

# Recovery of Vitamin E from Edible Oil – A Review

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**Abstract** - Vitamin E is a fat-soluble antioxidant that is important for the protection of essential fatty acids against oxidative deterioration in both plants and animals. Vitamin E plays an important role in the prevention of diseases. Soya fat contains high proportion of vitamin E and fatty acids. Soybean is considered an important crop in the world due to its unique nutritional composition. On an average dry matter basis, it contains about 20% fat. There are various methods for the extraction of vitamin E. At different operating condition the concentration level or the separation factor of the mixture has a strong effect on extraction of a component. Total tocopherols content gradually decreased until the end of the refining processes during extraction. Commercially, tocopherols with vitamin E actively are sold if they are concentrated at least 60%. Vitamin E is needed in food, cosmetics, pharmaceuticals, chemical industries, etc. The present paper deals with research work carried out in the extraction, recovery of vitamin E from different oils with the different processes. The feasibility of various processes are taken into consideration.

**Keywords** - Vitamin E, Edible Oil, Extraction Techniques, Deodorized Soybean Oil.

## INTRODUCTION

The edible oil used in food preparation contains the various compositions of vitamin E that increases the oxidative stability of edible oils. The byproduct of various edible oil refineries mainly from the deodorization process contains an appreciable amount of vitamin E, in the form of tocopherols and sterols. The deodorized soya oil is the best source of tocopherols (vitamin E). It plays an important role due to its nutritional properties. It is highly antioxidant, strong cholesterol reducing agent, protecting cell membranes from oxidative damages. Apart from various edible oil resources, it can also be recovered from used edible oils. The present paper deals with the study of recovery of vitamin E from various edible oils, their process conditions, operating conditions, and level of extraction.

## LITERATURE REVIEW

Tocopherol is one of the most important vitamins E that is useful for various biological properties in human beings. A large amount of vitamin E is being consumed in the society in the form of various medicines and for curing diseases. Various processes are available for the extraction of vitamin E from edible oils. All these processes have got own limitations and benefits. The literatures related to the recovery of vitamin E are as follows:

In the year (2018), Mathias Z. et al., studied the contribution of ratio of tocopherol homolog to the oxidative stability of commercial vegetable oils like soybean, sunflower and canola. The outline for the study includes the effect of different tocopherol concentrations, homolog and ratio of homolog on markers of lipid oxidation in the vegetable oil. The oil was stored in light/dark place for a period of a 12 hour cycle at  $22 \pm 2^\circ\text{C}$  for 56 days under household condition. It was noted that the  $\alpha$ -tocopherol-rich peroxide value strongly rise in the canola and sunflower i.e.  $25.1 \pm 0.03$  mEq  $\text{O}_2/\text{kg}$  and  $24.7 \pm 0.054$  mEq  $\text{O}_2/\text{kg}$  respectively. The food representative tocopherol ratio  $(\gamma+\delta)/\alpha$  found to be 4.77 in the soybean oil.

Waniek S. et al., (2017) reported the distribution of vitamin E level in the community, clinical and biochemical correlation and association with the dietary patterns. The study includes sample collection, design, clinical examinations, assessment of dietary variables, laboratory analysis, statistical analysis, correlates of circulating vitamin E biomarkers and the dietary pattern analysis. The parameter includes the distribution of both  $\alpha$ - and  $\gamma$ - tocopherol levels with a priori and a posteriori derived dietary patterns. HPLC with fluorescence detection was used in 641 individuals (mean age: 61 years; 40.6% women) to measure plasma  $\alpha$ - and  $\gamma$ -tocopherol concentrations. Correlation of both markers was determined by linear regression with the backward selection. All the supplementation was identified as correlates of vitamin E. It was observed that median concentrations of  $\alpha$ -tocopherol found  $31.54\mu\text{mol/L}$ , and  $1.35\mu\text{mol/L}$  for  $\gamma$ - tocopherol.

Fritsche S. et al., (2017), reviewed recent advances of tocopherol biosynthesis in plant with key genes, function, and breeding of vitamin E improved crops. The framework for the study includes species, Arabidopsis thaliana, tocopherol biosynthesis and functional role of tocopherols. The improvement of vitamin E content using transgenic approaches and classical breeding. The research efforts have led to new outcomes for the vitamin E biosynthetic and related pathways and possible alternatives for the biofortification of important crops. It was noted the biosynthetic bottlenecks detected, transgenic and non-transgenic breeding efforts have been undertaken to improve the antioxidant and nutritional values of crops.

