and Red-Chilies (*Capsicum annuum*L.) one of the plants that belong to the family Solanaceae, the cultivated area is estimated at about 2,570 fed. in Egypt (BAS, 2018/2019). The genus *Capsicum* has recognizable pharmacological properties due to the presence of alkaloids collectively known as capsaicinoids (Reyes-Escogido, *et al.*, 2011). The heat sensation is incited by the amount and the type of a group of capsaicinoids; the alkaloids found only in chilli pepper pods (Popelka, *et al.*, 2017). The high capsicum content makes it unique to use in fresh, dried and powdered forms to add taste, flavor and colour in pickles, curries, chutneys and other food preparations (Amruthraj, *et al.*, 2014). Postharvest losses in developing countries can range from 15 to 50% according to FAO, (2012). The pepper is a

perishable fruit with a short shelf-life (market life period) and, therefore, is highly susceptible to a rapid decrease in quality. Thus, peppers require a modified postharvest environment to reduce the nutritional loss (Tan, *et al.*, 2012). Use of packages like plastic trays, and polypropylene boxes also help in extending the shelf life of the fresh produce (Amjad, *et al.*, 2010). Packaging is required not only for food preservation, but also for protection during handling and safe transportation of products. The export market needs better influence of the packaging trends in food, increasingly (Farooqi, 1994). Regarding storage temperature, Lim, *et al.* (2008) who reported that *Capsicum* genus is susceptible to chilling injury at temperatures below 7 °C and less decay at 10 °C.

Essential oils (EOs) extracted from plants are good substitutes to fungicides during postharvest; EOs can also maintain the sensory acceptability when applied to fresh fruits and vegetables. Unfortunately, not many attempts have been explored to use EOs in reducing postharvest losses (Ding and Lee, 2019). Further, their widespread use are still restricted due to their poor physico-chemical properties; i.e., high volatility, low water solubility, thermal decomposition and stability issues. Currently, the most suitable approach to overcome such limitations is based on the development of proper formulation strategies. One of the approaches suggested to achieve this goal is the encapsulation process through the preparation of aqueous nano-dispersions. Among them, micro- and nanoemulsions (ME and NE) are the most studied thanks to the ease of formulation, handling and to their manufacturing costs (Pavoni, *et al.*, 2020). ME and NE are suitable in presence of lipophilic or low water soluble compounds, such as EOs, that required the dispersion in water media, i.e., pesticides or foodstuff ingredients (Khater, *et al.*, 2017 and Donsi, and Ferrari, 2016). Nanotechnology includes all submicron-size systems less than 1000 nm., preferably those measuring 100–500 nm (Mora-Huertas, *et al.*, 2010). Abd El-Gawad and El-Moghazy (2021), found that the major constituents of the EO (Caraway) were limonene (35.0 %) and carvone (57.0 %). Also by Khalil *et al.*, (2018); they found that Limonene and carvone were the major identified compounds in Caraway oil. The EO of Caraway has stronger antifungal and antibacterial effect than citronella oil (Simic *et al.*, 2008). The anti-aflatoxic, antioxidant, and antimicrobial effects of Caraway oil along with its reputation as spice help the industries to use it as a natural preservative and antioxidant agent (Mahboubi, 2019). Plant EOs incorporated in nanoemulsions seem to penetrate faster in the microbial membranes due to the increased area per weight unit. This would allow decreasing the concentration to achieve an equivalent or even greater microbial effect over conventional emulsions (Odriozola-Serrano, *et al.*, 2014). The polydispersity index (PDI) value below 0.2 indicates a narrow size distribution and thus provides long term stability to the formulated nanoemulsion (Sampathi, *et al.*, 2015).

Found that, all of tested NE of EOs (peppermint and caraway) application significantly decreased weight loss %, decay % and increased fruits storage period (Abd El-Gawad and El-Moghazy 2021). The weight loss percentage (WL %) of (*Capsicum chinense* Jacq.) fruits significantly ($P \leq 0.05$) increases with the storage time and fruit firmness continuously decreased during the storage period (Malakar, *et al.*, 2020). WL% and decay % increased, while fruit firmness decreased with increasing storage periods of sweet bell pepper (Tsegay, *et al.*, 2013). The high WL% in completely ripened fruits could be due to changes in the

permeability (cell membranes), making them more sensitive to the loss of water as confirmed by Antonialiet *al.*, (2007). Firmness changes because of lower metabolic activities at lower temperatures and also water loss in chilies might be a major factor affecting the firmness of chilies, and this was supported by an increasing trend in the WL% throughout the storage time (Lim, *et al* 2008). The decay could be due to the multiplication of microorganisms and the rapid growth of mold which significantly influenced by different storage conditions (Malakar, *et al.*, 2020). Regarding the capsaicin content was found increased up to 5 days of storage; thereafter, it began moderately decreasing during the storage period on King Chili (*Capsicum chinense* Jacq., Malakar, *et al.*, 2020). The decline in capsaicin content occurred due to the pigment of the color break as a result of inhibition of the capsaicin synthesis, degradation by peroxidase and chemical disintegration caused by photo-oxidation (Reyes-Escogido *et al.* 2011). The increment in total soluble solids (TSS) for stored fruits was probably due to increase of respiration and metabolic activity (Tsegay, *et al.*, 2013). And also, Ali *et al.* (2011) found that the higher respiration rate increases the synthesis and use of metabolites result in higher TSS due to the higher change from carbohydrates to sugars. Barbosa, *et al.*, (2020), found that although some small changes were observed during the storage of red peppers in Anthocyanin Content, these changes seemed to be random, with no apparent trend. Small differences are graphically observed for samples with 10 and 14 days of storage, respectively.

Therefore, the main target of the present work was to evaluate the efficacy of ME and NE of caraway EO as natural and environmentally friendly alternatives on improving quality and extending shelf life of red chili fruits (*Capsicum annuum* L.) fruits under cold storage and polypropylene.

MATERIALS AND METHODS

Plant material and experimental design:

Red-chili fruits (*Capsicum annuum* L.) were obtained from a private orchard at El-Mahmoudeya region, El-Behira governorate, Egypt. Fruits were harvested at optimal time in the first week of January in the two seasons (2019 and 2020) in the full color stage. The fruits were delivered on the same day to the laboratory, fruits homogenous in color, size and absence of defects. Non-defective fruits were selected, washed with fresh water, air dried, and used in the postharvest treatments. Dried Caraway fruits (*Carum carvi* L., Apiaceae) were obtained from a private farm, Kom Hamada, Al Buhayrah, Egypt.

Red chili fruits (*Capsicum annuum* L.) were randomly selected and divided into six equal groups; four groups were immersed in caraway oil at (0.05 and 0.1 %) in both microemulsion (ME) and nanoemulsion (NE) in polypropylene (PP) packages as well as two groups water; control (unpacked) and PP package, all solutions containing Tween-80 0.05% (v/v). After immersing for 2 min in treatments, fruits were air dried for half hour at room temperature. All fruits samples (250 g) and stored for 32 days at 7°C and the relative humidity (RH) of 90-95%. The complete randomized design was applied (6 treatments x 3 replicates x 5 period storage i.e. 0, 8, 16, 24, 32 days). The treatments were:

T1: Fruits, unpacked (**Control**).

