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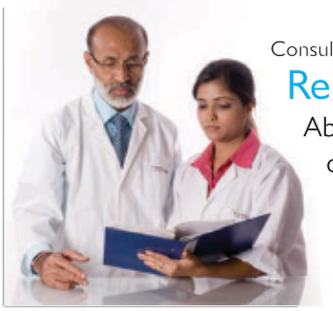

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From Editors Desk

India has exploited the potential of ricebran oil to the maximum extent as our rice production is only about 106 MMT which can potentially give 1.6 MMT of ricebran oil. But of this we are able to produce 1.0 MMT. However, China produces 145 MMT of rice and thus has a potential of recovery of 2.0 MMT of ricebran oil. Actual production is only 10% of this, i.e., 0.2 MMT of ricebran oil. The total rice produced in the world is 482 MMT and thus the world has a potential of 6.75 MMT of ricebran oil but the total world production of ricebran oil is only 1.5 MMT leaving 5.25 MMT as untapped. Our industry has taken lead in educating people towards the health benefits of ricebran oil.

Similarly, Indian cottonseed industry has seen a transformed scenario after the success received by BT cotton. No wonder, it has transformed the lives of large number of farmers engaged in cotton by significantly improving yields. In 2001-02, cotton seed oil production was only 4.4 lakh tonnes. This year, we are expecting production to skyrocket to 1.4 MMT. Cottonseed is now the biggest oilseed in the country (18% of the total domestic oil production). Put together, the production of ricebran oil and cottonseed oil is 35% of the total domestic production, still there is no significant development plan.

Overall contribution of groundnut oil has shrunk dramatically in the overall contribution to the oilseeds. A survey has been conducted and it seems that the crop is rebounding and it is estimated to have a crop of 3.15 MMT. This will be the highest ever kharif groundnut crop but the prices are hovering below the MSP which does not augur well for the future. The government must make a strategy to support the farmers so that they are not discouraged of producing groundnut oil.

In fact our dependence on import of edible oils has gone from 3% in nineties to 70% today. The reason is that the oilseed production has remained stagnant but the demand has grown faster. The possible measures to bridge the gap between demand and supply could be -

(a) Allowing BT crop in mustard which is high yielding.
(b) Encouraging research in oilseeds by agriculture scientists to increase productivity which is presently on an average of 1000 kg per hectare.
(c) Faster implementation of palm plantation which was already planned but actual plantation is too low.

R. P. Singh

(R.P. SINGH)

INDEX TO ADVERTISEMENTS / ANNOUNCEMENTS

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President's Message

Dear Friends,

I thank all the members of OTAI, especially the members of the CEC, for reposing their trust in me and putting on my shoulders the responsibility of the President of our esteemed organization. I am determined to put in my best efforts to meet the expectations of all OTAI stakeholders.

Leading the association is great responsibility, which I am aware as I have been witness of presidency of all my able predecessors, Dr J.G.Kane, Dr T.R. Shesadri, Dr N.Godbole, Prof M.M. Chakrabarty, Dr K.T.Acharya, Prof. A K Vasishtha, Sri N. B. Godrej, Sri S. C. Singhal, who have made great contributions towards actualizing the "vision" and "mission" perceived by our founder Late Shri RaoSaheb Athawale in 1943-44. However the challenges and issues which were before the leadership then are different from those that OTAI faces today. The rapid speed of change which we all are witnessing in every sphere of life is also affecting all of us at OTAI and it cannot be overlooked.

We need to understand, analyze and prioritize the modern day challenges and issues, which are being faced by the society, the country, the academicians and the industry. New plans need to be evolved after looking afresh at the important areas of concern, be they environmental, energy, consumer's health, food safety, quality, import, taxes or technology, etc.

I invite all the esteemed members and stake holders of OTAI to share the challenges and issues that they feel are important for our organization to address in the 21st century. Based on this feedback and through mutual discussions, I will endeavor to collectively evolve new plans, new strategies and channelize efforts to address these challenges and goals. I am sure that all OTAI members will offer their support in this endeavor and enable me to take this initiative forward. I wish and would like that our association should take up some work in skilling and education in the field of our domain and concerns.

The 4th issue of OTAI Journal 2017 is in your hand, I am confident that the papers and other items published in the Journal will be of interest. I shall keenly await your response!

I also take this opportunity to send season's greetings and wish you and your families a very prosperous and Happy 2018.

