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Journal of Lipid Science and Technology

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CONTENTS

- 05 **Stability Studies of β -Carotene in Peanut and Olive Oils**
K. Balaswamy, K Sathiya Mala, G Sulochanamma, G Narsing Rao and P G Prabhakara Rao
- 15 **Study of Erucic Acid-based Polyol Esters as Industrial Gear Oils**
Korlipara V. Padmaja, Venkateshwarlu Kontham, Amit Kumar Rajak and Saravanan Krishnasamy
- 20 **A Short Communication**
Sunit Mukherji

NEWS & GUIDELINES SECTION

- 22 **Research Roundup**
- 26 **Guidelines for Authors**
- 32 **Membership Form**

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From Editors Desk



Hello friends,

As we all know that oleochemicals is one of the most emerging field as a partial replacement of petroleum applications. It has found its applications in various domains such as surfactant, soap, detergent, food additives, bio lubricants, bio surfactant, and biopolymers with total estimated market of 14 MMT in FY13. The growth rate has been found at a CAGR of 6% which makes the oleochemicals market of 18.1 MMT in FY2018. The chemical industry like Solvay has planned to make their development sustainable by increasing the share of raw materials through bio sources from 11% in 2010 to 20% in 2020. Fatty acids contribute

around 88% of total consumed materials in oleochemicals and out of which 72% is used in soaps industries. The demand of oleochemicals excluding soaps is expected to grow at a CGAR of 10% over FY13 to FY20 which makes it market from 268, 000 MT to 524, 000 MT. Diversification of products portfolio makes industries not only profitably viable but also reduces the risk of carbon footprints.

A radical change has been observed in oleochemicals production scenario – two decades ago, ~90% of all oleochemicals were being produced in developed countries such as Europe, Japan and USA while in current scenario Asia holds 60% of total oleochemicals production and Asians consume around 68% of world consumption. Palm oil and palm kernel oil are mostly produced in Asian country like Indonesia and Malaysia and India is the third largest producer of mustard seeds and largest for castor beans. This opens a new door for chemical industries to explore their downstream manufacturing to cater the entire value chain. Indian companies have already taken this initiative in agrochemicals and have been quite successful. This can be explored for development of country, business and for better and safe environment by combined effort of industries and government bodies.

India is one of the large importing country for edible oil commodity and biodiesel, oleochemicals and food share in total edible oils consumption is 12%, 12% and 76%, respectively. Government regulation for blending of biodiesel in petroleum products can put a serious challenge for industries and it has seminal effect of price of edible oils. However, cultivation of palm oil and some of non-edible oils such as Jatropha, Algae can be promoted through government policy so that a synergized solution can be seen by Indian industries of oleochemicals and edibles oils. Though the policy for palm promotion exist for last so many years but it effective implementation is not here. Thus, the ground situation is very decimals. Promotion of non-edible ils for oleochemicals can also fulfil the additional requirement of olechemicals (8 MT in 2020).

(R.P. SINGH)

INDEX TO ADVERTISEMENTS / ANNOUNCEMENTS

ADVERTISEMENTS / ANNOUNCEMENTS	Page
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Stability Studies of β -Carotene in Peanut and Olive Oils

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Key words : β -carotene, pumpkin flour oil, fortification, peanut oil, olive oil, HPLC, GC-GCMS

ABSTRACT

The present study deals with the extraction of β -carotene from pumpkin and evaluation for stability in different vegetable oils. The pumpkin flour oil (PFO) extracted from pumpkin flour (PF) was rich in β -carotene (3.4%) when estimated with HPLC. The PFO contained 47% polyunsaturated fatty acids and 53% saturated and monounsaturated fatty acids during analysis by GC-GCMS. The PFO was used as a source of β -carotene in selected vegetable oils namely peanut and olive oil. The stability of β -carotene in fortified oils was studied at ambient temperature and accelerated storage conditions for 30, 15 days respectively. Degradation of β -carotene was found to be lower in olive oil exposed to the ambient conditions compared to peanut oil exposed to both storage conditions. Similarly, quality characteristics of olive oil exposed to the ambient temperature concerning free fatty acids and peroxide values were lower than fortified peanut oil samples.

INTRODUCTION

Pumpkin belonging to the family Cucurbitaceae is a widely grown vegetable all over the world. Pumpkin possesses a long storage life and high nutritive value comprising components such as β -carotene, vitamins, minerals and dietary fiber. Indian cultivars of pumpkin have been reported to contain β -carotene in between 132 to 527 mg/100g (db) (Gopal Krishnan et al., 1980)¹. A variety of food products such as jam, jelly, marmalade, puree, sauce, chutney, pickle, halwa etc. have been prepared from pumpkin (Dhiman et al. 2009)². The dried pumpkin flour is being applied as a supplement for β -carotene and as a natural dark yellow-orange colorant in various food products (Pongjanta et al., 2003)³.

Noodles made by replacing with 5% pumpkin flour resulted in an attractive yellow colour, good appearance, pleasant taste and texture (Lee et al. 2002)⁴. A shelf-stable pumpkin candy containing β -carotene (8.85 mg/100 g) was found to possess high antioxidant activity (Muzzaffar et al., 2016)⁵.

β -Carotene, a precursor to vitamin A, is fat soluble and easily absorbed into the body. The human body converts β -carotene into vitamin A is essential to maintain healthy skin, mucus membrane, immune system and good eye health. β -Carotene is used to colour vegetable oil products, such as margarine or buttery oils, bakery shortening, French fry or pan-fry oil, and popcorn oil at 2 - 132 ppm levels (Klaui and Bauernfeind, 1981)⁶. Rodriguez Amaya (2016)⁷ highlighted the carotenoids being highly unsaturated are susceptible to isomerisation and oxidation during processing and preservation of foods. The increase in carotenoid degradation is known with the destruction of food cellular structure, increase of surface area, processing conditions such as temperature and time, and storage conditions such as light and oxygen. It was observed, cooking causes higher losses in carotenoid content than commercial sterilization during preparing puree (Provesi et al., 2011)⁸. Retention of all trans β -carotene was higher (>75%) after processing and storage for 180 days compared to other xanthophylls. β -Carotene acts as an antioxidant and protects lipids from free radical autoxidation by reacting with peroxy radicals, thereby inhibiting polymerization reaction (Burton and Ingold, 1984; Palozza and Krinsky, 1991; Bradley and Min, 1992)⁹⁻¹¹. β -Carotene containing eleven double bonds also acts an efficient singlet oxygen quencher during inhibition of

photooxidation by transfer of energy from singlet oxygen to β -carotene (Foote and Denny, 1968)¹².

Literature is available on the utilization of pumpkin in different products and its β -carotene to improve the colour of foods. Studies on the β -carotene fortification of oils using pumpkin oil and their stability during storage are scanty. The present study deals with the extraction of fat from pumpkin flour and its application in selected vegetable oils such as peanut and olive oils to study the stability of β -carotene during ambient and accelerated storage conditions for further application in food products.

MATERIALS AND METHODS

Materials

Fully mature, green coloured skin pumpkin variety (*Cucurbita maxima*) rich in β -carotene was procured from Bowenpally vegetable market, Hyderabad. The oil samples such as double filtered peanut oil manufactured by Ushodaya Enterprise Private Limited, Hyderabad (March, 2016); and olive oil produced by Deoleo S.A., Spain (January, 2016) were procured from local supermarket, Tarnaka, Hyderabad. The chemicals were of analytical grade were purchased from M/s. Sd. Fine Chem., Mumbai, India. Standard β -carotene was purchased from Sigma-Aldrich Bangalore, India.

Preparation of pumpkin flour

A standard method was followed for the preparation of pumpkin flour (Sathiya Mala et al., 2016)¹³. Thoroughly washed pumpkins were peeled and cut into longitudinal slices and passed through a vegetable slicer to obtain fine shreds. Pumpkin shreds were blanched in hot water ($95 \text{ C} \pm 2$) for 2 min, followed by cooling in water. Then the shreds were dipped in 0.2% potassium metabisulphite solution for 45 min maintaining the shreds to solution ratio of 1: 1.5. They were dried in a cabinet tray dryer at $55 \pm 2^\circ\text{C}$ for 8 h. Dried shreds were ground using impact pulverizer

(Pilotsmith India, Kerala, India), sieved through BS 72 mesh to obtain pumpkin flour and stored in metalized polyester polyethylene pouches until further use.

Extraction of pumpkin flour oil

Pumpkin flour (500 g) was extracted with hexane using a soxhlet apparatus. The solvent was recovered using a rotavapor (Aditya Scientific Co, Hyderabad, India) at 45°C and the resultant pumpkin flour oil (PFO) was stored at refrigerated conditions for further experiments. β -Carotene content of PFO is estimated by a high performance liquid chromatographic (HPLC) method (Chidambara Murthy, 2005)¹⁴.

Preparation of fortified vegetable oil samples

The two vegetable oil samples were thoroughly mixed with calculated quantities of PFO to maintain different concentrations of β -carotene such as 30, 60, 80, 100 and 120 ppm. The oil samples (10 g) were stored in 20 ml glass test tubes and exposed to different storage conditions. One set of oil samples were stored at ambient conditions ($30 \pm 3^\circ\text{C}$) and analyzed for β -carotene after 0, 7, 14, 21 and 30 days. Another set of oil samples were exposed to accelerated conditions ($60 \pm 2^\circ\text{C}$) in an oven and analyzed after 0, 3, 6, 9, 12 and 15 days. The oil samples without fortification were also exposed as control to the above conditions.