T2: Fruits, polypropylene package (**PP**).

T3: Fruits immersed with nanoemulsions oil at 0.05% in PP package (**NE at 0.05% PP**).

T4: Fruits immersed with nanoemulsions oil at 0.1% in PP package (**NE at 0.1% PP**).

T5: Fruits immersed with microemulsions oil at 0.05% in PP package (**ME at 0.05% PP**).

T6: Fruits immersed with microemulsions oil at 0.1% in PP package (**ME at 0.1% PP**).

Extraction of essential oil (EO):

Dried caraway fruits were subjected to hydro distillation for three-hours using a Clevenger type apparatus as described by Egyptian Pharmacopoeia (2005). The obtained EO were dried over anhydrous sodium sulphate to remove traces of moisture and kept in sterilized dark bottles a refrigerator in at 4°C until use.

Chemical composition of essential oil (EO):

The GC analysis of the EO samples was carried out using DsChrom 6200 Gas Chromatograph equipped with a flame ionization detector, Column: BPX-5, 5% phenyl (equiv.) polysilphenylene-siloxane 30m x 0.25mm ID X 0.25 um film., Sample size: 1ul, Temperature program ramp increase with a rate of 1°C/min from 70 to 200°C, Detector temperature (FID): 28°C, Carrier gas: nitrogen, flow rate: N2 30ml/min; H2 30 ml/min; air 300 ml/min. Main compounds of the EO was identified by matching their retention times with those of the authentic samples injected under the same conditions. The relative % of each compound was calculated from the area of the peak corresponding to each compound.

Preparation of essential oil (EO) micro and nanoemulsions (ME and NE) and droplet size Analysis:

Nanoemulsion (NE) was prepared by adding 5 ml of oil and 3 ml of Tween 80 gentle stirring until homogeneous mixture formed then complete the volume with water to 100 ml, to help distribute and completely incorporate the essential oils and then stirred using a magnetic stirrer for 30 min. The mixture was sonicated using an Ultrasonicator (Bande-lin SONOPULS HD 2200, Germany) for 30 min. at 75 W. Microemulsion (ME) of EO was prepared as mentioned above without sonication. **Measurement of droplet size (nm)** of NE was performed by a dynamic light scattering analyses using Zeta Nano ZS (Malvern Instruments, UK) at room temperature. Prior to measurement, 30 µl of each NE was diluted with 3ml of water at 25 °C. Particle size data were expressed as the mean of the (Z-average) of 3 independent batches of the NE (Abd-Elsalam and Khokhlov 2015). The droplet size and the polydispersity index (PDI) of the formulated NE were measured (**Table A**). This work was performed by Central Laboratory, Faculty of Pharmacy, Alexandria University, Egypt.

Table A. Mean droplet size and PDI of investigated caraway essential oil NE.

Essential oil nanoemulsion	Mean droplet size (nm)	Polydispersity index (PDI)
Caraway oil	113.2	0.117

Determination of physical and chemical properties:

Weight loss (%):

The difference between the initial weight of the fruits and that recorded at the date of sampling was translated as weight loss percentage and calculated as follows:

$$\text{Weight loss \%} = \frac{\text{Average loss in fruit weight}}{\text{Average fruit weight at the beginning of storage}} \times 100$$

Decay (%):

The percentage of disordered fruits included all of the spoiled fruits resulted from rots, fungus, bacterial and pathogens were assessed and the defects were calculated as follows:

$$\text{Decay \%} = \frac{\text{No. of fruit decay}}{\text{No. of fruit at the beginning of storage}} \times 100$$

Fruit firmness (N):

It was measured on red-chili fruits (*Capsicum annumL.*) samples by pressure tester (N).

Total soluble solids (%):

A hand refractometer was used to determine the total soluble solids percentage in fruit juice.

Anthocyanin content (mg/100 g. F.W.):

Anthocyanin content was determined in fruits according to the method described by Yilids and Diken (1990).

Total acidity (%):

Titrateable acidity was determined by titrating 5 g of homogenized pulp in 100 (ml) of distilled water against 0.1 (N)NaOH, using phenolphthalein, expressed as a citric acid %, as described by Adolfo Lutz Institute (Brazil, 2008).

Capsaicin content (Bajaj, 1980)mg/ml:

Capsaicin was quantitatively analyzed in chili fruits by colorimetric. It was extracted from 0.5g of the material to be analyzed in 5 ml of ethyl acetate. After 24 hours, extract was filtered through what man no 1 paper to remove suspend impurities and 1 ml of sodium nitrite-sodium molybdate reagent (0.5 M sodium nitrite and 0.025 M sodium molybdate in aqueous solution) was added. The components are well mixed. After 15 min, 2 ml 1N NaOH was added. Absorbance was read at 430 nm in spectrophotometer (Bausch and Lomb) against reagent blank within 30 min of color development. Blank was prepared by replacing chromogenic reagent with distilled water. Standard curve was prepared by using capsaicin in the concentration range of 20-100 ug/ml according to Khetrapal, *et al.*, 2015 and Liljana *et al.*, 2013.

Statistical analysis:

Data of the present study were subjected to the analysis of variance test (ANOVA) as Randomized Complete Design (RCD). Where the first factor was for six treatments mentioned before, the second factor was for storage period. The least significant differences

(LSD) at the 5% level of probability were calculated using a computer program SAS according to Gomez and Gomez (1984).

RESULTS AND DISCUSSION

Chemical composition of EO:

The components of Caraway EO were determined by GC, Fig. (1) the main constituents of caraway EO were limonene 37.02 % and carvone 55.5%. These results agree with those mentioned by Abd El-Gawad and El-Moghazy (2021) and Khalil *et al.*, (2018); they found that Limonene and carvone were the major identified compounds in caraway oil. According to European Pharmacopeia, caraway fruits should contain 3% EO with D-carvone (50–65%) and (+)-limonene (up to 45%). D-carvone, the main component of caraway (Ravidet *al.*, 1992).

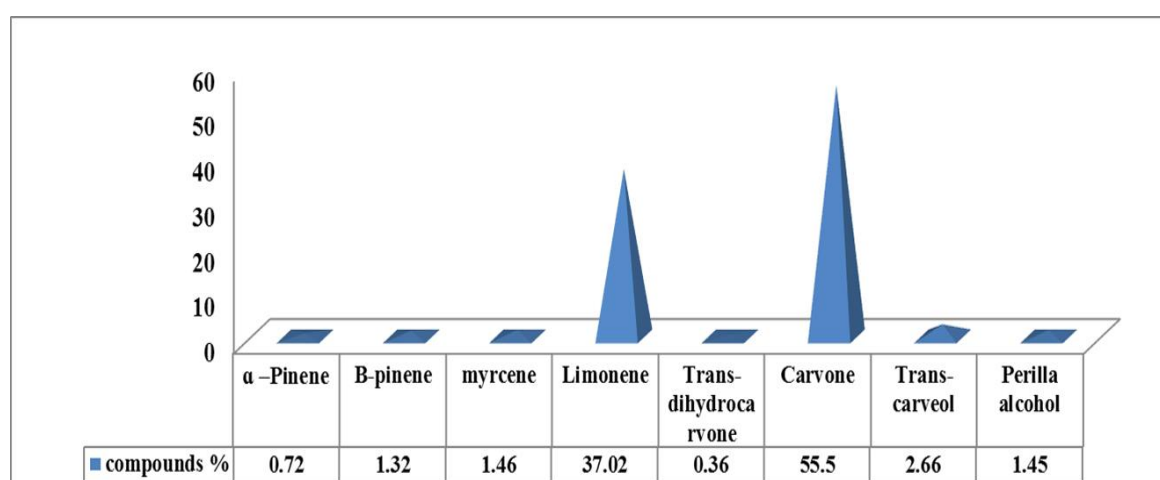


Fig. 1 Chemical composition of Caraway EO.