Prof. Rakesh K Trivedi
National President OTAI

Synthesis and Evaluation of Novel 10-Undecenoic Acid based β- Amino Thiols as Anti Cancer Agents

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Key words:

Abstract

The synthesis of five novel 10-undecenoic acid-based β-amino thiols was carried out using a facile, environmentally green ionic liquid mediated reaction. 10-Undecenoic acid was alkylated to methyl and n-butyl 10-undecenoates and were subjected to epoxidation followed by their treatment with ammonium thiocyanate in ionic liquid medium 1-methyl imidazolium tetrafluoro borate:H_2O system at 60ºC to obtain their corresponding epithio undecanoates. After complete conversion of epoxy to epithio ring, reaction temperature was raised to 80-90ºC and epithio ring was opened with different amines to obtain alkyl β-amino thiol derivatives. All the synthesized compounds were fully characterized and their structures were confirmed by spectral data. The prepared compounds were screened for their cytotoxic activity against four cancer cell lines. These compounds exhibited selective activity towards breast cancer cell lines with methyl-10-mercapto (11-anilino) undecanoate showing excellent cytotoxicity towards both breast cancer cell lines, which was comparable to control doxorubicin.

1. Introduction

Amino thiols constitute an important class of compounds for medicinal and synthetic chemistry. Some of them such as cysteine, homocysteine, cysteamine, and penicillamine are naturally occurring and are involved in important biological processes. They are constituents of complex biomolecules such as peptides and coenzyme A. Their biological and physicochemical specificities are especially due to the properties of the thiol function (acidity, metal affinity). They have various applications as enzyme inhibitors (Ocain et al, 1987) radio protective agents, as intermediates for the synthesis of a number of biologically active compounds (Mourtas et al., 2003), or in peptide synthesis when attached through their thiol group on resins (Gaumont et al., 2009), synthetic and medicinal chemistry, catalysis and materials chemistry (Fincke, 1956; Fahl et al., 2005; Sikorski et al., 2002; Gorla et al., 2014). To increase the effectiveness of mercapto amino compounds, their lipophilization is the choice of derivatization as it provides beneficial effects of both the functionalities and the lipid involved in one chemical entity.

Lipids, especially fatty acids and their derivatives are known for their broad spectrum of activity which expands their application in developing new hybrid biomolecules which help in host defences against potential pathogenic microbes (Kanjilal et al., 2008; Kaki et al., 2016; Gorla et al., 2016; Narra et al., 2017). Research interest in producing new lipid based derivatives is increasing due to their potential applications in biomedical fields. Earlier reports on the production of amino thiols were focused on the incorporation of mercapto amine groups into short chain hydrocarbons (Kuranova and Snetkova 1985; Taguchi et al., 1984; Stewart, 1964; Turk et al., 1964; Peudru et al.,2012; Hérault et al., 2006).

Amino thiols with general formula $PhCH_2CH_2CH(SH)CH_2NR_1R_2$ where (R_1 = n-Pr, t-Bu, Ph, R_2 = H; NR_1R_2 = NEt_2, 1-piperidinyl, 4-morpholinyl) were prepared by Guseinova et al. (2008) by ring opening of 1,2-epithio-4-phenyl butane with primary and secondary amines. Antioxidant activity of the amino thiols in cumol autoxidation was investigated. Filho et al. (1986) prepared four new N-alkyl-2-mercapto-1-octylamine hydrochlorides by the reaction of 1,2-epithio octane with propyl, butyl, isobutyl and cyclohexyl amines followed by conversion to their hydrochlorides. The thiols were converted into their respective disulfides by oxidation and biological assay of the products for activity against infection by *Schistosoma mansoni* was done. Only 2,2'-

dithiobis(N-isobutyl-1-octylamine) exhibited activity In view of developing new fatty acid based derivatives.

The conventional methods used for preparation of amino thiols or mercapto amines involve use of environmentally unfriendly solvents and toxic heavy metal catalysts. Herein we disclose a novel synthetic approach for the synthesis of 10-undecenoic acid based mercapto amine derivatives by reacting epoxy group in alkyl-10-epoxy undecanoates with ammonium thiocyanate as a source of sulphur in ionic liquid- $[HMIM]BF_4$ – H_2O (2:1) medium at 60°C. Followed by the ring opening of epithio group with different amines.