In the year (2016), G.G. Duthie et al., studied an oxidative stability of edible oil during the storage. that was dependent on the antioxidant capacity of oil. The parameter for the study includes an antioxidative properties of tocopherol arises from electron donations ability of the compounds. The spectrometer operate at 9.5 GHz, with cylinder cavity of oils for quenching a resonance stabilized free radical species (galvinoxyl). It was noted that the antioxidant properties of oils increase the stability of several vegetable oils resulting in enhancing the properties of oil during storage. The reactivity of vitamin E with galvinoxyl was the combination of  $\alpha$ - and  $\gamma$ -tocopherol homologs between the olive 20% and soya 85% of antioxidant capacity on the oils.

Ingle U.M. et al., studied the purification of vitamin E acetate by using a kinetic chromatography in the year (2016). The factor for the study includes the design of loading conditions for purification of VEA. The defined parameter RSF used successfully through developing the correlation of bed-height (170cm) and prediction of height required to achieve desired targeted purity (>98 wt. % ) of VEA. Critical design of equilibration, washing and elution conditions mentioned for the development of kinetic separation process to generate VEA of purity in one step with the recovery of 90%. The factor based on the maximum retention time of impurity (mixture) peak and VEA was used as a measure of the degree of separation in the work. The suitable adsorbent for separation was selected based on shallow-bed binding-elution characteristics. It was noted that in isocratic mobile phase, 2% (wt/wt) water in methanol provides high purity with high recovery of VEA.

Sherazi et al., (2016) studied the recovery of vitamin E from vegetable oil deodorizer distillates, which is a rich source of the natural bioactive components like tocopherols, sterols, FFA etc. The framework for the study includes degumming, bleaching and deodorization process steps in chemical refining and in the physical refining. The compositions of unsaponified deodorized distillates obtained from refining were processed by analytical methods and characterized by instrumental techniques. It was observed that the future perspective of current field may lead to cost-efficient processes and increased attention to the nutritional quality of the deodorized oil. The deodorize distillates, as well as their valuable components, may be one of raw material for extraction of vitamin E. Total tocopherols and sterols was high (16.3-18.2%), (19.4-21.4%) in soybean in chemical refining. while in the physical refining process, the tocopherols (7.5%) and sterols (10.8%) was high in soybean oil.

In the year (2015), Selina et al., studied the investigation of the natural antioxidants tocopherol content ( $\alpha$ -,  $\beta$ -,  $\delta$ -tocopherol) which was available in edible oils and vanaspati (hydrogenated vegetable fat). Free fatty acid percentage and peroxide value (PV) both are the main quality parameters. Proper seed collection, suitable storage condition and appropriate refining process were maintained to obtain the most favorable tocopherol contents. The changes in FFA% and PV in the oils were evaluated three-month interval through one year assessed for the anti-oxidation. The

oxidative deterioration of oil quality not only depends on the natural tocopherol content but also on its individual tocopherol content, storage conditions, temperature, moisture content etc. It was noted that the content of  $\alpha$ -tocopherol highest in sunflower oil was 20.76 mg/100gm and the content of gamma tocopherol highest in soybean oil was 59.3mg/100gm respectively. According to study the natural antioxidants as tocopherols play an imperative role to control the oil quality. The individual tocopherols content has also importance to stave off the oils from deterioration.

Malekbala M. R. et al., (2015) reviewed, the current technology for the extraction, enrichment and analytical detection of tocopherols and tocotrienols by extractions methods. The framework for the study includes the methods for the extraction of vitamin E by supercritical fluid extraction method, molecular distillation, and adsorption method with experimental conditions of temperature, pressure, and flow-rates. It was noted that these methods are sensitive to different experimental conditions with effect on the efficiency of the enrichment and extraction of vitamin E. The review covered the adopted extraction method for extraction yield under operational conditions.