Fatty acid composition

The oil samples and PFO were analyzed initially for fatty acid composition by preparing fatty acid methyl esters (FAMES) and determined by using Gas Chromatography and Gas Chromatography - Mass Spectrometry (GC-GCMS). The FAMES were prepared by refluxing the oil sample with 2% sulphuric acid in methanol (Christie, 1993)¹⁵. The FAMES were extracted with ethyl acetate, thoroughly washed with water and passed through anhydrous sodium sulphate. The dried esters were analyzed for fatty acid composition in GC and GC-MS.

Analysis of fatty acid profile

The GC-FID analyses were performed with an Agilent (Agilent Technologies, Palo Alto, CA, USA) 6850 series GC equipped with a FID detector using a DB-225 capillary column (30 m × 0.25 mm, 0.25 µm of film thickness). The column temperature was initially kept at 160 °C for 2 min, increased to 220 °C at 5 °C/min and maintained for 10 min at 220 °C. The carrier gas, nitrogen was used at a flow rate of 1.5 ml/min. The injector and detector temperatures were maintained at 230 and 250 °C, respectively with a split ratio of 50:1. Identification of the fatty acids was carried out by comparison of retention times with those of standard reference FAMES.

The GCMS analyses were performed using an Agilent (Palo Alto, USA) 6890N gas chromatograph equipped with an HP-5 MS capillary column (30 m × 0.25 mm i.d.). An Agilent 5973 mass spectrometer operating in the EI mode (70 eV; m/z 50 – 550; source temperature 230 °C and a quadrupole temperature 150 °C) was used. The column temperature was initially maintained at 200 °C for 2 min, increased to 300 °C at 4 °C/min, and kept for 20 min at 300 °C. The carrier gas, helium was used at a flow rate of 1.0 ml/min. The inlet temperature was maintained at 300 °C and the split ratio of 50:1. Structural assignments were based on interpretation of mass spectrometric fragmentation and confirmed by comparison of retention times as well as fragmentation pattern of authentic compounds and the spectral data obtained from the Wiley and NIST libraries.

Free fatty acid content

The free fatty acid (FFA) content was determined by a titrimetric method with a standard alkali and expressed as percent oleic acid (Ranganna, 1986)¹⁶. The oil sample (2 g) was taken into a 250 ml Erlenmeyer flask, and 50 ml petroleum ether was added. The contents were thoroughly swirled until the sample

dissolved. To the contents, 50 ml neutral ethyl alcohol was added and titrated with 0.1 N KOH solution in the presence of phenolphthalein indicator (1 % in 95 % ethyl alcohol) till persisting pink colour appeared.

Peroxide value

The peroxide values (PV) of the oil samples were determined by iodometric titration and expressed as Meq O₂/kg (Ranganna, 1986)¹⁶. The oil sample was dissolved in a solvent mixture (2 volumes of glacial acetic acid and one volume of chloroform), treated with potassium iodide, and the iodine liberated by the peroxides present in oil was titrated with standard sodium thiosulphate solution.

Hunter Lab Colour values

The fortified oil samples were periodically subjected to color measurement using HunterLab Colorimeter (Model Ultrascan, Hunter Associates Laboratory, USA) wherein the three colour coordinates, namely *L**, *a** and *b**, represents the lightness index, red-green and yellow-blue colour components (Hun, 1991)¹⁷.

β-Carotene

Standard β-carotene (1mg/ml) was prepared in hexane and stored in amber coloured volumetric flask. The solution was further diluted with hexane (1 ml/200 ml) and a volume of 10 µl was injected. The oil sample (0.5 g) was dissolved in 5 ml of hexane and 20 µl was injected into the HPLC system. The HPLC system (Shimadzu prominence LC-20AD, Japan) consisted of binary gradient fitted with an Ultra Restek HPLC C18 analytical column (25 cm x 4.6 mm ID) 5 µ particle size. Detection was carried out by SPD 20A series variable wavelength detector at a wavelength of 450 nm. The gradient mobile phase consisted of acetonitrile and chloroform (80:20) with a flow rate of 1ml/min for 25 min and β-carotene was quantified using LC solution software. The elution program involved a linear gradient from 80 to

20% of acetonitrile for 0-5 min and 20 to 80% of chloroform from 5-15 min and again 80% of acetonitrile for 15-20 min followed by 5 min equilibrium. The β -carotene was identified and calculated by comparing retention time of the sample peak with the standard.

Statistical Analysis

The fortified samples and control vegetable oils were periodically drawn from experimental storage conditions and evaluated for FFA, PV, colour and β -carotene. Mean values of duplicate analysis of fatty acid composition are presented. Analyses were carried out in triplicate for β -carotene, FFA, PV, colour and mean values along with standard deviation (SD) are presented. The values were treated for ANOVA for significance at $P < 0.05$.

RESULTS AND DISCUSSION

Composition of PFO and oil samples

In the present study, blanching the pumpkin slices followed by drying yielded 8.5% PF. The yield of PFO from dehydrated PF by Soxhlet extraction was 2.2%. The results are comparable with earlier finds of Adebayo et al. (2013)¹⁸ who stated that the pulp contained fat extract of 2.3% (db). It is known that blanching process reduces the degradation of β -carotene (Sungpuag et al., 1999)¹⁹. The fresh pumpkin exhibited the total carotenoid content of 12.1 mg/100 g in which 57.8 μ g/100 g β -carotene was estimated (Norshazila et al. 2012)²⁰. GC, GC-MS analysis revealed the fatty acid composition of peanut, olive and pumpkin flour oil (Table 1). Palmitic acid is a major fatty acid in saturated fatty acids in both vegetable oils. The presence of monounsaturated fatty acids (MUFA) is higher in olive oil (68.20%), and polyunsaturated fatty acids (PUFA) are higher in pumpkin flour oil (47.17%). According to Nagaraj (1995)²¹, oils rich in saturated fatty acids, MUFA are more stable that can be stored for longer period and the oils containing equal quantities of saturated, MUFA and PUFA were termed as healthy. A typical HPLC

chromatogram for standard β -carotene in Fig. 1 a and chromatogram for PFO in Fig. 1 b were presented. The other major peak could be due to presence of lutein or α -carotene which was ignored in the study. HPLC analysis of the other peanut and olive oils showed traces of β -carotene. The β -carotene content (3.29 g/100 g) was unchanged in PFO during the storage of four months at refrigeration conditions. However, β -carotene content in PFO decreased to 2.14 mg/100 g after storage of 8 months. The fresh PFO is used as a source of β -carotene in these experiments and fortified into oils at various concentrations of 30, 60, 80, 100 and 120 ppm levels.

Changes in FFA and PV during storage

The changes in free fatty acid content were measured as oleic acid (Table 2). The initial values of FFA in peanut oil and olive oils were 1.07 and 0.14% respectively at 0 days. No changes were observed in control oils. The fortified peanut oil showed increase in FFA values to maximum levels of 2.4 and 4.8 at ambient and accelerated conditions. Similarly, fortified olive oil exhibited increase in FFA values to maximum levels of 0.19 and 3.4 at ambient and accelerated conditions. The results reveal that olive oil is stable at ambient conditions for 30 days when β -carotene is added. However, a significant increase in acidity values were recorded in extra virgin olive oil stored at 30 °C for a storage period of 6 months (Gutierrez and Fernandez, 2002)²².

The changes in peroxide value were determined and expressed as Meq O₂/kg oil (Table 3). Initial peroxide values of peanut oil and olive oil were of 4.81 and 5.35 at 0 days. In control samples, PV increased to 13.18 and 26.03 at ambient conditions. Similarly, PV of peanut oil and olive oil increased to 69.58 and 43.62 respectively at accelerated conditions. In samples fortified with PFO, the peroxide values increased to 12.9 and 19.5 Meq O₂/kg respectively for peanut and olive oils at ambient conditions. The samples stored at accelerated conditions PV increased to 59.5 and 42.3 Meq

O₂/kg respectively for peanut and olive oils. The results are comparable with earlier findings (Goulson and Warthesen, 1999)²³.

Changes in HunterLab colour values during storage

Changes in Hunter Lab colour from 0 days to 30 days at ambient conditions and 15 days at accelerated conditions were presented Table 4. In the present study, storage at ambient conditions did not affect the L* and b* colour units. Accelerated conditions (60 °C) showed significant decrease in red colour units (a*) in both peanut and olive oils, which might be due to degradation of β -carotene. Dutta et al. (2006)²⁴ described the degradation kinetics of β -carotene and colour during processing of pumpkin puree which followed the first-order reaction for temperature dependence. Commercially, a thermo stable dark red coloured sunflower oil formulation containing 30% β -carotene (Altratene 30% OS-SF) with shelf life of 36 months is available in the market (Anon 2006)²⁵. The present study favours the fortification olive oil with β -carotene for preservation at ambient conditions without changes in red colour units.

Changes in β -carotene during storage

The percent changes in β -carotene content in fortified peanut and olive oils exposed to ambient condition during a period of 30 days are presented in Fig. 2 a. Minor changes were observed (2%) in all exposures up to 7 days. A significant loss was found in peanut oil (28.1%) with 120 ppm and in olive oil (17.5%) was noticed in different concentrations (30-120 ppm) after a storage period of 30 days. Earlier studies indicated a complete loss of carotene within 14 days in refined peanut oil fortified with 500 ppm carotene when stored at 37 °C (McDonald, 1933)²⁶. Fig. 2 b presents the data on changes in β -carotene content in peanut and olive oil exposed to accelerated conditions during a period of 15 days. Significant decrease in β -carotene (68%) was seen in oil samples when fortified with higher concentration of β -carotene and exposed to accelerated conditions after 15

days period. A minimum loss of 50% was observed in olive oil fortified with 30 ppm β -carotene was observed. The losses were lower in olive oil compared to peanut oil. The results indicate that olive oil appears to be better medium for β -carotene preservation at room temperature for a longer period. The fatty acid composition of olive oil containing higher amounts of MUFA and lower quantities of PUFA might be responsible for the preservation of β -carotene by increasing oxidative stability. The stability studies of standard β -carotene in canola oils indicated a minimum of 16.2 - 18.2% loss when exposed to photooxidation at 30 °C for 21 days and higher losses to the extent of 66% when exposed to 60 °C after 12 days storage period (Goulson and Warthesen, 1999)²³. The formation of 9-cis- β -carotene is an indication of thermal degradation of β -carotene in palm olein and copra oils during storage and processing (Achir et al., 2011)²⁷. Earlier, a shelf-stable formulation with virgin coconut oil containing 75% of saturated fatty acids enriched with β -carotene (24 ppm) and Vitamin D3 was produced as a value added functional oil (Arumugam et al., 2014)²⁸.