1. Experimental

2.1 Materials

10-Undecenoic acid was obtained from Jayant Oil Mills (Mumbai, India). Methanol, butan-1-ol, hexane and ethyl acetate were procured from Qualigens Fine Chemicals (Mumbai, India). Dichloromethane and m chloroperbenzoic acid (m-CPBA), aniline, ammonium thiocyanate, 1-methyl imidazole and HBF4 were purchased from Sigma Aldrich (St. Louis, USA). Hydrogen peroxide (30% aqueous solution), xylene and p-toluene sulphonic acid (p-TSA) were purchased from M/s sd fine-chem Ltd., Mumbai. 85% Formic acid solution and sulfuric acid (AR grade) were purchased from M/s Rankhem, New Delhi.

2.2 Methods

The cytotoxicity assay (MTT) was used for evaluation of all synthesized compounds. Four different cancer cell lines viz., A549 derived from human alveolar adeno carcinoma epithelial cells (ATCC No. CCL-185), HeLa derived from human cervical cancer cells (ATCC No. CCL-2), MDA-MB-231 derived from human breast adenocarcinoma cells (ATCC No. HTB-26) and MCF7 derived from human breast adenocarcinoma cells (ATCC No. HTB-22) using the MTT assay were obtained from the ATCC (Bethesda, MD, USA) and maintained in DMEM supplemented with 10% FBS, 2 mM L-glutamine, 100 U/mL penicillin, and 100 µg/mL streptomycin at 37 °C in a 5% CO2 incubator. After seeding of cells in 96 well culture plates, they were allowed to attach properly. Test compounds of different concentrations ranging from 1 to 50 µM were added in triplicates and incubated for 24 h. The cells were then incubated with MTT (0.5 mg/mL) for 3 h and, to dissolve the insoluble formazan crystals, 100 µL DMSO was added to each well. Finally the absorbance of the plates was measured using a Synergy H1 multi-mode plate reader (USA). Doxorubicin was used as the positive control for comparison.

2.3 Synthesis

Synthesis of 1-methylimidazolium tetrafluoroborate $[HMIM]BF_4$

1-Methylimidazole (6 g, 0.0738 mol) was taken in a three-necked round-bottom flask, and the temperature of the medium was controlled at 0 °C. 48% Tetrafluoroboric acid (9.54 mL, 0.0738 mol) was added to it drop wise over a duration of 40 min. The reaction contents were further stirred at room temperature for 2 h. After completion of the reaction, the contents were subjected to high vacuum at 80–90 °C to remove moisture. The excess imidazole was removed from the product by ethyl acetate washings, followed by drying under reduced pressure to yield a light yellow viscous liquid of 1-methylimidazolium tetrafluoroborate $[HMIM]BF_4$.

Synthesis of methyl-10-epithio-undecanoate

Methyl-10-epoxy undecanoate (3 g, 0.0140 mol) and ammonium thiocyanate (1.06 g, 0.0140 mol), were taken into a round bottomed flask. To the contents ionic liquid $[HMIM]\ BF_4$ (6 g, 0.0352mol) and water (3 g, 0.167 mol) were added in 2: 1 ratio and stirred at 60ºC. The reaction was monitored by TLC using solvent system hexane: ethyl acetate (90:10 v/v) and charred using anisaldehde charring solution. After completion of the reaction, diethyl ether was added and the ether layer containing the product was decanted from the top. The ether extraction was repeated and the ethereal solution was washed with water and dried over sodium sulfate. The crude product was purified using silica gel column with n-hexane and ethyl acetate (90:10 v/v) to afford the methyl-10-epithio undecanoate

(2.2 g, 70%). The product was characterized by ^{1}H NMR, IR, GC and GC-MS spectral data.

Synthesis of Butyl-10-epithio-undecanoate

Butyl-10-epoxy undecanoate (3 g, 0.0117 mol) and ammonium thiocyanate (0.889 g, 0.007812 mol) were taken into a round bottomed flask. To the contents ionic liquid [HMIM] BF_4 (6 g, 0.0352 mol) and water (3 g, 0.167 mol) were added in 2: 1 ratio and stirred at 60 ºC for a period of 23 h. After completion of the reaction as indicated by TLC, the crude product was purified using silica gel column with n-hexane and ethyl acetate (90:10 v/v) to afford the butyl-10-epithio undecanoate (2.16 g, 68%). The product was characterized by ^{1}H NMR, IR, GC and GC-MS spectral data.