Khamar R. et al., studied the biochemical profile, the fatty acid profile, & nutraceuticals profiles in the soybean oil, soy germ oil and SODD in the year (2015). The parameter for the study includes the limiting factors in biochemical profile were colors, acid value (AOCS: Cd-3d-63, 1997), Iodine value (AOCS: Cd-1 25-93, 1997) and Saponifiable value (AOCS: Cd-3c-25, 1997). Fatty acid profile was evaluated with gas chromatographic method (AOCS: Ce-1b-89, 1997) includes saturated fatty acid, mono saturated and polyunsaturated. In the nutraceutical profile was the gas chromatography (AOCS: Ce-7-87) with the High-Performance Liquid Chromatography (HPLC) (AOCS: Ce-8-89, 1997) with/includes tocopherol contents, phytosterols, squalene and nutraceutical. It was noted that amount of mixed tocopherols quantity was high (6.56 %) in soybean DOD.

Ubaid A. et al., (2014) studied the estimation of  $\alpha$ -tocopherol content available in vegetable oils and compared the amount of  $\alpha$ -tocopherols in different brands of vegetable oils. The parameter for the study includes the concentration with a height of peaks and retention time of the  $\alpha$ -tocopherol level decreases due to the refining, temperature, saponification in alkaline medium and exposure in the high-intensity light. The Reverse-Phase High-Performance Liquid Chromatography (RP-HPLC) with UV-Visible spectro-photometric detection method was used. It was noted that the concentration of tocopherol in Eva cooking oil was of 97.9007 $\mu$ g/l, Paris soybean oil was of 93.1764 $\mu$ g/l and Maryam cooking oil was of 85.0289 $\mu$ g/l.

Benites C.I. et al., studied the neutralization of Soybean Oil Deodorizer Distillate (SODD) for Vitamin Supplement Production in the year (2014). The parameter for FFA content reduction and concentration or purification of tocopherols study includes the neutralization process by the

lowest leaching of tocopherols with the greatest yield of alkali ( $\text{Na}_2\text{CO}_3$ ) concentration of 4.34N, the temperature of 45.8°C and homogenization time 3 min 20 sec. It was found that the FFA content was reduced after the initial neutralization. It was reduced from 53.4% to 6.1% after the initial neutralization, thus required a second neutralization step. The FFA content 1.8% for alpha tocopherol homologs and total tocopherols (TT) was about 11% of SODD.

Grilo E.C. et al., (2014), studied the concentration of Alpha tocopherol and Gamma tocopherol in vegetable oils and compared the  $\alpha$ -tocopherol value for the nutritional requirement of Vitamin E for adults. The framework for the study includes the analysis by High-Performance Liquid Chromatography. It was noted that the soybean oil was not only considered a source of vitamin E, the canola and corn oil were considered sources and sunflower oil was considered an excellent source. It was observed that concentrations of alpha and gamma-tocopherols were in sunflower  $432.3 \pm 86.6$  and  $92.3 \pm 9.5$  mg/kg, corn oil  $173.0 \pm 82.3$  and  $259.7 \pm 43.8$  mg/kg respectively. It was found that the alpha- and gamma-tocopherol in the soybean oil was of  $71.3 \pm 6.4$  and  $273.3 \pm 11.1$  mg/kg respectively.

In the year (2013), Bele C. et al., studied the quick and direct method of routine analysis of tocopherols ( $\alpha$ -,  $\beta$  +  $\gamma$ -, and  $\delta$ -tocopherol) in vegetable oils by HPLC method with fluorescence detection. The parameter for the study includes for tocopherols in edible oil are standard linearity (the range ( $\mu\text{g}/\text{gram}$ ), the precision (CV %)), sensitivity and accuracy. The quantification of tocopherols was performed by the detector at 290nm excitation wavelength and 325nm emission wavelength. It was noted that the detection method was good in limits to measures the tocopherols and it was separated at 25°C in less than 10min after injection. The  $\alpha$ -tocopherol concentration was 8ng/gram, and 9ng/gram for  $\beta$ -,  $\gamma$ - and  $\delta$ -tocopherols respectively.

Ergonul P.G. et al., studied the changes in  $\alpha$ -,  $\beta$ -,  $\gamma$ -, and  $\delta$ -tocopherols content of most consumed vegetable oils during the refining process in the year (2013). The parameter for the study includes the effect of refining physical, chemical process on total and individual tocopherol content present in vegetable oils. The Changes were in sunflower oil, corn oil, rapeseed oil, soybean oil, olive oil and refined-bleached-deodorized (RBD) palm oil by HPLC with UV detection analysis. It was noted that total tocopherol ( $\alpha$ -,  $\beta$ -,  $\gamma$ -, and  $\delta$ -tocopherols) contents gradually decreased until the end of the refining process in all oil types. The tocopherols found in vegetable oil were  $\alpha$ - and  $\gamma$ -tocopherols. Soybean contains 1328mg/kg tocopherol, which is highest among plant oils.