CONCLUSIONS

Pumpkin flour oil rich in β -carotene is extracted from dehydrated pumpkin. The flour oil contains 47% PUFA and 53% of MUFA and saturated fatty acids. The oil is rich in β -carotene (3.3%) fortified with olive oil was found to be stable at ambient temperature storage and the data can be beneficial for the preparation of formulations for vitamin A supplementation for required population and further application in food products.

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TABLE 1
Fatty Acid Composition of Peanut Oil, Olive Oil and Pumpkin Flour Oil

Fatty acid	% composition		
	Peanut oil	Olive oil	Pumpkin flour oil
14:0(Myristic)	-	-	0.90
16:0(Palmitic)	12.60	14.90	23.32
18:0 (Stearic)	4.00	2.60	3.80
20:0 (Arachidic)	1.90	0.40	0.30
22:0 (Behenic)	4.50	-	0.20
24:0 (Lingoceric)	1.70	-	-
Saturated fatty acids (SFA)	24.70	17.90	27.62
16:1 (Palmitoleic)	0.01	1.60	0.10
18:1 (Oleic)	41.80	66.40	23.20
20:1 (Eicosenoic)	1.40	0.20	0.90
Mono unsaturated fatty acids (MUFA)	43.20	68.20	24.20
18:2 (Linoleic)	31.90	13.30	19.10
18:3 (γ-Linolenic acid, GLA)	0.10	0.60	3.06
18:3 (α-Linolenic acid, ALA)	-	-	25.01
Polyunsaturated fatty acids (PUFA)	32.00	13.90	47.17

TABLE 2
Changes in Free Fatty Acids in β-Carotene Enriched Vegetable Oils Exposed to Different Storage Conditions

Storage condition	Storage period (Days)	Peanut oil [1.07]					Olive oil [0.14]				
		Added β-carotene (ppm)					Added β-carotene (ppm)				
		30	60	80	100	120					
Ambient (30 ± 3 °C)	7	1.73	1.76	2.33	2.25	2.20	0.15	0.18	0.16	0.18	0.18
	14	1.82	1.70	1.84	1.21	1.71	0.18	0.18	0.16	0.18	0.18
	21	2.15	2.07	2.15	2.22	2.14	0.18	0.18	0.18	0.19	0.18
	30	2.24	2.13	2.21	2.39	2.22	0.18	0.19	0.18	0.19	0.18
Accelerated (60 ± 2 °C)	3	1.23	2.03	2.37	1.32	2.12	0.27	0.50	0.26	0.29	0.32
	6	1.43	2.38	2.42	1.73	2.24	0.30	0.78	0.32	0.40	0.37
	9	2.42	2.48	2.94	2.31	2.26	0.37	0.92	0.48	0.66	1.32
	12	2.38	4.30	3.18	4.19	3.89	1.24	1.39	2.75	2.13	2.59
	15	2.45	4.60	3.26	4.78	4.26	1.94	1.69	3.32	2.99	3.37

Values shown in parenthesis indicate the initial values(o days) Standard deviation (SD) values are in the range of 0.01 to 0.05 Values are significantly different at P 0.05 compare to 0 days

Values are mean of duplicate analyses

TABLE 3
Changes in Peroxide Values in β -Carotene Enriched Vegetable Oils Exposed to Different Storage Conditions

Storage condition	Storage period (Days)	Peanut oil [4.81]					Olive oil [5.35]				
		Added β -carotene (ppm)					Added β -carotene (ppm)				
		30	60	80	100	120	30	60	80	100	120
Ambient (30 \pm 3 $^{\circ}$ C)	7	9.92	11.39	11.60	11.12	11.38	11.32	12.14	12.82	15.23	15.50
	14	10.93	11.79	11.72	11.22	12.30	12.52	12.46	14.11	16.19	16.24
	21	11.19	11.95	11.84	12.52	12.58	12.91	13.92	15.11	17.24	17.71
	30	12.60	12.00	11.96	12.74	12.90	13.16	15.45	15.42	19.54	19.56
Accelerated (60 \pm 2 $^{\circ}$ C)	3	14.33	14.32	14.61	14.97	16.42	18.29	21.12	17.07	18.03	15.71
	6	17.40	18.36	17.50	18.68	21.32	19.75	21.28	19.98	20.85	20.24
	9	35.79	34.65	32.42	35.09	31.36	36.55	33.72	32.97	34.02	31.91
	12	38.71	41.42	36.49	35.09	45.14	37.15	36.45	36.83	32.00	32.29
	15	39.33	48.11	57.11	52.32	59.51	39.79	39.40	42.35	37.73	42.24

Value shown in parenthesis indicate the initial values (0 days)

Standard deviation (SD) values are in the range of 0.75 to 2.5

Values are significantly different at $P < 0.05$ compare to 0 days

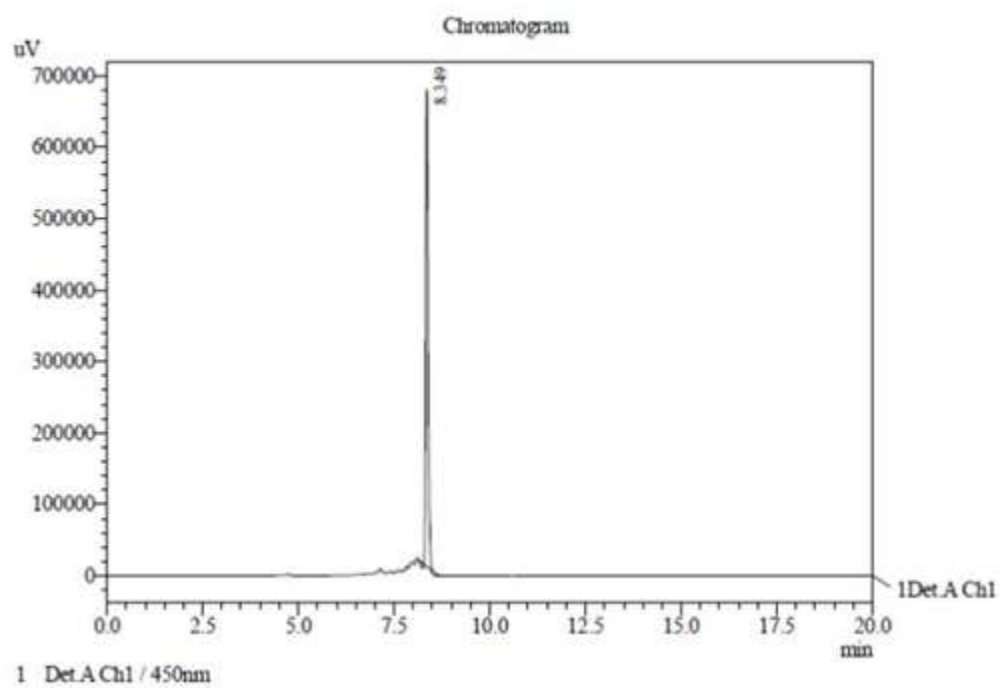
TABLE 4
Changes in Hunterlab Color Values in β -Carotene Enriched Vegetable Oils Exposed to Different Storage Conditions

Storage condition/ period (d)	HunterLab color values	Peanut oil Added β -carotene (ppm)					Olive oil Added β -carotene (ppm)				
		30	60	80	100	120	30	60	80	100	120
0 days	L^*	31.57 \pm 0.02	29.16 \pm 0.09	27.66 \pm 0.02	27.91 \pm 0.08	28.85 \pm 0.03	30.64 \pm .04	29.62 \pm .04	28.77 \pm 0.02	27.51 \pm 0.10	27.35 \pm 0.09
	a^*	4.86 \pm 0.06	5.55 \pm 0.03	5.36 \pm 0.12	5.74 \pm 0.09	5.43 \pm 0.24	3.79 \pm 0.02	5.29 \pm 0.04	5.58 \pm 0.12	5.41 \pm 0.23	5.72 \pm 0.02
	b^*	13.86 \pm 0.15	10.83 \pm 0.08	7.91 \pm 0.01	7.45 \pm 0.08	6.61 \pm 0.16	12.76 \pm 0.02	10.86 \pm 0.13	9.46 \pm 0.04	7.41 \pm 0.04	6.83 \pm 0.08
Ambient /30 days	L^*	31.27 \pm 0.08	29.06 \pm 0.01	27.36 \pm 0.03	27.61 \pm 0.16	28.65 \pm 0.12	30.46 \pm 0.09	29.26 \pm 0.12	28.76 \pm 0.10	27.21 \pm 0.02	27.05 \pm 0.07
	a^*	4.80 \pm 0.03	5.47 \pm 0.06	5.06 \pm 0.03	5.64 \pm 0.14	5.33 \pm 0.09	3.69 \pm 0.04	5.09 \pm 0.11	5.38 \pm 0.09 ^j	5.31 \pm 0.06	5.67 \pm 0.08
	b^*	13.66 \pm 0.11	10.43 \pm 0.06	7.41 \pm 0.09	7.19 \pm 0.26	6.31 \pm 0.18	12.62 \pm 0.17	10.5 \pm 0.23	9.16 \pm 0.20	7.21 \pm 0.04	6.73 \pm 0.17
Accelerated /15 days	L^*	31.53 \pm 0.16	29.05 \pm 0.02	27.38 \pm 0.08	27.99 \pm 0.13	28.79 \pm 0.08	28.4 \pm 0.01	27.95 \pm 0.08	27.08 \pm 0.08	26.7 \pm 0.10	26.34 \pm 0.03
	a^*	2.71 \pm 0.04 ^l	3.96 \pm 0.02 ^l	4.29 \pm 0.03 ^l	2.90 \pm 0.13 ^l	2.66 \pm 0.0 ^l	0.59 \pm 0.01 ^l	1.63 \pm 0.04 ^l	2.27 \pm 0.04 ^l	2.62 \pm 0.06 ^l	2.14 \pm 0.01 ^l
	b^*	12.36 \pm 0.24	10.09 \pm 0.04	7.48 \pm 0.15	7.36 \pm 0.28	5.69 \pm 0.27	8.43 \pm 0.03	8.12 \pm 0.13	6.13 \pm 0.18	6.15 \pm 0.17	4.28 \pm 0.12