Synthesis of Methyl-10-mercapto-11-(phenylamino) undecanoate

Methyl 10-epithio undecanoate (1.0 g, 0.004347 mol), aniline (0.404 g, 0.004347 mol), and ionic liquid [HMIM]BF_4 (3 g, 0.0176 mol) were taken in a 25ml RB under magnetic stirring at 80-85ºC. The progress of the reaction was monitored by TLC using solvent system hexane:ethyl acetate (80:20 v/v), charred the T.L.C using anisaldehyde solution. After completion of the reaction, the crude product was extracted using ether and the ethereal solution was washed with water and dried over sodium sulfate. The combined ether extracts were concentrated using rotary evaporator. The product was characterized by ^{1}H NMR, IR spectral data.

^{1}H NMR: ($CDCl_3$), δ (ppm/TMS): 1.22-1.60 (m, 14 H, -C<u>H</u>$_2$); 2.2-2.3(t, 2H, -CO-C<u>H</u>$_2$); 2.9-3.0 (s,1H, (-C<u>H</u>-SH); 3.2-3.3 (dd, 2H, -NH-C<u>H</u>$_2$-CH-SH); 3.9-4.0 (s broad, 1H, -N<u>H</u>-, -S<u>H</u>-); 3.65-3.7 (s sharp,3H, -O-C<u>H</u>$_3$); 6.9-7.4 (m, 5<u>H</u>, Aryl);

IR (neat, cm^{-1}: 3339 (-SH, -NH); 1599 (N-H bending); 2928(C-H stretching);

EIMS, m/z 323 $[M]^+$, $C_6H_5NHCH_2^+$ (m/z 106).

Synthesis of Butyl10-mercapto-11-(phenyl amino) undecanoate

Butyl 10-epithio undecanoate (1.0 g, 0.003676 mol), aniline (0.341 g, 0.003676 mol) and ionic liquid [HMIM]BF_4 (3 g, 0.0176 mol) were taken in a 25 mL RB flask under magnetic stirring at 80-85 ºC. The progress of the reaction was monitored by TLC using using hexane:ethyl acetate (80:20 v/v) solvent system and charring the TLC plate with using anisaldehyde solution. After completion of the reaction as indicated by TLC, the crude product was extracted with ether, and the ethereal solution was washed with water and dried over sodium sulphate. The ether extract was concentrated using rotary evaporator and purified using silica gel column chromatography using hexane and ethyl acetate (80/20) to afford the butyl10-mercapto-11-(phenyl amino) undecanoate (0.45 g, 51%). The product was characterized by ^{1}H NMR, ^{13}C NMR, IR and EI-MS spectral data

^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm/TMS): 0.9-1.0 (t, 3H, -C<u>H</u>$_3$); 1.2-1.65 (m, 18 H, -C<u>H</u>$_2$); 2.2-2.3 (t, 2H, -CO-C<u>H</u>$_2$); 2.9-3.0 (m,1H, (-C<u>H</u>-SH); 3.25-3.3 (dd, 2H, -NH-C<u>H</u>$_2$-CH-SH); 3.8-3.85 (s, 1H, -N<u>H</u>-, -S<u>H</u>-); 4.05-4.1 (t,2H, -O-C<u>H</u>$_2$) 6.65-7.3 (m, 5<u>H</u>, Aryl).

^{13}C NMR ($CDCl_3$, ppm): 13.6 (<u>C</u>H$_3$-CH$_2$-); 18.9-31.2 ((-CH$_2$-)$_8$); 33.9 (-<u>C</u>H$_2$-C=O); 36.3 (-CH-SH); 60.3 (CH$_2$-NH-); 64.8 (-<u>C</u>HO-C=O); 120-147 (aromatic C-NH-); 173.1 (-COO-).

IR (neat, cm^{-1}): 3403 (-NH); 1603 (-N-H bending); 2929 (C-H stretching).

EI-MS, m/z: 366 $[M+1]^+$, 332, 290, 199, $C_6H_5NHCH_2^+$ (m/z 106).

Synthesis of butyl10-mercapto-11-(p-nitro phenyl amino) undecanoate

Butyl 10-epithio undecanoate (1.0 g, 0.003676 mol), p-nitro aniline (0.452 g, 0.003676 mol) and ionic liquid [HMIM]BF_4 (3 g, 0.0176 mol) were taken in a 25 mL R B flask under magnetic stirring at 80-85 ºC. Similar experimental procedure was followed as described above. The crude product was purified using silica gel column chromatography using hexane and ethyl acetate (80/20) to afford the butyl10-mercapto-11-(p-nitrophenyl amino) undecanoate (0.35 g, 48%). The product was characterized by ^{1}H NMR, ^{13}C NMR, IR and EI-MS spectral data.