Nalan A. A. et al.,(2013), studied the comparison of two different strategies for the tocopherols, phytosterols and squalene from the by-product of the edible oils by using the supercritical  $\text{CO}_2$  method which is fast, cheap and reliable. The factor for the study includes temperature, pressure, the number of modifiers, static and dynamic extraction time, amount of sample loading on the purity evaluation was

based on the design. Moreover, gas chromatography (GC) method was developed to examine the chemical composition of the samples. It was observed that the extraction vessel does not have the significant effect on the number of extracts and quality. Dynamic extraction and time were founded to be effective on the quality and amount of total extract with the recovery of sterols and tocopherols.

In the year (2013), El-Shami S. et al., studied the bioactive component of deodorizer distillates produced from the edible oil processing that used as a potential source of natural components. The isolations of the different minor components, tocopherols, and triacylglycerols by HPLC, whereas whole sterols and fatty acids as methyl esters were determined by the GLC. It was noted that the sample of deodorizer distillates contains high tocopherol, (DD1) gamma tocopherol and delta tocopherol i.e.  $(53 \pm 2)\%$  and  $(47 \pm 2)\%$ , whereas the (DD2), delta tocopherol and alpha tocopherol of  $(80.0 \pm 0)\%$ , and  $(11.50 \pm 0.45)\%$  respectively.

B.Goossens et al.,(2011) studied the quantification of vitamin E from vegetable oils. The parameter for the study includes the analysis of  $\alpha$ -, and  $\delta$ -tocopherols in canola and soybean oil by reverse phase high-performance liquid chromatography. The tocopherols were resolved with complete baseline separation. It was noted that the canola oil contained  $19 \pm 2.3$ ppm  $\alpha$ -tocopherol and  $22.9 \pm 0.65$ ppm  $\delta$ -tocopherol. The soybean oil contains  $16 \pm 2.3$ ppm of  $\alpha$ -tocopherol and  $147 \pm 1.8$ ppm of  $\delta$ -tocopherol respectively.

In the year (2010), Kasim et al., studied the Isolations for the tocopherols and free phytosterols from Soybean Oil Deodorizer Distillate (SODD) with high purity and high recovery. The saponification was applied in a polar fraction to remove free fatty acids and acylglycerols. The cold saponification temperature maintained at 60°C. It was observed that the SODD contains both the tocopherols of this  $9.13 \pm 0.28\%$  and this phytosterols  $9.75 \pm 0.12\%$ . It was converted to the final product which contained tocopherols  $38.08 \pm 0.36\%$ , phytosterols  $55.51 \pm 0.56\%$ . The total recovery was  $94 \pm 0.19\%$  for tocopherols and phytosterols respectively.

Yang H. et al., studied the recovery of phytosterols (vitamin E) from the waste residue of soybean oil deodorizer distillate (WRSODD) in the year (2010). The parameter for the study includes a catalytic decomposition and crystallization to recover the phytosterols from WRSODD and the composition was analyzed by column chromatography. The amount of phytosterols in the form of fatty acid steryl esters, reported 20 wt% of WRSODD. It was observed that the yield of recovered phytosterols was 22.95 wt% after the 1st crystallization. The purity of phytosterols reached 91.82, 92.73, and 97.17 wt % after 1st, 2nd, 3rd crystallization respectively.

In the year (2008) B. Edison reported the analysis of tocopherols by high-performance liquid chromatography. The HPLC play an important role to take part the handling of less usual samples, prevention of degradation of heat

sensitive functional group and for the micro preparative purpose. It was noted that the HPLC analysis easy to collect the fractions by other techniques such as chemical degradation or mass spectrometry (MS) or nuclear magnetic resonance spectroscopy.