Weight loss %:

Data illustrated in Table 1 showed the effect of ME and NE of caraway EO on weight loss % of fruits during storage in PP package. Data revealed that, PP packaged decreased weight loss % than control, and also, ME and NE decreased weight loss % when compared with other treatments and control. The differences of all treatments were significant when compared with control. The best treatment was NE at 0.05% PP and NE at 0.1% PP in keep weight loss % of fruits during storage compared other treatments. The good effect of caraway EO in keeping the fruits may be due to, it covers the fruits and does act as a barrier inhibiting moisture loss and retaining weight loss % of fruits during storage. Similar results obtained by Abd El-Gawad and El-Moghazy 2021, NE of EO_s (peppermint and caraway) application significantly decreased weight loss during storage of "Canino" Apricot fruits. Use of packages like plastic trays and polypropylene boxes also help in extending the shelf life of the fresh produce (Amjad, *et al.*, 2010), Packaging is required not only for food preservation but also for protection during handling and safe transportation of products. The export market needs better influence of the packaging trends in food, increasingly (Farooqi, 1994).

Regarding storage periods, the data in Table 1 showed that, in both seasons of study, weight loss % significantly increased gradually with the progress of cold storage. And the

differences among all tested storage periods were significant comparing with the initial date in the two seasons of study. These results are in agreement with those obtained by Malakar, *et al.*, 2020, found that the percentage of weight loss of (*Capsicum chinense* Jacq.) significantly ($P \leq 0.05$) increases with the storage time during the storage period. Similar trend, in weight loss % increased with increasing storage periods of sweet bell pepper fruits (Tsegay, *et al.*, 2013), the high weight loss % in completely ripened fruits could be due to changes in permeability of cell membranes, making them more sensitive to the loss of water as confirmed by Antonialiet *al.*, (2007). Similar weight loss pattern was also noted in the case of peppers (Kabiret *al.*, 2019; Samira *et al.*, 2013) and Ali, *et al.*, 2014, weight loss was observed to gradually increase during the storage period of chilli fruit.

The interaction influence between treatments x storage period, the obtained results indicated that the interactions registered the lowest values of weight loss % with NE at 0.1% PP in both seasons after 8, 16, 24, 32 days under cold storage when compared with treatments other.

Table (1):Effect of ME and NE of Caraway EO on weight loss % of red chili fruits (*Capsicum annum* L.) under cold storage at 7°C and 90-95% R.H. in 2019 and 2020 seasons.

Treatments	Storage Periods (Days)					
	0	8	16	24	32	Means
Season 2019						
Control	0.00	10.50	17.50	28.00	36.23	18.44
PP	0.00	1.68	1.73	2.37	2.67	1.69
NE at 0.05% PP	0.00	0.00	1.66	2.00	2.44	1.22
NE at 0.1% PP	0.00	0.00	1.33	1.75	2.02	1.02
ME at 0.05% PP	0.00	1.50	1.68	2.26	2.63	1.61
ME at 0.1% PP	0.00	0.00	1.66	2.25	2.51	1.28
Means	0.00	2.28	4.26	6.43	8.08	
LSD at 0.05	(T):0.21 (D):0.19(T×D): 0.48					
Season 2020						
Control	0.00	10.09	16.40	27.12	36.79	18.08
PP	0.00	1.79	1.85	2.48	2.99	1.82
NE at 0.05% PP	0.00	0.00	1.76	2.15	2.56	1.29
NE at 0.1% PP	0.00	0.00	1.46	1.91	2.08	1.09
ME at 0.05% PP	0.00	1.65	1.75	2.35	2.80	1.71
ME at 0.1% PP	0.00	0.00	1.70	2.38	2.70	1.35
Means	0.00	2.25	4.15	6.39	8.32	

LSD at 0.05	(T): 0.13(D): 0.12(T×D): 0.29
T: Treatments D: Storage Periods (Days) T×D: Interaction	
PP: polypropyleneME:MicroemulsionNE: NanoemulsionEO: Essential oil	

Decay %:

The presented data in Table (2) showed that the effect of ME and NE of caraway EO on decay % of fruits during 2019 / 2020 seasons. The obtained results revealed that unpackaged (control) recorded the highest significant decay %. The decay could be due to the multiplication of microorganisms and the rapid growth of mold which significantly influenced by different storage conditions (Malakar, *et al.*, 2020). On the other hand NE at 0.05% PP and NE at 0.1% PP treatments recorded the lowest decay % while ME at 0.1% PP treatment was ranked second than other treatments. The positive effect of caraway essential oil in preserving the fruits may be due to, it covers the fruits and does act as a barrier inhibiting multiplication of microorganisms and the rapid growth of mold. The results correspond to the finding of Simic *et al.*, 2008, who found the EO of caraway has stronger antifungal and antibacterial effect than citronella oil. The anti-aflatoxigenic, antioxidant, and antimicrobial effects of caraway oil along with its reputation as spice help the industries to use it as a natural preservative and antioxidant agent (Mahboubi, 2019). Plant EOs incorporated in NE seem to penetrate faster in the microbial membranes due to the increased area per weight unit. This would allow decreasing the concentration to achieve an equivalent or even greater microbial effect over conventional emulsions (Odriozola-Serrano, *et al.*, 2014).

Regarding storage periods, data in Table 2 showed that, in both seasons of study, decay % significantly increased gradually with the progress of cold storage. And the differences among all tested storage periods after 16, 24, 32 days were significant comparing with the zero time in the two seasons of study while after 8 days of storage, no significant difference was found. These results are in line with those stated by Tsegay, *et al.*, 2013 found that starting from two weeks of storage, postharvest decay % of both sweet pepper varieties (Telmo-Red and Velez-Yellow) harvested at all maturity stages was increased with increasing storage periods. The interaction influence between treatments x storage period, the obtained results indicated that the interactions registered the lowest values of decay % with NE at 0.1% PP, NE at 0.05% PP and ME at 0.1% PP treatments respectively, in both seasons after period's storage when compared with treatments other. Fruit decay due to rotting also increased as the storage period. This might be due to condensation of moisture in the surface of fruits during storage period, anaerobic condition, and break down of enzymes etc., which helped in multiplication of micro flora as confirmed by Nath *et al.*, (2012).

Table (2): Effect of ME and NE of Caraway EO on decay % of red chili fruits (*Capsicum annuum* L.) under cold storage at 7°C and 90-95% R.H. in 2019 and 2020 seasons.