Values are mean of triplicates \pm Standard deviation (SD)

^l *Indicates significantly difference at $P < 0.05$*

A.



B.

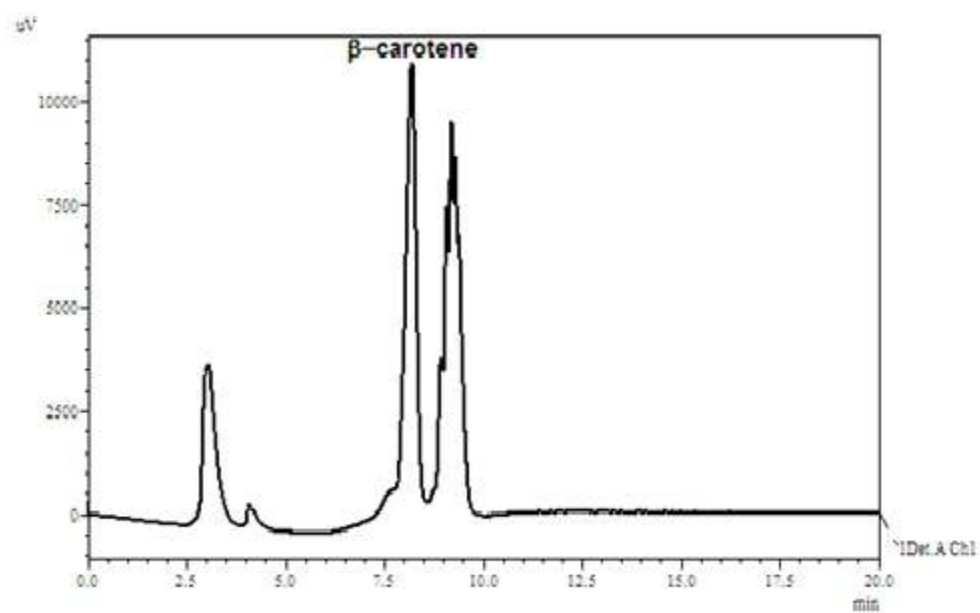
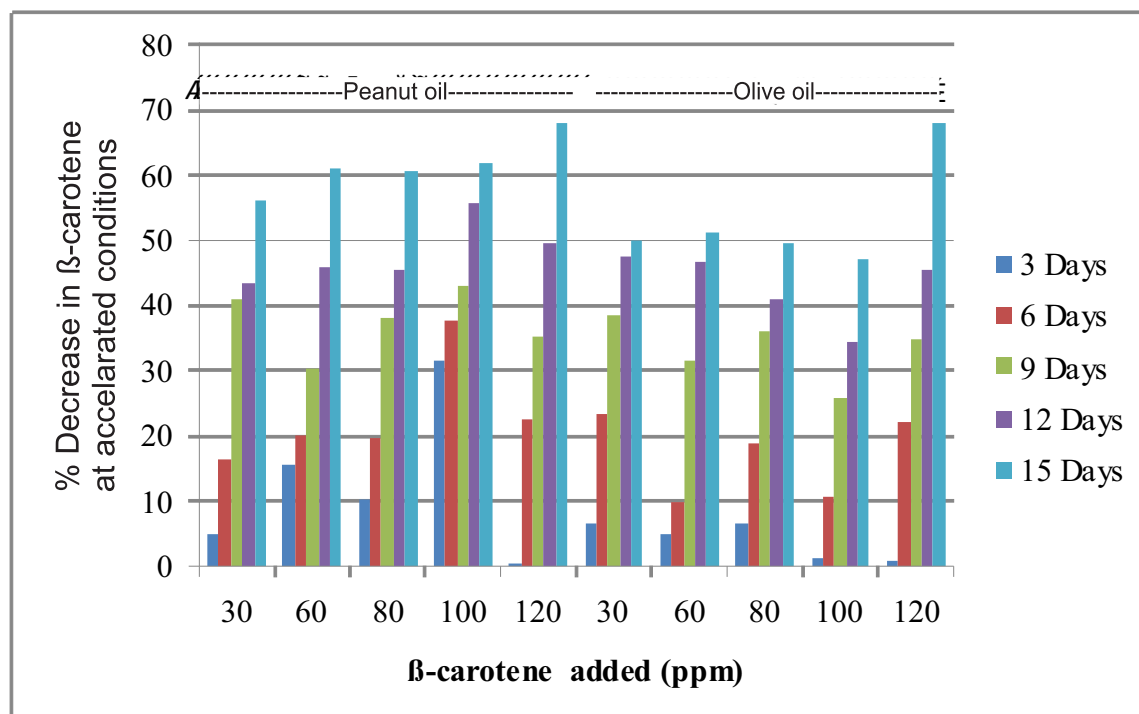
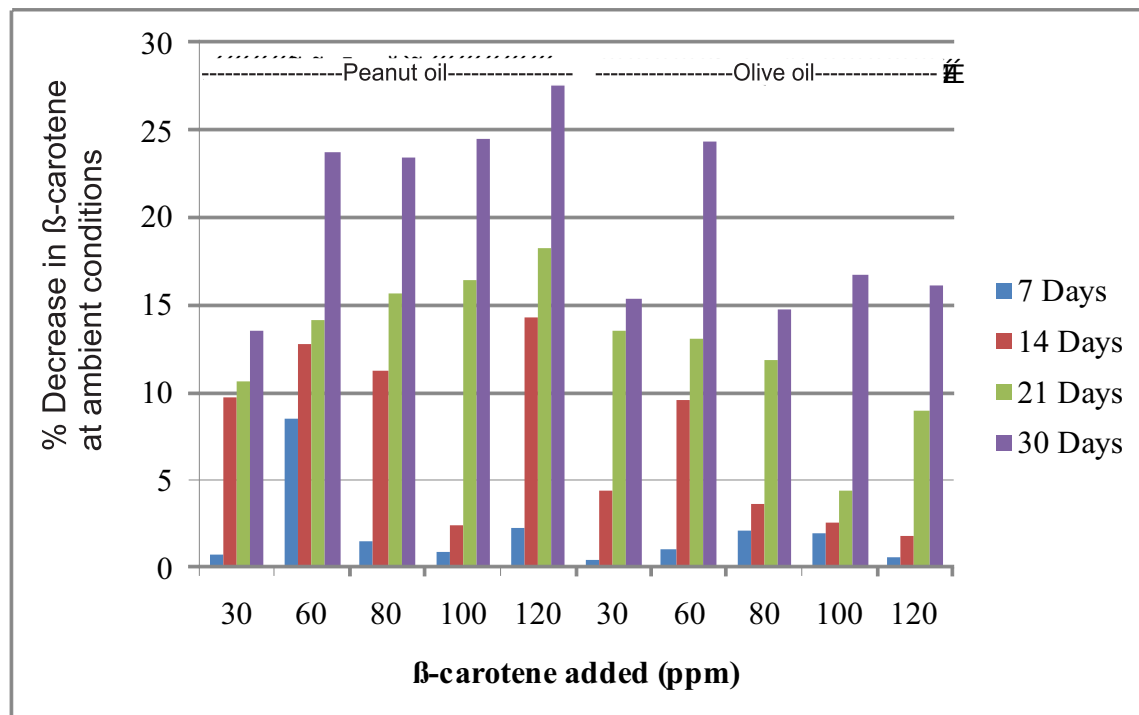


Fig. 1: HPLC profiles of β -carotene:
 (A) Standard β -carotene
 (B) β -carotene in pumpkin flour oil

A.



B.
Fig. 2: % Decrease in β -carotene in peanut and olive oils during storage
(A) ambient conditions ($30 \pm 3^\circ\text{C}$)
(B) accelerated conditions ($60 \pm 2^\circ\text{C}$).