Zhang W. et al., (2008), studied the separation of individual tocopherols  $\alpha$ -Tocopherol,  $\gamma$ -tocopherol, and  $\delta$ -tocopherol from soybean distillates. The parameter for the study includes the effect of flow-rate, effluent, and sample loading separation efficiency. The concentration of vitamin E with 53% purity, from soybean oil deodorizer distillate by using a low-pressure glass column (500 mm  $\times$  25 mm, I.D., packed with silica gel) chromatography. The conditions were cyclohexane-ethanol 99.7:0.3 (v/v), flow-rate at 25 ml/min and loading amount being 2 ml concentration 1 g/ml. It was noted that the purity of the  $\alpha$ -tocopherol,  $\gamma$ -tocopherol and  $\delta$ -tocopherol were 92.35%, 91.23%, 89.95%, respectively. The recovery was 35.21%, for Alpha-tocopherol, 36.25% for Gamma-tocopherol and for Delta-tocopherol was of 61.25%.

M. Patrisha et al., evaluated the selective solid-phase extraction method for  $\alpha$ -tocopherols by using the adsorbent (ionic liquid modified mesoporous SBA-15) in the year (2008). It was applied for the separation of  $\alpha$ -tocopherol from mixture of soybean oil deodorizer distillates. The parameter for the study includes the adsorption time, structure, loading of ionic liquids, adsorption isotherm and the reusability of adsorbent was investigated by the liquid-solid extraction method. It was found that the concentration of  $\alpha$ -tocopherol was significantly increased from 15.6% in original feedstock solution that contained fatty acid methyl ester, triglyceride and  $\alpha$ -tocopherol of 73.0% respectively.

C. F. Torres et al., (2007) studied the two-step enzymatic procedure for the sterol esters, tocopherols and fatty acid ethyl esters from soybean oil deodorizer distillate. The parameter for the study includes the procedure in a two-step enzymatic reaction system with the catalyst *C rugosa* and *Candida antarctica* lipases. The reaction mixture was carried out with a ratio of free fatty acids (FFA) and progression of sterol esterification. The enzymatic steps were used in order to separate sterols esterification and ethyl esterification in time and space. It was noted that the first stage in presence of *Candida rugosa* lipase (catalyst) was efficient to transform more than 90% of the original sterols in short period time of 5hr. While the second enzymatic step in presence of *Novozym 435* (catalyst) convert more than 95% of the FFA in less time 3hr respectively.

In the year (2007), S. Quek et al., studied the commercial extraction of vitamin E from a food source. The parameter for the study includes the enlargement of the optimal vitamin E recovery, the temperature, and other parameters favorable for the method. Various methods are available for vitamin E like extraction, esterification, saponification, liquid-liquid extraction, crystallization, distillation, ion-exchange and adsorption chromatography were used for vitamin E concentration. These are based on sources and

extraction limitation techniques. It was noted that commercial methods are available according to the requirement, nature and source, recovery and concentration of vitamin E.

Vanessa et al., studied the optimization for tocopherols concentration from Soybean Oil Deodorizer Distillate (SODD) in the year (2007). The parameter for the study includes feed flow rate (F) of 4-12 ml/min and evaporator temperature (T) of 130-200°C. Molecular distillation was applied for the tocopherol concentration. Response surface methodology was used to optimize free fatty acids (FFA) elimination. Tocopherol concentration was obtained in the residue and in the streams. It was noted that high concentration of tocopherols was obtained in the residue stream at the operating condition in a lower value of the feed flow-rate and high evaporator temperature.

In the year (2007), Vastharuba et al., evaluated the changes in essential fatty acid contents and interrelationships in soybean seed during germination and storage. The framework for the study includes the estimation of the content of vitamin E, linoleic and linolenic acid in soybean seeds. The germination time taken was 24, 48, and 72 hrs for storage. The packaging materials Aluminum foil, polythene, paper and unpack were used for the 6 months period time. It was noted that the highest vitamin E content was 12.63 $\mu$ g/g observed in the sample germinated after 48 hrs. The highest amount of linoleic acid 107.57 mg/g, and linolenic acid was 18.27mg/g, respectively.

In the year (2007), Swiglo et al., evaluated the tocopherol content available in edible oils. The parameter for the study includes the concentration, the anti-oxidation & photo-oxidation of edible oils for tocopherol by reversed-phase high-performance liquid chromatography (RP-HPLC). It was observed that tocopherol concentrations varied in the range from 121 to 829 mg/kg. The contents of individual  $\alpha$ -, ( $\beta + \gamma$ )-, and  $\delta$ -tocopherols showed the great diversity that dependent on the kinds of the oil.