Treatments	Storage Periods (Days)					
	0	8	16	24	32	Means
Season 2019						
Control	0.00	0.00	6.60	10.83	12.13	5.91
PP	0.00	0.00	4.83	6.70	7.70	3.84
NE at 0.05% PP	0.00	0.00	0.00	5.77	6.40	2.43
NE at 0.1% PP	0.00	0.00	0.00	4.90	5.83	2.14
ME at 0.05% PP	0.00	0.00	4.33	7.13	7.75	3.83
ME at 0.1% PP	0.00	0.00	0.00	7.05	7.36	2.88
Means	0.00	0.00	2.62	7.06	7.85	
LSD at 0.05	(T):0.61(D):0.56(T×D): 1.37					
Season 2020						
Control	0.00	0.00	6.83	11.90	12.51	6.24
PP	0.00	0.00	5.10	7.15	7.80	4.01
NE at 0.05% PP	0.00	0.00	0.00	6.05	6.76	2.56
NE at 0.1% PP	0.00	0.00	0.00	5.20	5.84	2.20
ME at 0.05% PP	0.00	0.00	5.77	7.33	8.16	4.25
ME at 0.1% PP	0.00	0.00	0.00	7.26	7.57	2.96
Means	0.00	0.00	2.95	7.48	8.10	
LSD at 0.05	(T): 0.39 (D): 0.36 (T×D): 0.89					
T: Treatments D: Storage Periods (Days) T×D: Interaction						
PP: polypropylene ME: Microemulsion NE: NanoemulsionEO: Essential oil						

Fruit firmness:

The ME and NE of EO (caraway) was shown to affect the fruit firmness of red chili fruits during storage 2019 / 2020 seasons (Table 3). Data revealed that, NE at 0.1% PP increased fruit firmness when compared with other treatments. And also, NE at 0.05% PP, ME at 0.1% PP and ME at 0.05% PP treatments, respectively recorded average values from fruit firmness during this study than PP and Control. The positive effect of caraway essential oil in preserving the fruits firmness may be due to, it covers the fruits and does act as a barrier inhibiting moisture loss and retaining firmness.

Regarding storage periods, data in Table 3 showed that, in both seasons of study, fruit firmness significantly decreased gradually with the progress of cold storage. And the differences among all tested storage periods were significant comparing with the initial date in the two seasons of study. These results are harmonious with Malakar, *et al.*, 2020, found that fruit firmness and continuously decreased during the storage period. Firmness changes because of lower metabolic activities at lower temperatures and also water loss in chilies might be a major factor affecting the firmness of chilies, and this was supported by an increasing trend in the weight loss (%) of throughout the storage time (Lim, *et al.*, 2008). Also Ali, *et al.*, 2014, firmness of chillies significantly ($p < 0.05$) declined with storage time. In

general, water loss is the main reason for loss of firmness in peppers. Moreover, decay organisms which produce pectolytic enzymes and infection itself may be the cause of accelerated softening. On the other hand the interaction influence between treatments x storage period, not significant in this study.

Table (3): Effect of ME and NE of Caraway EO on fruit firmness of red chili fruits (*Capsicum annuum* L.) under cold storage at 7°C and 90-95% R.H. in 2019 and 2020 seasons.

Treatments	Storage Periods (Days)					
	0	8	16	24	32	Means
Season 2019						
Control	4.71	3.22	2.90	2.21	2.02	3.01
PP	4.71	3.36	3.29	2.65	2.47	3.29
NE at 0.05% PP	4.71	4.14	3.91	3.60	3.23	3.91
NE at 0.1% PP	4.71	4.30	4.12	4.17	4.18	4.29
ME at 0.05% PP	4.71	3.52	3.65	3.33	3.34	3.71
ME at 0.1% PP	4.71	3.60	3.72	3.66	3.76	3.89
Means	4.71 A	3.69 B	3.59 B	3.27 C	3.16 C	
LSD at 0.05	(T):0.34(D):0.31(T×D): NS					
Season 2020						
Control	4.59	3.39	3.10	3.07	2.30	3.29
PP	4.59	3.93	3.69	3.60	2.74	3.71
NE at 0.05% PP	4.59	4.20	4.09	4.05	3.72	4.13
NE at 0.1% PP	4.59	4.58	4.56	4.47	4.11	4.46
ME at 0.05% PP	4.59	4.00	3.75	3.67	3.04	3.81
ME at 0.1% PP	4.59	3.96	3.85	3.79	3.38	3.91
Means	4.59	4.01	3.84	3.77	3.21	
LSD at 0.05	(T): 0.45 (D): 0.41 (T×D): NS					
T: Treatments D: Storage Periods (Days) T×D: Interaction						
PP: polypropylene ME: Microemulsion NE: NanoemulsionEO: Essential oil						

Total soluble solids

The influence of postharvest applications of various used treatments were reported in Table (4) in terms of their influence on total soluble solids of red chili fruits (*Capsicum annuum* L.) fruits during 32 days of cold storage in 2019 and 2020 seasons. Results indicated that, in two seasons, all treatments significantly increased fruit total soluble solids compared with control. In addition, storage periods effective on increasing total soluble solids contents and the differences were big enough to be significant compared with zero time. These results are harmonious with Panigrahi, *et al.*, 2018 found that, with the extension of storage, the total soluble sugar tended to increase in both the castor oil coated and non-coated fruits (*Capsicum annuum* L.). Interestingly, the degree of this increase in total soluble sugar was significantly

higher in non-coated fruits at the very early stage (9 days) of storage; however, the coated fruits surpassed the total soluble sugar content in its later stages of storage (18–36 days). The present results of change in total soluble sugar content corroborate previous studies on the use of coatings in tomato (Beckles 2012). On the other hand the interaction influence between treatments x storage period, not significant. TSS content was increased during the first two weeks storage under PRS followed by a decreasing trend with increase in storage duration (Tsegay, *et al.*, 2013); The increment in TSS for stored fruits was probably due to increase of respiration and metabolic activity. Rao *et al.* (2011) who found an increase in TSS as fruits were stored for short period followed by a decreasing trend during prolonged storage periods. In this regard, Ali *et al.* (2011) found that the higher respiration rate increases the synthesis and use of metabolites result in higher TSS due to the higher change from carbohydrates to sugars.

Table (4): Effect of ME and NE of Caraway EO on total soluble solids of red chili fruits (*Capsicum annum* L.) under cold storage at 7°C and 90-95% R.H. in 2019 and 2020 seasons.