^{1}H NMR: (400 MHz, $CDCl_3$), δ (ppm/TMS): 0.8-0.95 (t, 3H, -C<u>H</u>$_3$); 1.2-1.65 (m, 18 H, -C<u>H</u>$_2$); 2.25-

2.35 (t, 2H, -CO-C$\underline{H}_2$); 2.9-3.0 (m,1H, (-C$\underline{H}$-SH); 3.19-3.44 (dd, 2H, -NH-C$\underline{H}_2$-CH-SH); 4.3-4.45 (s broad, 1H, -N$\underline{H}$-, -S$\underline{H}$-); 4.0-4.15 (t,2H, -O-C$\underline{H}_2$) 6.6-8.1 (m, 4$\underline{H}$, Aryl);

^{13}C NMR ($CDCl_3$, ppm): 13.6 ($\underline{C}H_3$-CH_2-); 18.9-31.2 ((-CH_2-)$_8$); 33.9 (-$\underline{C}H_2$-C=O); 36.3 (-CH-SH); 60.3 (CH_2-NH-); 64.8 (-$\underline{C}$HO-C=O); 120-147 (aromatic C-NH-); 173.1 (-COO-).

FT-IR (neat, cm^{-1}): 3403 (-NH); 1603 (-N-H bending); 2929 (C-H stretching);

EI-MS, m/z: 411 $[M+1]^+$, 378, 336, 245, $C_6H_5NHCH_2^+$ (m/z 151).

Synthesis of Butyl10-mercapto-11-(p-tolyl amino) undecanoate

Butyl 10-epoxy undecanoate (0.5 g, 0.0019 mol), para toluidine (0.208 g, 0.0019 mol) and ionic liquid [HMIM]BF_4(1.5 g, 0.0088 mol) were taken in a 25 mL R. B flask under magnetic stirring at 80-85 ºC. Similar experimental procedure was followed as described above. The crude product was purified using silica gel column chromatography using hexane and ethyl acetate (80/20) to afford the butyl10-mercapto-11-(p-tolylamino) undecanoate (0.28 g, 54%). The product was characterized by ^{1}H NMR, ^{13}C NMR, IR and EI-MS spectral data.

^{1}H NMR: (400, MHz, $CDCl_3$), δ (ppm/TMS): 0.8-0.95 (t, 3H, -C$\underline{H}_3$); 1.2-1.65 (m, 18 H, -C$\underline{H}_2$); 2.25-2.35 (t, 2H, -CO-C$\underline{H}_2$, C$\underline{H}_3$-Aryl); 3.2-3.3 (m,1H, (-C$\underline{H}$-SH); 3.45-3.6 (dd, 2H, -NH-C$\underline{H}_2$-CH-SH); 3.85-4.0 (s broad, 1H, -N$\underline{H}$-, -S$\underline{H}$-); 4.05-4.15 (t,2H, -O-C$\underline{H}_2$) 6.8-7.2 (d, 4$\underline{H}$, Aryl);

^{13}C NMR ($CDCl_3$, ppm): 14 ($\underline{C}H_3$-CH_2-); 20.9(CH_3 of aniline); 18.9-31 (-CH_2-); 34.5 (-$\underline{C}H_2$-C=O); 36.5 (-CH-SH); 55.2 (CH_2-NH-) 64.2 (-$\underline{C}$HO-C=O); 149 (aromatic C-NH-); 173.5 (-COO-);

FT-IR (neat, cm^{-1}): 3447 (-NH); 1592 (-N-H bending); 2929 (C-H stretching)

EI-MS, m/z: (M+HCOO): 424, 379, 332, 243, 239.

Synthesis of Butyl-10-mercapto-11-(pyridin-2-ylamino) undecanoate

Butyl 10-epithio undecanoate (1 g, 0.003676 mol), amino pyridine (0.349 g 0.003676 mol) and ionic liquid [HMIM]BF_4(3 g, 0.0176 mol) were taken in a 25 mL R. B flask under magnetic stirring at 80-85 ºC. Similar experimental procedure was followed as described as above. The crude product was purified using silica gel column chromatography using hexane and ethyl acetate (80/20) to afford the butyl10-mercapto-11-(pyridin-2-ylamino) undecanoate (0.40 g, 56%). The product was characterized by ^{1}H NMR, IR and ^{13}C NMR spectral studies.