Study of Erucic Acid-based Polyol Esters as Industrial Gear Oils

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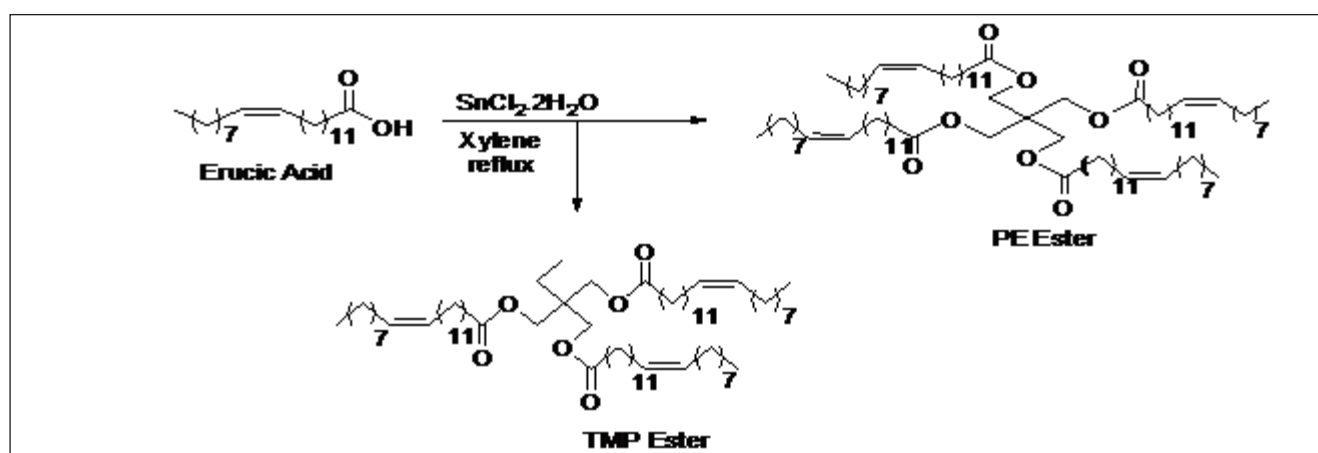
ABSTRACT

The growing environmental concern increased the exploitation of renewable materials for the development of biobased lubricants. Synthetic and vegetable oil-based esters offer the best choice in formulating environmentally friendly lubricants. In the present study, erucic acid-based polyol esters were prepared and characterized using IR, ¹H NMR, ¹³C NMR and mass spectral techniques and evaluated for lubricant properties namely kinematic viscosity, viscosity index, oxidation stability, wear, weld load and copper corrosion value. The products were compared with industrial gear oils of VG-68 and VG-100 grade. The products were found to have good potential for use as basestocks for formulation as extreme pressure type of industrial gear oils.

1. Introduction

The growing environmental protection and energy conservation have driven technologists to substitute sustainable resources to petro-based feed stocks. The use of renewable sources in the manufacturing of industrial products received special attention because they present excellent raw materials of a readily available sustainable resource and constitute a suitable alternative for replacing petrochemical derivatives [Bunemann et al., 2000; Gere and Hazelton 1994; Givens and Michael, 2004]. Agriculturally-derived oils and fats are renewable resources that are biodegradable and are low toxic to environment. Petroleum importing countries can reduce their oil imports by substituting industrial products derived from vegetable oils and fats. Research on oleochemistry indicates

that the use of vegetable oils (VO) allows the development of high performance products, which are both consumer-friendly and environment-friendly. VO possess most of the desirable lubricity properties, such as good contact lubrication, high viscosity index, and low volatility [Mortier and Orszulik 1997; Sraj et al., 2000]. In some applied areas, such as chain saw oil, gearbox oils, hydraulic oils, and lubricants for crude oil production, these oleochemical products are already well established. Vegetable oils may be used for specialized lubricant applications in their unmodified forms. However, to enhance their properties, various types of compatible additives need to be added. Vegetable oils also may be used to develop synthetic esters to overcome their low- and high-temperature limitations. The most natural synthetic esters are the branched polyol esters derived from neopentyl alcohol, trimethylolpropane, and pentaerythritol. These synthetic esters can be derived from combinations of various fatty acids and polyols. The significant feature of these esters is that, there is no hydrogen atom on the β -carbon on the alcohol portion of the polyol molecular structure. This feature gives these esters high degree of oxidative and thermal stability which are rarely found in vegetable oil based lubricants [Bunemann et al., 2000; Gryglewicz et al., 2003; Lawate and Lal 1998; Mckee et al., 2007; Petran et al., 2008; Wilson, 1998]. Hence, the present study is aimed at employing erucic acid, which is a commercially available derivative of rapeseed oil, for the synthesis of polyol esters using TMP (trihydric) and PE (tetrahydric) to assess their potential as base stocks (Scheme 1) for environmentally friendly industrial gear oils.



Scheme1: Synthesis of Erucic acid-based Polyol Esters.

2. Materials and methods

2.1 Materials

Erucic acid was procured from M/s. VVF private limited, Mumbai. TMP, PE, p-TSA, xylene, aluminium oxide active basic, sodium sulphate, were procured from M/s S.D. Fine chemicals Pvt. Ltd., Mumbai, India. All the solvents and reagents were of analytical grade and were used directly without purification.

2.2 Methods

2.2.1 Analysis

^1H NMR spectra were obtained using a Bruker AR X 400 Spectrometer (400, 200 MHz). IR spectra were recorded on a Perkin Elmer (Model: Spectrum BX; Connecticut, USA) FT-IR spectrometer neat as thin film and density by Anton Paar DMA 4500 M density meter. The acid value was determined by AOCS Official Method Cd 3d-63.

2.2.2 Kinematic viscosity and viscosity index (VI)

Viscosity measurements were made at 40 °C and 100°C using calibrated Cannon Fenske viscometer tubes in a Cannon Constant Temperature Viscosity Bath (Cannon Instrument Co., U.S.A.). Viscosity and viscosity index (VI) were calculated using ASTM D 445 and ASTM D 2270 methods, respectively. Measurements were made in duplicate and the average values were reported.

Kinematic Viscosity (cSt) = Viscometer constant (cSt/s) \times time (s)

2.2.3 Flash point

Flash point of the products was determined as per ASTM D 93 method using Tanaka Scientific Ltd., Japan, apparatus. The lowest temperature at which application of the test flame causes the vapour above the surface of the liquid to ignite is taken as the flash point of the product at ambient barometric pressure.

2.2.4 Pour point

The pour points were determined by ASTM D 97 method with an accuracy of ± 3 °C using pour point test apparatus manufactured by Dott Scavini & Co, Italy. All runs were carried out in duplicate. Sample temperature was measured in 3 °C increments at the top of the sample until it stopped pouring.

2.2.5 Copper corrosion

Determination of corrosiveness of the products was done as per ASTM D 130 method using Koehler Inc., U.S.A. apparatus. A polished Cu strip was immersed in 30 mL of the sample being tested at 100 °C for 3 h. After 3 h, the Cu strip is removed, washed and the colour and tarnish level were assessed against the ASTM Copper Strip Corrosion Standard.

2.2.6 Weld load

Weld load of the products was determined by using Four Ball Tester manufactured by Stanhope Seta, U.K. as per IP 239 method.

Out of four balls, three clean balls are placed in the ball cup securely and the fourth ball is placed into the upper ball chuck. The ball cup assembly is positioned centrally under the spindle in contact with the fourth ball. Then the series of loads are applied on the weight pan with an increment of 10 kg and started the motor for a period of 1 minute. The load at which the balls are welded or fused together is taken as the Weld load.

2.2.7 Synthetic methods

Preparation of trimethylolpropane triesters of erucic acid

Trimethylolpropane (16.99g, 0.1267 mol), erucic acid (150g, 0.4437 mol) and xylene (75 mL) were taken in a three necked reaction flask equipped with a thermometer, dean stark apparatus and a condenser. The contents were stirred at 135-140 C in presence of stannous chloride (3.0 g) as catalyst until theoretical amount of water was collected. The product was distilled at 110-130 C under reduced pressure of 2-3 mm Hg to remove xylene. The residual erucic acid in the reaction mixture was removed by passing through basic alumina column chromatography. The ester was analyzed for acid value and hydroxyl value. The structure of the triester was established by IR, ¹H NMR, ¹³C NMR and IR.

IR (neat, cm⁻¹): 3009.81 (C=C-H); 1742.93 (-C(O)-); 1156.00 (C-O-C(O)-). ¹H NMR (CDCl₃, ppm): 0.88 (t, -CH₂-CH₃); 1.22-1.36 (m, (-CH₂-)_n); 1.44-1.51 (q, -C-CH₂-CH₃); 1.56-1.64 (m, -C(O)-CH₂-CH₂-CH₂-, -C-CH₂-CH₃); 1.98-2.07 (m, -CH₂-CH₂-CH=CH-); 2.30 (t, -C(O)-CH₂-CH₂-); 4.01 (s, -C-CH₂-O-C(O)-); 5.31-5.38 (m, -CH₂-CH=CH-CH₂-). ¹³C NMR (CDCl₃, ppm): 14.1, 22.6, 22.9, 24.9, 27.1, 29.1- 29.7, 31.9, 34.2, 40.5, 63.6, 129.7, 130.0, 173.4.

Preparation of pentaerythritol tetraesters of erucic acid

Pentaerythritol (12.078, 0.0887 mol), erucic acid (150g, 0.4437 mol) and xylene (75 mL) were taken in a three necked reaction flask equipped with a thermometer, dean stark apparatus and a condenser. The contents were

stirred at 135-140 C in presence of stannous chloride (3.0 g) and carried out the reaction by following the same procedure as described in the above method. The ester was analyzed for acid value and hydroxyl value. The structure of the tetra ester was characterized by ¹H NMR, ¹³C NMR and FT-IR.