Treatments	Storage Periods (Days)					
	0	8	16	24	32	Means
Season 2019						
Control	5.13	5.50	5.68	6.10	6.30	5.74
PP	5.13	5.27	5.51	5.68	6.26	5.57
NE at 0.05% PP	5.13	5.75	5.86	6.43	6.58	5.95
NE at 0.1% PP	5.13	5.65	5.78	6.26	6.45	5.85
ME at 0.05% PP	5.13	5.58	5.73	6.16	6.41	5.80
ME at 0.1% PP	5.13	5.55	5.70	6.21	6.37	5.79
Means	5.13	5.55	5.71	6.14	6.39	
LSD at 0.05	(T):0.19(D):0.18(T×D): NS					
Season 2020						
Control	5.26	5.60	6.19	6.40	6.53	5.99
PP	5.26	5.36	5.70	6.27	6.43	5.80
NE at 0.05% PP	5.26	5.85	6.57	6.60	6.73	6.20
NE at 0.1% PP	5.26	5.78	6.33	6.48	6.65	6.10
ME at 0.05% PP	5.26	5.70	6.30	6.48	6.62	6.07
ME at 0.1% PP	5.26	5.63	6.28	6.42	6.58	6.03
Means	5.26	5.65	6.22	6.44	6.59	
LSD at 0.05	(T): 0.15(D): 0.13(T×D): NS					
T: Treatments D: Storage Periods (Days) T×D: Interaction PP: polypropylene ME: Microemulsion NE: NanoemulsionEO: Essential oil						

Anthocyanin content:

Results in Fig. (2) indicated that the effect of ME and NE of EO (caraway) on anthocyanin content of chili fruits (*Capsicum annuum*L.) during storage, the NE at 0.05% PP, NE at 0.1% PP and ME at 0.1% PP treatments saved anthocyanin content in acceptable level without big changes than other treatments. During storage period increased anthocyanin Content compared with Starting point Fig. 3. As well as Barbosa, *et al.*, 2020 found that although some small changes were observed during the storage of red peppers in anthocyanin content, these changes seemed to be random, with no apparent trend. Small differences are graphically observed for samples with 10 and 14 days of storage, respectively.

The interaction between treatments x storage period Fig. 4, the obtained results indicated that the interactions registered the lowest values of anthocyanin Content with NE at 0.05% PP, NE at 0.1% PP and ME at 0.1% PP in both seasons after period's storage, 8, 16, 24, 32 days under cold storage when compared with treatments other. Colour is a crucial component of quality and end users acceptability, especially in the context of chillies (Ali, *et al.*, 2014). Pigment degradation occurs in red chillies where the red lycopene pigment is degraded while anthocyanin is concomitantly synthesized during ripening (Aza-González *et al.* 2012).

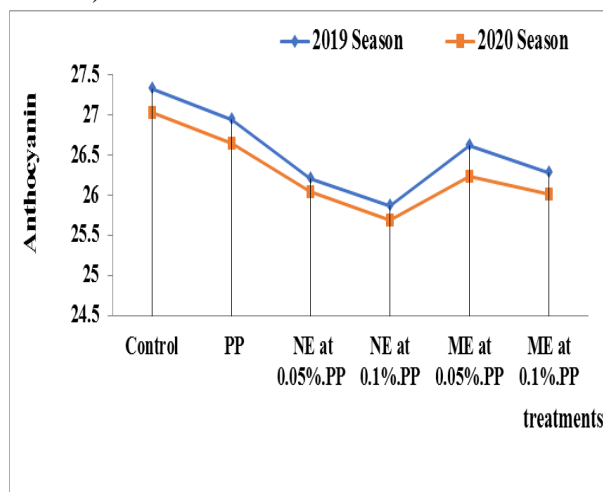


Fig. 2. Effect of treatments on anthocyanin

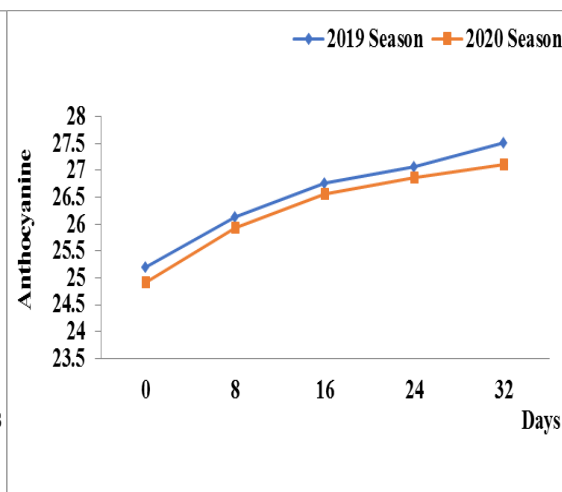


Fig. 3. Effect of period storage on anthocyanin

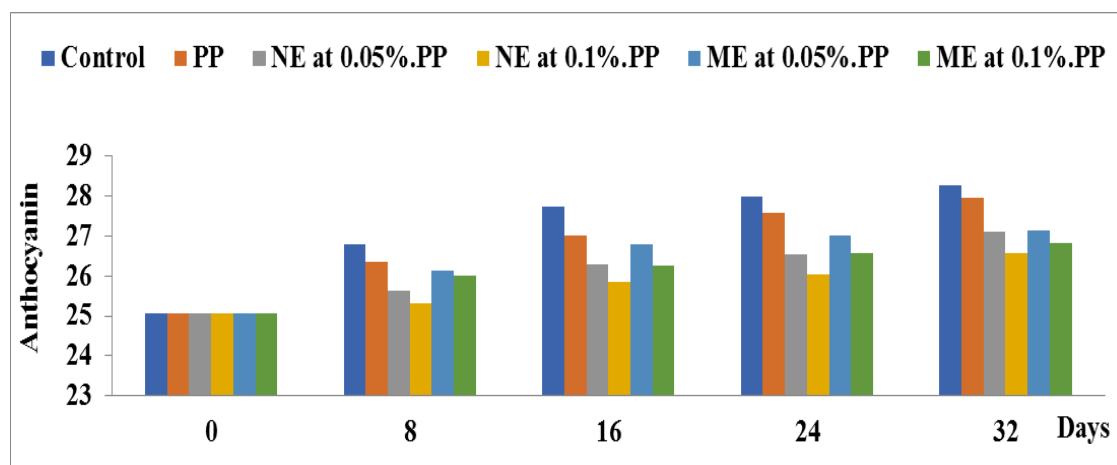


Fig. 4. Effect of interaction between treatments x storage period on anthocyanin content of red chili fruits (*Capsicum annuum*L.) under cold storage at 7°C and 90-95% R.H. in 2019 and 2020 seasons.

Total acidity:

Data illustrated in Table 5 showed the effect of ME and NE of caraway EO on total acidity % of fruits during storage in PP package. Data revealed that, PP package, ME and NE saved total acidity % than control. The best treatments were NE at 0.05% PP, NE at 0.1% PP, ME at 0.05% PP and ME at 0.1% PP in keep total acidity % of fruits during storage compared control treatment. After the harvest of fruits, the respiration increases with a decrease in citric acid and other intermediate products of TCA cycle. The coating on fruit surface might have hindered or prevents the sudden rise in respiration and consequent postharvest maturation (Panigrahi, *et al.*, 2018). Which was also reported earlier time by Yaman and Bayindirli (2001). The probable reason might be attributed to lesser metabolic activities (Özden and Bayindirli 2002) and delay in consumption of citric acids (Yaman and Bayindirli 2001). Citric acid is the mainstay of respiration, wherein a decrease in acidity level and a rise in pH level are coupled with highly respiring fruits (Panigrahi *et al.*, 2017). The decrease in titratable acidity is a vital event during ripening, since it turns the fruits less acidic or sour (Valero and Serrano 2010). Regarding storage periods effective on decreasing total acidity % and the differences were big enough to be significant compared with zero time. These results are harmonious with (Panigrahi, *et al.*, 2018), the titratable acidity showed a decreasing trend in both non-coated and coated fruits (castor oil) with the passage of storage period on *C. annuum* fruits. The coated fruits showed an extended storage-life up to 36 days, which was significantly longer as compared to the non-coated fruits having a limited storage period (18 days) only. On the other hand the interaction influence between treatments x storage period, not significant during first season while, it was significant in second season.