Lto V.M. et al., (2007) reported the concentration of tocopherols and phytosterols in soybean oil deodorized distillates. Molecular distillation was applied to eliminate the FFAs and concentrate the tocopherols and phytosterols after ethanolic extraction. The parameter for the study includes the saponification at 65°C by acidulation step with high vacuum, reduced temperature, and low residence time. The separation of tocopherols from phytosterols was difficult by similar properties like molecular weights, boiling point, and vapor pressure. The ethanolic extraction was applied for separation of tocopherols from phytosterols. It was found that the purity of tocopherols was of 26wt%, and 52.5weight% for phytosterols.

Fregolente et al. studied the enrichment details of natural products using an integrated solvent-free molecular distillation process in the year (2006). The study includes separation, purification, and concentration of natural products. The parameter for the study includes the

temperature, high vacuum (around 10-4 mmHg) distillation space, evaporator and condenser (around 2 cm). It was noted that molecular distillation was effective to separate FFA from the reaction. The gamma-linolenic acid (GLA) obtained around 33.6% (1.5 higher than the initial concentration), after 180 min of reaction.

In the year (2006), Martins et al., studied the free fatty acids and tocopherols separation from vegetable oil deodorizer distillate through molecular distillation. The framework for the study includes temperature 100 to 180°C and the fluid flow rate 1.5 to 23.0 g/min was used. An efficient FFA separation from SODD with the lowest loss of tocopherols under specific operating conditions. It was found that 6.4% of FFAs and 18.3% of tocopherols from raw material composed of 57.3% of FFAs and 8.97% of tocopherols. The elimination of FFA was of 96.16 % with tocopherol recovery of 81.23%.

Mendes et al., (2005) studied on the recovery of high aggregated compounds present in deodorizer distillate of the vegetable oil. The parameter for the study includes the oil taken in the research work like corn, sunflower, canola, soybean known as deodorized distillate for concentration and separation % with Supercritical carbon dioxide, semi-batch operating conditions & varying temperature from 40-80°C, and pressure 90-350 bar. It was noted that the temperature of 40°C was optimum to promote the effective separation between all the components involved. The pressure was necessary to concentrate the tocopherol and to separate linoleic acid (90 bar), stigmaterol (250 bar), and squalene (350 bar) from tocopherols.

Nagao et al. studied the improvement process for purification of tocopherols and sterols from the Soybean Oil Deodorizer Distillates (SODD) in the year (2005). The parameter for the study includes a mixture of SODDTC/water (95:5, w/w) with *Candida rugosa* lipase catalyst. It was agitated at 40°C for 24h with dehydration at pressure 20mmHg. The next step reaction was then conducted at 30°C for 24 h. It was noted that tocopherols purified to 72% with a yield of 88% recovery whereas the steryl ester purified up to 97% respectively.

Mendes et al., (2005) studied the concentration of vitamin E from soybean oil deodorizer distillate (SODD) by using the supercritical CO<sub>2</sub> as a solvent. The parameter for the study includes the separation of tocopherol from squalene and linoleic acid at the temperature range 40, 60, and 80°C and pressure from 90 to 350bar. The best results for the concentration was at pressure 150 bar and temperature of 40°C respectively. It was noted that the efficiency and concentration factor decreases with the increase in temperature. The result was promised for the implementation of industrial supercritical extraction because of the high level of vitamin E without fatty acids.

In the year (2004), Fang et al., studied the alpha-tocopherol concentration from the oil by-product by using methanol and CO<sub>2</sub>. The parameter for the study includes the DOD,

methyl esterification and methanolysis. The fatty acid and glycerides converted into fatty acid methyl esters (FAMES) at a temperature (250-300°C) for the period (20-30 min) with the existence of methanol (5/3 v/v DOD). The reactions (esterification and methanolysis) are separately carried out by the catalytic difference. Such process cause problem like energy consumption, long period of time, and the waste-water obtained during the extraction. It was noted that the supercritical methanol leads into more FAMES and sterols than the conventional pretreatment and the novel process was simplest and more effective than the conventional pretreatment method.

Nogala et al., worked on the evaluation of tocopherols content and antioxidant capacity from distillate obtained after refining of edible oils (rapeseed, soybean, and sunflower oil) in the year (2004). The parameter for the study includes the elimination of majority matrix from deodorizing distillate by freezing with an acetone solution at - 70°C. The antioxidant activity was investigated and observed by the peroxide value at 25°C temperature using the oxidograph test. It was noted that the tocopherol concentration of plant oils (Natural tocopherols concentrate obtain from oils) are good food antioxidants and increases the biological and nutritional value of food against synthetic tocopherols.