IR (neat, cm⁻¹): 3008.37 (=C-H); 1747.24 (-C=O); 1163.74 (C-O). ¹H NMR (CDCl₃, ppm): 0.98 (t, -CH₂-CH₃); 1.25-1.28 (m, (-CH₂-)_n); 1.6 (m, -CO-CH₂-CH₂-CH₂-); 1.98-2.13 (m, -CH₂-CH₂-CH=CH-); 2.3 (t, -CO-CH₂-CH₂-); 4.11 (s, -C-CH₂-O-CO-R); 5.28-5.43 (m, -CH₂-CH=CH-CH₂-). ¹³C NMR (CDCl₃, ppm): 14.0, 22.6, 25.5, 27.1-29.6, 31.5-31.8, 34.0, 41.8, 62.0, 128, 129.9, 173.1.

3. Results and discussion

Vegetable oil derived polyol esters have been used as lubricants and they are expanding their applications due to their superior lubricant properties relative to mineral oils. Most of the vegetable oil-based polyolesters reported in the literature so far are mainly based on high oleic oils such as sunflower, canola and soybean oils. These high monounsaturated oils are optimal performance raw materials to generate environment friendly higher performance lubricants. Erucic acid, a derivative of rapeseed oil is also a monounsaturated fatty acid like oleic acid and commercially available in bulk. Hence, the polyol esters developed based on erucic acid are expected to have similar lubricant properties compared to oleic polyol esters. Keeping this in view, we have developed erucic-based polyol esters with trihydric and tetrahydric alcohols namely, TMP and PE in presence of stannous chloride (0.1% based on reactants) under nitrogen atmosphere. The use of this acidic commercial catalyst affords the derived product with negligible acidity. The process has superiority with respect to easy handling, less reaction time, lower molar ratio of acid to alcohol, high purity, cost effectiveness because of negligible amount of catalyst, energy saving and yields of the order of 90% and above. Completion of the esterification reaction was monitored by

collecting the generated water using Dean Stark Apparatus and by checking disappearance of the hydroxy group at 3450–3480 cm⁻¹ in the infrared spectrum and also by TLC. The synthesized compounds were characterized using IR and NMR. The infrared spectra of esters shows characteristic peak of ester at 1742–47 cm⁻¹. Strong bands at lower frequency between 1156 and 1163 cm⁻¹ are of aliphatic esters. ¹H NMR spectra of products exhibited the following peaks: 1.9–2.0 (–CH₂–CH=CH–), 4.0 ppm (–CH₂–O–C=O), 2.30 (–CH₂–C=O), 1.60–1.7 (–CH₂–), and 0.88–0.97 ppm (–CH₃). ¹³C NMR confirmed the presence of the following group's, carbonyl at around 173 ppm, –CH₂–carbon over the range from 22–34 ppm, and terminal methyl carbon at 14 ppm. Acid values (0.17 and 0.34) confirmed almost complete conversion of polyols with erucic acid. All the polyol esters were characterized for their physico-chemical properties like density, specific gravity, total acid number and evaluated for lubricant properties namely, viscosity, viscosity index (VI), pour point, flash point, copper corrosion value, oxidation stability and tribological properties like wear and weld load properties (Table 1).

The viscometric properties of esters are dependent upon the molecular weight of the ester and the degree of branching in the acid. The prepared TMP ester has a viscosity of 64.06 cSt at 40°C matching the specification of mineral oil based ISOVG-68 lubricant. The pentaerythritol ester has viscosity of 90.47 cSt matching the specification of mineral oil based ISO VG-100 lubricant base stock. Both the products exhibited high viscosity index above 190. High viscosity indices of these oils improve the efficiency of a system and increase the life of components subject to wear.

The esters showed superior flash point (>320°C) against the mineral oil based lubricants indicating a much better fire resistant property. The synthesized esters also pass the

copper strip corrosion test. The oxidation stability as indicated by DSC onset temperature was found to be good.

The load carrying capacity of the polyesters was determined using extreme pressure test. The weld load of esters was 260 for TMP esters and 310 kg for pentaerythritol esters. Both the esters exhibited superior weld load capacity compared to the mineral oil base stocks. The weld load of PE esters was higher, compared to TMP esters, due to more polar functional groups in the molecule and their interaction with the metallic surfaces [Havet et al., 2001].

The antiwear property of the lubricant between two sliding surfaces prevents the surfaces from the dimensional loss of one solid, with or without any actual decoupling and loss of material. Polyol esters are known to show appealing tribological characteristics, in particular with respect to boundary and antiwear properties. These properties are attributable to the presence of ester groups. The oxygen atom of the latter adsorbs on the metal surfaces resulting in good antiwear property. Both the polyol esters exhibited good antiwear property very close to the specifications of mineral oil based base stocks ISOVG-68 and 100. From these studies, it can be concluded that the synthesised polyol esters have good potential for use as biodegradable base stocks for formulation of EP type of industrial gear oil of VG-68 and VG-100 grade.

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Table 1 Physicochemical properties

<i>Test</i>	<i>Method</i>	<i>ETMPE</i>	<i>Industrial gear oil ISO VG-68</i>	<i>EPEE</i>	<i>Industrial gear oil ISO VG-100</i>
Total acid number	AOCS Te 2a-64	0.17	---	0.34	---
Density at 15 °C (g/cm ³)		0.9118	---	0.9061	---
Specific gravity at 15 °C		0.9126	---	0.9062	---
Kinematic Viscosity (cSt) 40 °C	ASTMD 445	64.06	61.2-74.8	90.47	90-110
100 °C		12.12	---	15.17	---
Viscosity index (VI)	ASTMD 2270	192	90	173	90
Flash point (°C)	ASTMD 93	322	180	330	220
DSC Onset temperature (°C)		190	---	181	---
Copper corrosion	ASTMD 130	1a	1	1a	1
Weld load (Kg)	IP 239	260	250	310	250
Wear (mm)	ASTMD 4172	0.4	0.36	0.4	0.36

A Short communication

Sunit Mukherjee

“Unknown facts about sauerkraut”

Indian sauerkraut as characterised by “Hand book of Indigenous Fermented Foods, Ed K.H. Steinkraus, Pub: Mercel Dekker Inc, N.Y & Basel, 1983” is a debatable item in Indian culinary.

Highly perishable primary foods like fruits and vegetables should be processed in India at least to an intermediate stage at the village level where the raw materials are grown. For several reasons, such approach was effective in the early stage of development in U.S.A in the thirties and much later in Canada. Continued training, pilot production and preservation at low cost, developing appropriate products and processes may be required to achieve success.

It should be appreciated that the waste from the processing of fruits and vegetables could be as much as 40-50%, which is fully biodegradable & can be a powerful pollutant of the environment. However, the waste could be used as organic manure in the nearby agriculture field. It would be appropriate if biogas plants are set up to utilize the waste efficiently for production of energy and simultaneous generation of organic manure containing nitrogen, phosphorous and potassium for use in the agricultural field.

It is known that traditionally & commercially Sauerkraut is prepared in the western world by fermentation with natural flora present in the cabbage, which is to certain extent lactic acid bacteria. Under appropriate conditions the vegetable will undergo spontaneous lactic acid fermentation. A definite sequence occur in the growth of Lactic acid bacterial species, wherein the fermentation is initiated by heterofermentative *Leuconostoc mesenteroides*, and followed by heterofermentative rods such as *Lb brevis*, homofermentative *Lb plantarum* and *Pediococcus cerevisiae*. *L. mesenteroides* is

an unique flora which produces acetic acid, ethyl alcohol, carbon dioxide and hydrogen peroxide apart from lactic acid each of which has special significance. Sauerkraut is known to provide certain laxative properties, which are due to the formation of acetyl choline and lactyl choline coming from lecithin. It is also known that an optimum temperature of 18°C. favours the development of desirable flavour, taste and texture and when the Ph falls below 4, no pathogen can grow and the sauerkraut is largely safe for consumption. The author's basic studies (Sunit Mukherjee & H. Ganguly, J.F.S.T. 8,127-131, 1971) indicate that though the flora followed the traditional path the Indian sauerkraut did not have the flavour and texture similar to the one in western world, as the temperature is generally above 18°C. Further Studies at Dr. Subhas Mukherjee Memorial R.B. Research centre, Food & Nutrition Division indicate (Ref 1) that the loss of solid in the Kraut juice is as much as 20-25%. The juice can be diluted and mixed with sugar & spices. Such product can be an acceptable drink for the Indian population. At the level of 3% salt the development of acid and subsequently change in pH is enumerated in the following table.

Changes in pH & acidity at different temperature during Indian sauerkraut fermentation at a salt level of 3%

Based on the above considerations it is suggested that during the cabbage season in West Bengal in particular and others states in India in general, when ambient temp. Ranges from 20°C to 30°C, the time of fermentation may be maintained at 5 to 7 days. After which the sauerkraut may be dipped in a solution of sodium benzoate for further preservation up to 4 months, such that the final concentration of sodium benzoate in the sauerkraut comes to 1000ppm.

In such way mixed vegetables can be

preserved where the share of cabbage should be 80%, preferably. Sliced cauliflower, carrot, radish, green papaya and peas can be used in the mixture. Green vegetables should be avoided as in acid pH they turn brown. With our efforts the Food Safety & Standards Act of India -2006 has incorporated the standards of sauerkraut in India, where the allowable limit for addition of sodium benzoate has been set at 1000 ppm.

It should be appreciated that large

majority of the population in India do not have the facility of canning in tiny or small scale sector, also they do not have the facility for refrigeration, hence class II preservative is allowed by the FSSAI. Furthermore, after long experimentation it is seen that the fermented sauerkraut is not well accepted in the raw form, hence the kraut was washed with hot water to remove much of the lactic acid as well as the preservative, and then the washed cabbage and vegetables are cooked in Indian style with

Av. Temp	No. of days of Fermentation	pH (approx)	Acidity %lactic acid
18°C	3 days	4.2	0.70
18°C	7 days	3.8	0.85
30°C	3 days	3.9	0.94
30°C	7 day	2.8	1.77

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The potential for utilisation of seasonal vegetables exists in India and should be explored in practical programs wherever needed.