Table (5): Effect of ME and NE of Caraway EO on total acidity of red chili fruits (*Capsicum annuum* L.) under cold storage at 7°C and 90-95% R.H. in 2019 and 2020 seasons.

Treatments	Storage Periods (Days)					
	0	8	16	24	32	Means
Season 2019						
Control	0.496	0.331	0.293	0.266	0.251	0.327
PP	0.496	0.477	0.457	0.406	0.390	0.445
NE at 0.05% PP	0.496	0.481	0.448	0.417	0.408	0.450
NE at 0.1% PP	0.496	0.482	0.446	0.435	0.430	0.457
ME at 0.05% PP	0.496	0.485	0.460	0.417	0.405	0.452
ME at 0.1% PP	0.496	0.491	0.482	0.414	0.413	0.459
Means	0.496	0.457	0.431	0.392	0.382	
LSD at 0.05	(T):0.032(D):0.029(T×D): NS					
Season 2020						
Control	0.479	0.433	0.365	0.351	0.255	0.376
PP	0.479	0.460	0.426	0.409	0.402	0.435
NE at 0.05% PP	0.479	0.461	0.447	0.410	0.400	0.439
NE at 0.1% PP	0.479	0.476	0.454	0.433	0.428	0.454
ME at 0.05% PP	0.479	0.465	0.442	0.410	0.407	0.440

ME at 0.1% PP	0.479	0.478	0.489	0.419	0.418	0.456
Means	0.479	0.462	0.437	0.405	0.385	
LSD at 0.05	(T): 0.012 (D): 0.011(T×D): 0.028					
T: Treatments D: Storage Periods (Days) T×D: Interaction						
PP:polypropylene ME:Microemulsion NE: Nanoemulsion EO:Essential oil						

Capsaicin content:

Results in Fig. (5) indicated that the effect of ME and NE of EO (Caraway) on capsaicin content of chili fruits (*Capsicum annuum*L.) during storage, the control treatments saved capsaicin content in low level than other treatments. Found that NE at 0.1% PP, NE at 0.05% PP and ME at 0.1% PP treatments were better in kept capsaicin content compared with all treatments.

As for the influence of storage period on the capsaicin content; the data in Fig. (6) generally revealed that capsaicin content decreased gradually with the progress of cold storage. The decline in capsaicin content occurred due to the pigment of the color break as a result of inhibition of the capsaicin synthesis, degradation by peroxidase and chemical disintegration caused by photo-oxidation (Reyes-Escogido *et al.* 2011). This result is in agreement with those obtained by Malakar, *et al.*, 2020, Capsaicin content was found increased up to 5 days of storage; thereafter, it began moderately decreasing during the storage period on (*Capsicum chinense* Jacq.). The capsaicin was increasing in the initial stage during the storage period (Bae *et al.*, 2014 and Manikharda *et al.*, 2018). Also, capsaicin content of king chili was moderately changing during storage period (Sarwa *et al.*, 2012)

The interaction among treatments and storage period Fig. 7; the results revealed that NE at 0.1% PP, NE at 0.05% PP and ME at 0.1% PP treatments after 16, 24, 32 days were better than other treatments in the same periods storage.

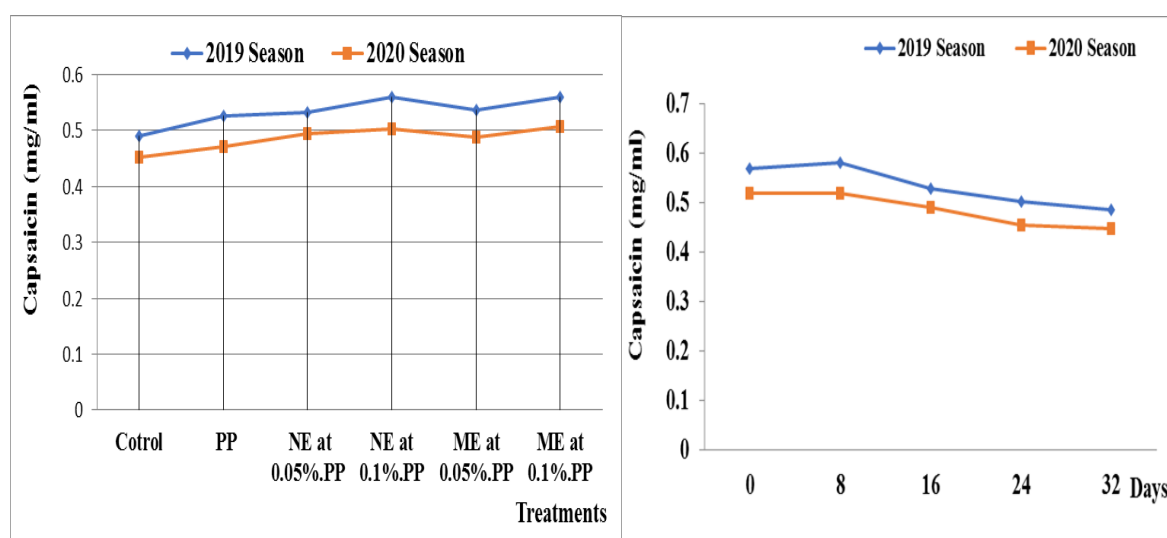


Fig. 5.Effect of treatments on capsaicin

Fig. 6. Effect of period storage on contentcapsaicin content

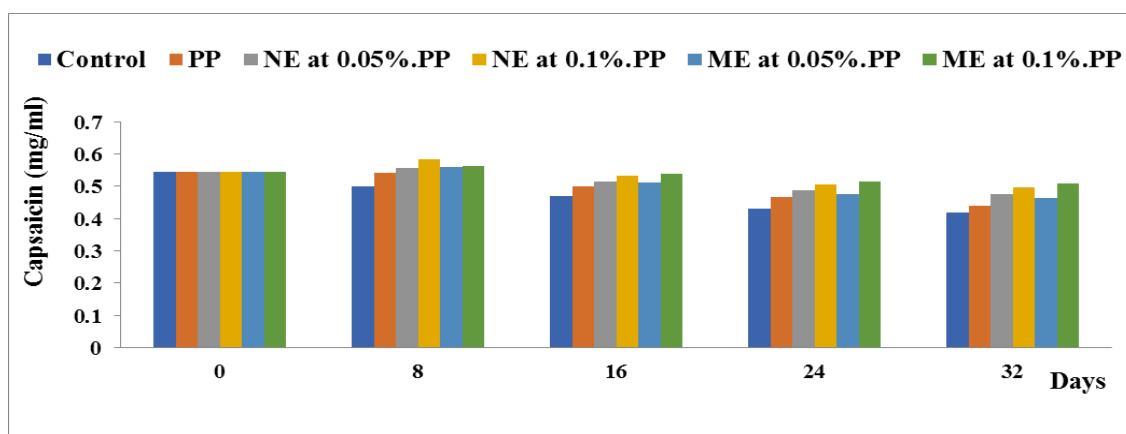


Fig. 7.Effect of interaction between treatments x storage period on capsaicin content(mg/ml) of red chili fruits (*Capsicum annuum*L.) under cold storage at 7°C and 90-95% R.H. in 2019 and 2020 seasons.