In the year (2003), Buczenko et al., carried out research work on extraction of tocopherols from the deodorize distillate of soybean oil with liquefied petroleum gas. The parameter for the study includes the separation of tocopherols and sterols by using liquefied petroleum gas (LPG) extraction method. LPG was used as a solvent to improve extract recovery and prevent the tocopherol degradations. It was noted that tocopherols extraction significantly improved at a temperature below 0°C. The result obtained using LPG at - 81°C with yield a separation factor of 11.6 and selectivity of 2.9. The extract (tocopherol) was easily separated from LPG through the exhaust valve of the extractor in the extraction process.

In the year (2003), Nagesha et al., studied about the selectivity of membrane (a nonporous denser polymeric membrane) for tocopherol and fatty acid (FA) system. The parameter for the study includes the selectivity of the membrane for tocopherols improved with esterified soya DOD. The presence of FAMES decreased the viscosity of the feed and thereby increased convective flow, which in turn improved permeates flux. It was noted that the separation in the membrane is generally based on a solution-diffusion mechanism, tocopherol concentration contained 71.2% of total tocopherols with other lipid components (mainly unsaponifiables, includes TG).

In the year (2002), Mendes et al., studied an economic aspect based on an experimental study of vitamin E concentration present in deodorizer distillate of soybean oil. The soybean sludge was used for vitamin E extraction at operational condition of 40, 60, and 80°C temperature and the pressure at 90 and 170bar. The sampling in the extractor

was done with the micrometric valve. It was noted that the tocopherols were concentrated in the extractor and fatty acid was extracted during the process. The process was economically and technically viable. The tocopherols were concentrated at least 40% from a natural source. A better efficiency of the process was obtained at the lower condition of pressure and temperature.

Shimada et al., (2000) studied facile purification of tocopherol from soybean oil deodorizer distillate by the catalytic (lipase) reaction. The parameter for the study includes the deodorization steps of vegetable oil by molecular distillation (MD). A mixture of SODDTC and water (4:1, w/w) was stirred at 35°C for 24hr. with 200U of Candida lipase per 1gm of the reaction mixture under optimum condition. It was noted that more than 95% of the sterols was esterified in the purification. The resulted reaction mixture was fractionated. The distillate fraction contains about 65 wt% of tocopherols with low contents of FFA and sterols.

Gimeno et al., studied the simple method for extraction of tocopherols ( $\alpha$ -,  $\beta$  +  $\gamma$ , and  $\delta$ -tocopherol) available in vegetable oils in the year (2000). The oil was diluted with hexane and an aliquot was mixed with ethanol containing an Internal Standards ( $\alpha$ - tocopherol acetate). Tocopherols were detected at 292nm in less than 5 min after injection. It was observed that this method precise (with RSD = 2.69 %), and had a high mean recovery found 98.14% of tocopherols.

In the year (2000), Chang et al., studied the supercritical carbon dioxide (SF-CO<sub>2</sub>) extraction for high-value substances (free fatty acids, tocopherols, sterols, squalene) from soybean oil deodorizer distillate. The parameter for the study includes the temperature (50-90°C) and pressure (24.1-31.0MPa). The Reverse-phase high-performance liquid chromatography was performed in the valuable components for the extracts after the deodorization. The experimental results showed that when tocopherol was extracted at 31.0MPa, 90 (top) to 70 (bottom) °C, and 1000-L usage, the recovery reached 83.6% and the average value of the concentration factor was 1.38.

Regina B. et al., (1999) worked on the extraction of vitamin E, its function and metabolism. The framework of the study includes summarized knowledge on the function of vitamin E, with emphasis on its antioxidants vs. other properties like the preference of the organism for RRR- $\alpha$ -tocopherol and its metabolism to CEHCs. It was noted that the possibility of vitamin E has ameliorative effect in chronic disease has spurred interest in its specific molecular function and whether these are related to its antioxidant functions, the importance of tocopherol in cell signaling its recognition by the  $\alpha$ -tocopherol transfer protein and metabolism. The relationship to human vitamin E deficiency, normal metabolism and chronic diseases was also discussed.