RESEARCH ROUNDUP

Biolubricant Production of 2-Ethylhexyl Palmitate by Transesterification Over Unsupported**Potassium Carbonate**

Owing to the decrease of global oil price, development of downstream value-added products is important to biodiesel industry. In this study, Zheng et al. used palmitic acid methyl ester (PAME) as a starting material to produce 2-ethylhexyl palmitate (2-EHP), an environmentally friendly biolubricant product, which was derived from the transesterification of fatty acid methyl esters and long chain fatty alcohols. Conventional synthetic routes of 2-EHP have disadvantages, including high catalyst price, low conversion efficiency, and pollution issues. To solve these problems, in situ transesterification of PAME with 2-ethylhexanol (2-EH) was conducted over unsupported potassium carbonate as heterogeneous catalyst [J. Am. Oil Chem. Soc. (2018) 95: 79–88]. The optimal reaction temperature, 2-EH to PAME molar ratio, and catalyst to PAME mass ratio were 180 °C, 3:1, and 3.0 wt%, respectively. The PAME conversion reached up to 100% within 1 hour under the optimal conditions. In addition, a kinetic model describing the experimental data over a temperature range of 160–180 °C was developed. The dependence of kinetic rate constant (k) on temperature was evaluated, and the activation energy (E_a) for the transesterification of PAME with 2-EH was calculated to be 57.04 kJ mol⁻¹.

Enzymatic synthesis of andiroba oil based polyol for the production of flexible polyurethane

Andiroba oil is a popular Amazonian product that stands out for its numerous medicinal properties, such as anti-inflammatory, antimicrobial, and repellent action. This work by Silva et al. reports the synthesis of andiroba oil polyol (AOP) by enzymatic glycerolysis and its use as raw material for the production of polyurethane foams (PUF). The AOP synthesis was carried out in a tubular fixed bed reactor containing commercial lipase *Novozym 435*[®], using a reaction mixture composed of andiroba oil, glycerol and *t*-butanol [Ind. Crops & Products 113, p.55-63 (2018)]. Three percent solvent contents (50, 100, and 150% volume of solvent per weight of substrate) and temperatures (40, 50, and 60 °C) were evaluated. The AOP synthesized at 50 °C with 150% of solvent showed the highest yield of monoacylglycerol – MAG (66.47 %), being the most suitable for PUF production. PUFs were prepared using AOP or AOP/glycerol blends, hexamethylene diisocyanate biuret, dibutyl-tin dilaurate as catalyst and water as blowing agent. Polyurethane formation was confirmed by FTIR. Due to the low average functionality of AOP ($F^-(OH) = 1.23$), the production of dimensionally stable foams, was possible only through its combination with glycerol.

Production of chemicals from microalgae lipids – status and perspectives

Recently The engineering of algae strains, cultivation, and further processing steps in microalgae production are being considered actively in view of the utilization of microalgae lipids for chemicals. Insights from biofuel production trials on the one hand and existing processes for very high-value pharmaceuticals on the other hand are instructive to this end. A recent example of the production of a surfactant from chemical intermediates gained from algae oil and further opportunities are discussed in this review article by Hess et al. microalgae oil lipids are discussed as an alternative source for chemicals. The tuning of lipid production in microalgae via genetic engineering and cultivation optimization for this purpose is reviewed [Eur. J. Lipid Sci. Technol. 120, 20181700152 (2018)]. Authors illuminated the bottlenecks of upscaling and the state of the art in industrial algae production by means of three concrete case studies. Further insights into the catalytic functionalization to high-value products were provided.

Synthesis of Dimer Fatty Acid Methyl Esters by Catalytic Oxidation and Reductive Amination: An Efficient Route to Branched Polyamides

Novel and versatile route toward dimer fatty acid methyl esters (dimer FAMES) via catalytic oxidation and reductive amination is reported by Czapiewski and Meier. The oxyfunctionalization of mono-unsaturated FAMES bearing different chain lengths (C11, C18, C22) is accomplished by a co-catalyst-free Wacker Oxidation process in a high pressure reactor. The applied catalytic system of palladium(II) chloride in a dimethylacetamide/water mixture enabled the formation of keto-FAMES in the presence of molecular oxygen as sole re-oxidant. In a first attempt, partially renewable dimer FAMES are synthesized by reductive amination of keto-FAME (C18) in the presence of various aliphatic and aromatic diamines and sodium triacetoxyborohydride as selective reducing agent. In another approach, the keto-FAMES directly underwent reductive amination using Raney-Nickel in order to obtain the corresponding amino-FAMES. Subsequently, the keto- and amino-FAMES are used for the synthesis of fully renewable dimer FAMES via reductive amination with sodium triacetoxyborohydride as reducing agent [Eur. J. Lipid Sci. Technol. 120 (1), 20181700350 (2018)]. In order to demonstrate a possible application for these new dimer FAMES, three out of the thirteen synthesized dimer FAMES are selected and studied in a polycondensation with renewable 1,10-diaminodecane using TBD as catalyst. The polyamides are obtained in molecular weights (M_n) of up to 33 kDa and are carefully characterized by ¹H-NMR spectroscopy, FTIR, SEC, and DSC analysis.

Epoxidation of Methyl Oleate and Subsequent Ring-Opening Catalyzed by Lipase from *Candida* sp.

A green method for chemo-enzymatic epoxidation of methyl oleate is developed by Niuniu et al. to get epoxidized methyl oleate. Subsequently, excessive hydrogen peroxide and extended reaction time led to polyols through ring-opening reaction catalyzed by immobilized lipase from *Candida* sp. 99–125. The corresponding reaction conditions are systematically investigated [Eur. J. Lipid Sci. Technol. 120 (2), 20181700257] (2018)]. High epoxy number is obtained at 70 °C for 5 h when 1.5 wt% immobilized lipase and 1.5 equivalent of hydrogen peroxide are used. Interestingly, no hydrolysis reaction of epoxidized methyl oleate is observed. Moreover, epoxidized methyl oleate generated shows good plasticizing effect on PVC resin. On the basis of epoxidation reaction, more hydrogen peroxide and extended reaction time are employed in the ring-opening reaction, resulting in more polyols. Polyurethane rigid foams with good thermal conductivity, dimensional stability, and compress strength can be successfully prepared through the use of polyols acquired by ring-opening reaction of epoxidized soybean oil. In addition, the enzyme shows good reusability toward epoxidation and ring-opening reaction.

Physicochemical Properties of Enzymatically Produced Palm-Oil-Based Cocoa Butter Substitute (CBS) With Cocoa Butter Mixture

Biswas et al. prepared cocoa butter substitute (CBS) using ternary-blend of palm mid-fraction/palm kernel oil/palm stearin enzymatic interesterification to investigate physicochemical properties of CBS and compatibility of CBS/CB and fatty acid. Ternary-blend and commercial stearic/oleic acids are mixed to produce blend (80% ternary-blend/15% stearic/5% oleic) with three major fatty acids composition comparable to CB. Interesterified blend under optimized conditions of 4% lipase (w/w), incubation time of 6 h at 60 °C shows a melting endotherm at 33.5 °C, similar to CB. The composition of triacylglycerols (POSt and StOSt) of interesterified blend is significantly ($P < 0.05$) increased compared to non-interesterified blend. Triacylglycerol, melting profile, solid fat content (SFC), polymorphism, and crystal morphology are determined using GC, HPLC, DSC, pNMR, XRD, and PLM. SFC and polymorphism of interesterified fat are different from CB at 24 °C. A total of 5–20% of CBS/CB shows similar melting behavior and polymorphism to CB. A desirable monotectic effect is observed at 15–25 °C for these blends [Eur. J. Lipid Sci. Technol. 120 (3), 20181700205] (2018)]. Therefore, interesterified blend is potentially used as CBS to be added up to 20% with CB for chocolate production.

Efficient microwave-assisted synthesis of glycerol monodecanoate

Solvent-free microwave-assisted synthesis was carried out to prepare 2,3-dihydroxypropyl decanoate, by esterification of decanoic acid in the presence of two distinct glycerol derivatives, glycidol, and glycerol carbonate, respectively by Mhanna et al.. The process described is based on microwaves as heating source with electrical power in the range of 200–400 W, involving stoichiometric proportions of decanoic acid and glycerol derivatives, and using catalytic amounts of TBAI used as organocatalyst. Conversion and selectivity rates of esterification reactions were monitored by ^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectroscopy. The predominantly formed ester, 2,3-dihydroxypropyl decanoate was fully characterized by infrared and NMR spectroscopy, mass spectrometry, and elemental analysis. Compared with the classical heating procedures, and whatever the glycerol derivatives used, total conversions were obtained with considerably reduced reaction times [Eur. J. Lipid Sci. Technol. 120, 20181700113] (2018)]. Thus, under 300 W, esterification requires only 1 min exposure from glycidol and 5 min from glycerol carbonate. The use of heating with conventional oil bath conditions needs residence times of more than 1 h (even 24 h in the case of glycerol carbonate). The microwave-assisted synthesis also notably enhances the selectivity in 2,3-dihydroxypropyl decanoate (at 300 W, 90, and 50%, respectively), reinforcing the efficiency and the interest of the method.