CONCLUSION

The impacts of ME and NE of Caraway EO on the quality parameters of fruits stored at 7°C and 90-95% R.H. for 32 days were investigated. The NE of EO at 0.1 and 0.05% from caraway gave the best efficacy on weight loss, decay, firmness, total soluble solids, titratable acidity, anthocyanin content and capsaicin content than microemulsions. Generally, NE at 0.1% PP, NE at 0.05% PP and ME at 0.1% PP treatments were the best among all treatments during the storage period. NE of EO extracted from caraway fruits are natural products can be used as alternative treatment in order to reduce (*Capsicum annuum*L.) fruits postharvest losses because cheap and safe. The caraway oil contains limonene and carvone which have antibacterial, antifungal and antioxidant effects.

This study recommends that, we can use NE at 0.05, 0.1% and ME at 0.1% of caraway EO as a natural product in order to keep fruits quality and extend cold storage period of red chili fruits (*Capsicum annuum*L.).

REFERENCES

- [1] Abd El-Gawad, M. G., & El-Moghazy T. F. A. (2021). Efficacy of Nanoemulsions of Peppermint and Caraway Oils on Quality of "Canino" Apricot Fruits Under Cold Storage. *Plant Cell Biotechnology and Molecular Biology* 22(1&2):65-83; 2021 ISSN: 0972-2025.
- [2] Abd-Elsalam, K.A., & Khokhlov, A.R. (2015). Eugenol oil nanoemulsion: antifungal activity against *Fusarium oxysporum* f. sp. *vasinfectum* and phytotoxicity on cottonseeds. *Appl. Nanosci.* 5, 255–265. <https://doi.org/10.1007/s13204-014-0398-y>
- [3] Ali, A., Chow, W. L., Zahid, N., & Ong, M. K. (2014). Efficacy of propolis and cinnamon oil coating in controlling post-harvest anthracnose and quality of chilli (*Capsicum annuum* L.) during cold storage. *Food and bioprocess technology*, 7(9), 2742-2748.
- [4] Ali, A., Muhammad, M. T. M., Sijam, K., & Siddiqui, Y. (2011). Effect of chitosan coatings on the physicochemical characteristics of Eksotika II papaya (*Carica papaya* L.) fruit during cold storage. *Food chemistry*, 124(2), 620-626.
- [5] Amjad, M., Iqbal, J., Iqbal, Q., Nawaz, A., Ahmad, T., & Rees, D. (2010). Effect of packaging material and different storage regimes on shelf life and biochemical composition of green hot pepper fruits. *Acta Horticulturae* 876 (ISHS 2010):227-234.
- [6] Amruthraj N.J., Preetam Raj, J.P., & Antoine Lebel L. (2014). Effect of vegetable oil in the solubility of capsaicinoids extracted from *Capsicum chinense* Bhut Jolokia. *Asian J Pharm Clin. Res.*, 7(1), 48-51.

- [7] Antoniali S, Leal, P.A. M., Magalhães,A.M., Fuziki,R.T.,&SanchesJ. (2007). Physicochemical characterization of ‘zarcos’ yellow bell pepper for different ripeness stages. *J. Sci. Food Agric.* 64:19-22.
- [8] Aza-González, C., Núñez-Palenius, H. G., & Ochoa-Alejo, N. (2012). Molecular biology of chili pepper anthocyanin biosynthesis. *Journal of the Mexican Chemical Society*, 56 ,93–98.
- [9] Bae, H., Jayaprakasha, G. K., Crosby, K., Yoo, K. S., Leskovar, D. I., Jifon, J., &Patil, B. S. (2014). Ascorbic acid, capsaicinoid, and flavonoid aglycone concentrations as a function of fruit maturity stage in greenhouse-grown peppers. *Journal of Food Composition and Analysis*, 33(2), 195-202.
- [10] Bajaj K. L. (1980). Colorimetric determination of capsaicin in *Capsicum* fruits. *Assos Anal Chem*63: 1314-16.
- [11] Barbosa, C., Machado, T. B., Alves, M. R., & Oliveira, M. B. P. (2020). Fresh-Cut Bell Peppers in Modified Atmosphere Packaging: Improving Shelf Life to Answer Food Security Concerns. *Molecules*, 25(10), 2323.
- [12] BAS. 2018/2019. Bulletin of the Agricultural Statistics, 2019/2018, part 1 2.
- [13] Beckles, D. M. (2012). Factors affecting the postharvest soluble solids and sugar content of tomato (*Solanumlycopersicum* L.) fruit. *Postharvest Biology and Technology*, 63(1), 129-140.
- [14] Brasil –, Ministério da Saúde., &Instituto Adolfo Lutz. (2008). Métodosfísicos e químicosparaanálise de alimentos (4th ed.). São Paulo: Instituto Adolfo Lutz.
- [15] Lim, C. S., Lim, J. M., Kim, B. S., Kang, S. M., Cho, J. L., & Hwang, H. J. (2008). Changes in fruit quality of paprika and color pimento (*capsicum annuum* l.) stored at low temperatures. *ActaHorticulturae*, (768), 539-544.<https://doi.org/10.17660/ActaHortic.2008.768.72>.
- [16] Ding, P., & Lee, Y. L. (2019). Use of essential oils for prolonging postharvest life of fresh fruits and vegetables. *International Food Research Journal*, 26 (2). 363- 366.
- [17] Donsi, F., &Ferrari, G. (2016). Essential oil nanoemulsions as antimicrobial agents in food. *J. Biotechnol.*, 233, 106–120.
- [18] Egyptian Pharmacopoeia (2005). Central Administration of pharmaceutical Affairs (CAPA). Cairo, Egypt : Ministry of Health and Population. , 4th ed. PP. 31-33.
- [19] FAO. IFAD (2012) The State of Food Insecurity in the World 2012: economic growth is necessary but not sufficient to accelerate reduction of hunger and malnutrition. Rome: FAO; 2014.
- [20] Farooqi, W.A. 1994. Post-Harvest Handling. p.401-427. In: M.N. Malik (ed.), *Horticulture*. National Book Foundation, Islamabad, Pakistan.
- [21] Gomez, K.A. & A.A. Gomez (1984). Statistical procedure for agricultural research. 2nd Edition. pp. 8-22.
- [22] Kabir, M. S. N., Chowdhury, M., Lee, W. H., Hwang, Y. S., Cho, S. I., & Chung, S. O. (2019). Influence of delayed cooling on quality of bell pepper (*Capsicum annuum* L.) stored in a controlled chamber. *Emirates Journal of Food and Agriculture*, 31(4), 271–280. <https://doi.org/10.9755/ejfa.2019.v31.i4.1936>.
- [23] Khater, H., Govindarajan, M.,&Benelli, G. (2017). *Natural Remedies in the Fight Against Parasites*; IntechBoD-Books on Demand: London, UK, ISBN 953513289X.
- [24] Khalil, N., Ashour, M., Fikry, S., Singab, A. N., &Salama, O. (2018). Chemical composition and antimicrobial activity of the essential oils of selected Apiaceous fruits. *Future Journal of Pharmaceutical Sciences*, 4(1), 88-92.
- [25] Khetrpal, V., Chawla, N. &Sandhu, J.S. (2015). Effect of nutrient limitation on bio-synthesis of capsaicin. *Int. J. of Advanced Res.*, Vol. 3, Issue 3, 1223-1230.
- [26] Liljana K. G., Viktorija M., Marija, S. D., Rubin G., &Emilija I. J. (2013).The effect of different methods of extractions of capsaicin on its content in the capsicum oleoresins. *Scientific Works: Food Science, Engineering and Technology* 60, 917-922.
- [27] Lim, C. S., Lim, J. M., Kim, B. S., Kang, S. M., Cho, J. L., & Hwang, H. J. (2008). Changes in fruit quality of paprika and color pimento (*capsicum annuum* l.) stored at low temperatures. *ActaHorticulturae*, 76, 539–544. <https://doi.org/10.17660/ActaHortic.2008.768.72>.
- [28] Mahboubi M. (2019). Caraway as Important Medicinal Plants in Management of Diseases. *j. Nat. Prod. Bioprospect.* Feb; 9(1): 1–11.
- [29] Malakar, S., Kumar, N., Sarkar, S., & Mohan, R. J. (2020). Influence of Modified Atmosphere Packaging on the Shelf Life and Postharvest Quality Attributes of King Chili (*Capsicum chinense*Jacq.) during Storage. *J.ofBiosyst. Eng.*, 1-10.