Ramamurthi et al., (1993) studied the separation of sterols and tocopherols from modified fatty acids in deodorizer distillate (canola, mixed, and soya deodorizer distillate)

through the lipase-catalyzed reaction. The parameter for the study includes fatty acid esterification with methanol catalyst. The conversion of the fatty acid to methyl ester in 5hrs was 96.5, 83.5 and 89.4% respectively. Simple vacuum distillation (1-2 mmHg) was employed to remove the volatile fraction of the esters and tocopherols. It was noted that sterols and tocopherols were retained in the residue fraction with recoveries in the range of 95% and tocopherols recovered after the esterification.

Snyder et al. studied the analysis of tocopherols in vitamin E containing mixtures by the chromatography and spectrometry method in the year (1993). A gas chromatography (GC) and supercritical fluid chromatography (SFC) was used. The SFC analysis conditions were optimized with respect to column type and density/pressure programming. A density program applied 0.20g/ml to 0.66g/ml in programming rate 0.002 g/ml/min to produce an excellent separation of the tocopherols and phytosterols at 120°C column temperature. It was noted that both (SFC and GC) were separated qualitatively and quantitatively.

Lee et al., (1991) studied the concentration of tocopherols from soybean sludge by the supercritical fluid extraction. The parameter for the study includes carbon-dioxide range from 35 to 70°C temperature and 200 to 400 bar pressure. The supercritical solubility of the esterified soybean sludge was 4- 6 times greater than that of original soybean sludge. Overall soybean sludge initially contained about 13-17wt% of tocopherols. A batch type one stage method was used for the separation of tocopherols from esterified soybean sludge. The recovery of 10%  $\alpha$ -tocopherol, 63.2%  $\gamma$ -tocopherol, 26.8%  $\delta$ -tocopherol and the trace amount of  $\beta$ -tocopherol was noted.

Warner et al., studied the analysis of tocopherols and phytosterols in vegetable oils for separation, detection, and quantification of the tocopherols ( $\alpha$ -,  $\beta$ -,  $\gamma$ -, and  $\delta$ -tocopherol) and phytosterols in the year (1990). The framework of study includes the analysis of soybean, sunflower, low-erucic acid rapeseed (LEAR) and corn oils by High-Performance Liquid Chromatography (HPLC) with an evaporative light scatterings detector was used. It was noted that edible oils contained a low level of  $\alpha$ -tocopherols, from 12 to 20 mg/100g oil, whereas sunflower oil contained an average of 61 mg/100g oil which was 92% of the total tocopherol. Whereas the  $\beta$ -tocopherol present in the small amounts in soybean, sunflower and corn oil. The soybean oil had the greatest amount of  $\gamma$ - tocopherols and soybean had a higher level of total tocopherols than the other oil with a mean of 100 mg/100 g oil.

Charles Marks, (1988), evaluated the percentage of free tocopherols in deodorizer distillate by chromatography method for the elimination of interferences and reduce labor time. The parameter in this study includes the composition of sterols, steryl esters, sterol hydrocarbons, tocopheryl esters, free fatty acids, tocopherol etc were 2-15% in deodorizer distillation. It was noted that no interference

found with any of the tocopherols or the internal standard, no saponification was necessary. The time for complete analysis was less than one hour. The Packed Column Chromatography (PCC) showed an increase in the tocopherol content due to the presence of esters.

In the year (1970), Nelson et al., studied the determination of tocopherols and sterols in soya sludge and residues by the gas chromatography an improved method. The objective of the study includes the analysis or saponification of the sample by extraction. Several significant modifications conversion of tocopherols and sterols to esters were conducted resulting in precision and accuracy. It was noted that the old method does not resolve completely the tocopherols peak from small extraneous peak during the gas chromatography. The new method was displaced from all interfering and unresolved peaks with resultant improvement in accuracy.

Conclusion – Edible oil is one of the important gradient in food preparation. The edible oil undergoes deterioration in the physical and chemical characteristics during storage. Vitamin E present in the edible oil increase the oxidative stability. There are number of methods available in the literature for extraction of vitamin E like molecular distillation, adsorption, supercritical fluid extraction, extraction with different solvent etc. All the methods have got their own limitations and benefits. Soya oil is the one of the best source of vitamin E. Used edible oil also contained appreciable amount of vitamin E. Total tocopherol content decrease with the number of refining stage during refining of edible oil. The method includes catalytic decomposition followed by crystallization with high purity of vitamin E. From the literature it has been concluded that used oil and SODD may be one of the important raw material for extraction and recovery of vitamin E.

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