Nanoemulsions From Unsaturated Fatty Acids Concentrates of Carp Oil Using Chitosan, Gelatin, and Their Blends as Wall Materials

This work by Esquerdo et al. was aimed to study the development of food-grade nanoemulsions containing unsaturated fatty acids concentrates from carp oil, using chitosan, and gelatin as wall materials. The effects of chitosan:gelatin ratio, polymer concentration and homogenization time on the nanoemulsions characteristics were evaluated. Phase separation occurred when the chitosan:gelatin ratio was higher than 50:50. Nanoemulsions using proportion of chitosan over than 70% remained visually stable, with no phase separation, for more of 7 days. The highest homogenization time (20 min) and the lowest biopolymers concentration (1% w/v) resulted in smaller particle sizes for the ratios of 100:0, 90:10, and 70:30 (respectively, 292.0, 52.3, and 34.8 nm). The zeta potential increased with the amount of chitosan (from 26.5 to 31.0 mV), while pH and refractive index were not affected by the biopolymers ratio [Eur. J. Lipid Sci. Technol. 120, 20171700240] (2018)]. After 7 days of storage, the nanoemulsion with 90:10 of chitosan:gelatin ratio was in the acceptable range of the legislation, showing peroxide value of 4.8 meq kg⁻¹, p-Anisidine value of 9.8 meq kg⁻¹, and Tox value of 19.4 meq kg⁻¹. Chitosan and gelatin provided high

stability to the emulsions and also behaved as good wall materials, demonstrating the importance of studying its combination to form food-grade nanoemulsions.

GC-MS Characterization of Hydroxy Fatty Acids Generated From Lipid Oxidation in Vegetable Oils

Lipid oxidation has long been described as following a radical chain reaction mechanism, where hydrogen abstraction is considered the preferred pathway. Hydroxy compounds are, in theory, major products formed from hydrogen abstraction but their presence is rarely monitored. In this study by Xia et al., a GC-MS technique to characterize hydroxy fatty acids (FA) formed during the oxidation of sunflower and canola oils is described. First, hydroxy FA in oxidized oils are methylated and isolated from non-oxygenated structures using solid phase extraction (SPE). Then they are converted into their trimethylsilyl (TMS) derivatives using a N,O-bis (trimethylsilyl) trifluoroacetamide (BSTFA)-pyridine method and identified by their electron ionization (EI) and positive chemical ionization (PCI) spectra. Separation of most isomeric hydroxy FA with very similar structures is accomplished using a DB-23 capillary column with (50% - cyanopropyl)-methylpolysiloxane phase. The fragmentation patterns of the TMS derivatives are discussed in detail and several easily applicable rules for spectral interpretations are presented [Eur. J. Lipid Sci. Technol. 120, 20171700313] (2018)]. The major hydroxy FA arising from oxidation of sunflower and canola oils are allylic and conjugated structures, specifically 8-, 9-, 10-, and 11-hydroxyoctadecenoic acid (OH-C18:1) and 9- and 13-hydroxyoctadecadienoic acid (OH-C18:2).

Analytical Methods to Evaluate the Quality of Edible Fats and Oils: The JOCS Standard Methods for Analysis of Fats, Oils and Related Materials (2013) and Advanced Methods

Edible fats and oils are among the basic components of the human diet, along with carbohydrates and proteins, and they are the source of high energy and essential fatty acids such as linoleic and linolenic acids. Edible fats and oils are used in for pan- and deep-frying, and in salad dressing, mayonnaise and processed foods such as chocolates and cream. The physical and chemical properties of edible fats and oils can affect the quality of oil foods and hence must be evaluated in detail. The physical characteristics of edible fats and oils include color, specific gravity, refractive index, melting point, congeal point, smoke point, flash point, fire point, and viscosity, while the chemical characteristics include acid value, saponification value, iodine value, fatty acid composition, trans isomers, triacylglycerol composition, unsaponifiable matters (sterols, tocopherols) and minor components (phospholipids, chlorophyll pigments, glycidyl fatty acid esters). Peroxide value, *p*-anisidine

value, carbonyl value, polar compounds and polymerized triacylglycerols are indexes of the deterioration of edible fats and oils. This review by Endo describes the analytical methods to evaluate the quality of edible fats and oils, especially the Standard Methods for Analysis of Fats, Oils and Related Materials edited by Japan Oil Chemists' Society (the JOCS standard methods) and advanced methods [J.Oleo Science 67, p.1-10 (2018)].

Viscous Flow Behaviour of Karanja Oil Based Bio-lubricant Base Oil

Karanja oil (KO) is widely used for synthesis of bio-fuel karanja oil methyl ester (KOME) due to its competitive price, good energy values and environmentally friendly combustion properties. Bio-lubricant is another value added product that can be synthesized from KO via chemical modification. In this work by Sharma et al. karanja oil trimethylolpropane ester (KOTMPE) bio-lubricant was synthesized and evaluated for its viscous flow behaviour. A comparison of viscous flow behaviours of natural KO and synthesized bio-fuel KOME and bio-lubricant KOTMPE was also made. The aim of this comparison was to validate the superiority of KOTMPE bio-lubricant over its precursors KO and KOME in terms of stable viscous flow at high temperature and high shear rate conditions usually encountered in engine operations and industrial processes. The free fatty acid (FFA) content of KO was 5.76%. KOME was synthesized from KO in a two-step, acid catalyzed esterification followed by base catalyzed transesterification, process at 65°C for 5 hours with oil-methanol ratio 1:6, catalysts H₂SO₄ and KOH (1 and 1.25% w/w KO, respectively). In the final step, KOTMPE was prepared from KOME via transesterification with trimethylolpropane (TMP) at 150°C for 3 hours with KOME-TMP ratio 4:1 and H₂SO₄ (2% w/w KOME) as catalyst [J.Oleo Science 67, p.105-111 (2018)]. The viscosity versus temperature studies were made at 0–80°C temperatures in shear rate ranges of 10–1000 s⁻¹ using a Discovery Hybrid Rheometer, model HR-3 (TA instruments, USA). The study found that viscosities of all three samples decreased with increase in temperature, though KOTMPE was able to maintain a good enough viscosity at elevated temperatures due to chemical modifications in its molecular structure. The viscosity index (VI) value for KOTMPE was 206.72. The study confirmed that the synthesized bio-lubricant KOTMPE can be used at high temperatures as a good lubricant, though some additives may be required to improve properties other than viscosity.

Synthesis of Fatty Acid Methyl Esters Using Mixed Enzyme in a Packed Bed Reactor

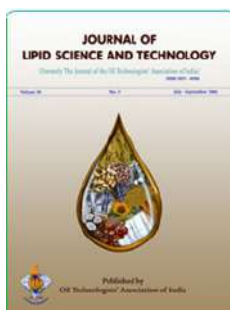
Fatty acid methyl esters were synthesized by Ryu et al. from palm fatty acid distillate (PFAD) and methanol in a

packed bed reactor via lipase-catalyzed esterification. The PFAD consisted of 91 wt% of free fatty acids, 2 wt% monoacylglycerides, 3 wt% diacylglycerides, and 4 wt% triacylglycerides. *t*-Butanol was employed as a reaction medium and a mixed enzyme consisting of Lipozyme TL IM from *Thermomyces lanuginosus* and Novozym 435 from *Candida antarctica* was employed as the biocatalyst [J.Oleo Science 67, p.321-326 (2018)]. The effect of mixed enzyme was investigated and the optimum blending ratio (w/w) of Novozym 435 to Lipozyme TL IM was 5:95. Using the mixed enzyme, the optimum molar ratio (PFAD to methanol) and temperature were determined to be 1:6 and 30°C, respectively. Under the optimized conditions, the maximum yield of ca. 96% was achieved.

Hydrogel-Templated Solid Base Catalysts for Transesterification of Soybean Oil

A new method for utilization of hydrogel is proposed in this work by Meechai et al. for the preparation of solid base catalysts for the transesterification of vegetable oil. When a solution of KF is mixed with a solution of $\text{Ca}(\text{NO}_3)_2$, CaF_2 is obtained and inactive as a catalyst in the transesterification of vegetable oils. The catalysts

were synthesized by the sequential incorporation of KF and/or $\text{Ca}(\text{NO}_3)_2$ solutions into the hydrogel upon microwave irradiation and then the as-obtained hydrogel was calcined at 800°C for 5 hours to eliminate the template and yield catalysts for the biodiesel productions. The prepared catalysts obtained by the different ways in the incorporation of ions into the hydrogel showed different physical properties and catalytic activities in the transesterification of soybean oil [J.Oleo Science 67, p.355-367 (2018)]. All catalysts, except the low concentration of $\text{Ca}(\text{NO}_3)_2$, exhibiting the high activity yielding more than 90% FAME after 1 hour at 65°C, using oil to methanol molar ratio of 1:15 and 10 wt% of catalyst amounts.



JOURNAL OF LIPID SCIENCE AND TECHNOLOGY

Disseminating Innovative Technologies

About Journal

The *Journal of Lipid Science and Technology (JLST)* is a quarterly published journal by Prof. R. P. Singh, on behalf of the Oil Technologists' Association of India (OTAI), C/o OTAI Building, Harcourt Butler Technical University, Kanpur-208002, UP (INDIA). It aims to provide a central path for exchanging and disseminating new ideas and technologies in the fields of vegetable oils and their derivatives, alternative fuels (especially bio fuel), surface coatings, lubricants, detergents, polymers, foods and food products. The JLST publishes the original research paper, review paper, general article, newsletters and short communication on various aspects such as new process development for storage, production and refining, by-products utilization, environmental impact and economical aspects of above mentioned fields.

The technology for vegetable oils include harvesting and storage of oil bearing materials, extraction of oil, refining, packaging and supply chain management for both edible as well as non-edible oils. Vegetable oil derivatives include confectionery products, green polymer, surfactants, etc. If new ideas are outstanding, but do not belong to above fields, will also be considered for publication.

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