- [30] Manikharda, Takahashi, M., Arakaki, M., Yonamine, K., Hashimoto, F., Takara, K., & Wada, K. (2018). Influence of fruit ripening on color, organic acid contents, capsaicinoids, aroma compounds, and antioxidant capacity of shimatogarashi (*Capsicum frutescens*). *Journal of Oleo Science*, 67(1), 113–123. <https://doi.org/10.5650/jos.ess17156>.
- [31] Mora-Huertas C. E., Fessi H., & Elaissari A. (2010). Polymer-based nanocapsules for drug delivery. *Int. J. Pharm.* 385:113–142.
- [32] Nath, A., Deka, B. C., Singh, A., Patel, R. K., Paul, D., Misra, L. K., & Ojha, H. (2012). Extension of shelf life of pear fruits using different packaging materials. *Journal of food science and technology*, 49 (5), 556-563.
- [33] Özden, Ç., & Bayindirli, L. (2002). Effects of combinational use of controlled atmosphere, cold storage and edible coating applications on shelf life and quality attributes of green peppers. *European Food Research and Technology*, 214(4), 320-326.
- [34] Odrizola-Serrano I., Oms-Oliu G., & Martín-Belloso O. (2014). Nanoemulsion-based delivery systems to improve functionality of lipophilic components. *Front Nutr.* 5: 1:24.
- [35] Panigrahi J., Gheewala B., Patel M., Patel N., & Gantait S. (2017). Gibberellic acid coating: A novel approach to expand the shelf-life in green chilli (*Capsicum annuum* L.). *SciHortic* 225:581–588.
- [36] Panigrahi, J., Patel, M., Patel, N., Gheewala, B., & Gantait, S. (2018). Changes in antioxidant and biochemical activities in castor oil-coated *Capsicum annuum* L. during postharvest storage. *3 Biotech*, 8, 1-8. <https://doi.org/10.1007/s13205-018-1284-1>.
- [37] Pavoni, L., Perinelli, D. R., Bonacucina, G., Cespi, M., & Palmieri, G. F. (2020). An overview of micro- and nanoemulsions as vehicles for essential oils: Formulation, preparation and stability. *Nanomaterials*, 10(1), 135.
- [38] Popelka, P., Jevinová, P., Šmejkal, K., & Roba, P. (2017). Determination of capsaicin content and pungency level of different fresh and dried chilli peppers. *Folia Veterinaria*, 61(2), 11-16.
- [39] Ravid U., Putievsky E., Katzir I., Weinstein V., & Ikan R. (1992). Chiral GC analysis of (S)(+)- and (R)(-)-carvone with high enantiomeric purity in caraway, dill and spearmint oils. *FlavourFragr. J.*;7:289–292.
- [40] Reyes-Escogido, M. D. L., Gonzalez-Mondragon, E. G., & Vazquez-Tzompantzi, E. (2011). Chemical and pharmacological aspects of capsaicin. *Molecules*, 16(2), 1253-1270.
- [41] Rao, T.V. R., Gol, N. B., & Shah K.K. (2011). Effect of postharvest treatments and storage temperatures on the quality and shelf life of sweet pepper (*Capsicum annum* L.). *Sci. Hortic.* 132:18-26.
- [42] Samira, A., Woldetsadik, K., & Workneh, T. S. (2013). Postharvest quality and shelf life of some hot pepper varieties. *J. of Food Science and Technology*, 50(5), 842–855. <https://doi.org/10.1007/s13197-011-0405-1>.
- [43] Sarwa, K. K., Kiran, J., Sahu, J., Rudrapal, M., & Debnath, M. (2012). A short review on *Capsicum chinense* Jacq. *J. of Herbal Medicine and Toxicology*, 6(2), 7-10.
- [44] Simic, A., Rančić, A., Sokovic, M.D., Ristic, M., Grujic-Jovanovic, S., Vukojevic, J., & Marin, P.D. (2008). Essential Oil Composition of *Cymbopogon winterianus* and *Carum carvi* and Their Antimicrobial Activities. *Pharma. Biolo.*, Vol. 46, No. 6, pp. 437–441.
- [45] Sampathi S., Mankala S. K., Wankar J., & Dodoala S. (2015). Nanoemulsion based hydrogel of itraconazole for transdermal drug delivery. *J. Sci&Indust.Res.* 74:88-92.
- [46] Tan, C. K., Ali, Z. M., Ismail, I., & Zainal, Z. (2012). Effects of 1-methylcyclopropene and modified atmosphere packaging on the antioxidant capacity in pepper “kulai” during low-temperature storage. *The Scientific World Journal*, Volume 2012, Article ID 474801, 10 pages.
- [47] Tsegay, D., Tesfaye, B., Mohammed, A., Yirga H., & Bayleyegn A. (2013). Effects of harvesting stage and storage duration on postharvest quality and shelf life of sweet bell pepper (*Capsicum annuum* L.) varieties under passive refrigeration system. *Int. J. Biotechnol. Mol. Biol. Res.* 98-104.
- [48] Valero D., Serrano M. (eds) (2010) Postharvest biology and technology for preserving fruit quality. CRC Press, USA, Boca Raton.
- [49] Yaman Ö., Bayindirli L. (2001). Effects of an edible coating, fungicide and cold storage on microbial spoilage of cherries. *Eur Food Res Technol* 213:53–55.
- [50] Yilidiz, F.A. and D.S. Dikmen (1990). The extraction of anthocyanin from black grape skins Doga. Degisi, 14(1): 57-66.