^{1}H NMR: ($CDCl_3$), δ (ppm/TMS): 0.9-1.0 (t, 3H, -C$\underline{H}_3$); 1.2-1.65 (m, 18 H, -C$\underline{H}_2$); 2.2-2.3 (t, 2H, -CO-C$\underline{H}_2$); 2.7-2.78 (m,1H, (-C$\underline{H}$-SH); 3.2-3.8 (dd, 2H, -NH-C$\underline{H}_2$-CH-SH); 3.8-3.85 (s, 1H, -N$\underline{H}$-, -S$\underline{H}$-); 4.05-4.14 (t, 2H, -O-C$\underline{H}_2$) 6.65-8.07 (m, 4$\underline{H}$, Aryl)

^{13}C NMR ($CDCl_3$, ppm): 14 ($\underline{C}H_3$-CH_2-); 20.9(CH_3 of aniline); 18.9-31 (-CH_2-); 34.5 (-$\underline{C}H_2$-C=O); 36.5 (-CH-SH); 55.2 (CH_2-NH-) 64.2 (-$\underline{C}$HO-C=O); 149 (aromatic C-NH-); 173.5 (-COO-);

FT-IR (neat, cm^{-1}): 3448 (-OH, -NH); 1603 (-N-H bending); 2927 (C-H stretching)

2. Resuls&discussion

In the present study, ionic liquid mediated synthesis of 10-undecenoic acid based amino thiol derivatives was carried out starting from alkyl epoxy undecanoates (C_1 and C_4). Alkyl epoxy undecanoates were reacted with ammonium thiocyanate in ionic liquid 1-methyl Imidazolium tetrafluoro borate-H_20 (2:1) solvent system to produce the corresponding epithio undecanoates at 60 ºC. After complete conversion of epoxy to epithio ring, reaction temperature was raised to 80-90 ºC and epithio ring was opened with aniline, para toluidine, amino pyridine and *p*-nitro aniline to obtain alkyl-10-mercapto-11-aminoundecanoates monitoring by TLC analysis for the disappearance of episulphide group (**Scheme 1**). All the reactions were completed within 20-24 h, affording alkyl 11-anilino-10-hydroxy undecanoates with an isolated yield of 48-56 %. Although the conversion of mercapto-aminol is high, the obtained isolated product yield is less comparatively. It is due to the free -S-H group which binds very easily with the silica gel during the purification process. The synthesized products were characterized by ^{1}H NMR, IR, GC and GC-MS analysis and screened for their cytotoxic properties.

Table 1:Cytotoxic activity of alkyl-10-mercapto(11-amino) undecanoates

Test compounds	IC_{50} in μM			
	A549	MDA-MB-231	MCF-7	HeLa
HSUME	-	1.707	0.974	-
HSUB	-	15.0	16.71	-
NHSB	-[a]	1.258	44.78	-
PAHSB	-	20.98	34.98	13.87
PHSTB	-	17.09	15.87	11.98
Doxorubicin	0.451	0.501	1.05	1.21

HSUME: Methyl-10-mercapto-11-(phenylamino) undecanoate
HSUB: Butyl-10-mercapto-11-(phenylamino) undecanoate
NHSB: Butyl-10-mercapto (11-*p*-nitro anilino) undecanoate
PAHSB: Butyl-10-mercapto (11- pyridin-2-ylamino) undecanoate
PHSTB: Butyl-10-mercapto (11- p-tolylamino) undecanoate

Methyl-10-mercapto (11-anilino) undecanoate had shown excellent cytotoxicity towards both MDA-MB-231 and MCF-7 (breast cancer cell lines), which was comparable to doxorubicin (standard), while butyl-10-mercapto (11-*p*-nitro anilino) undecanoate exhibited good cytotoxicity towards MDA-MB-231 and moderate cytotoxicity towards MCF-7. In addition, butyl-10-mercapto (11-anilino) undecanoate, butyl-10-mercapto (11-p-tolylamino) undecanoate, butyl-10-mercapto (11- pyridin-2-ylamino) undecanoate exhibited moderate cytotoxicity towards both MDA-MB-231 and MCF-7 cell lines (Table 1). These compounds were selective towards breast cancer cell lines and showed no cytotoxicity towards A549 (alveolar) where as two compounds butyl-10-mercapto (11-anilino) undecanoate, butyl-10-mercapto (11- p-tolylamino) undecanoate, butyl-10-mercapto (11-pyridin-2-ylamino) undecanoate, exhibited moderate cytotoxicity towards HeLa (cervical) cancer cell lines.

1. Conclusions

In conclusion, synthesis of five novel alkyl 10-undecenoate based β- amino thiols is reported. Alkyl epithio undecanoates were subjected to ring opening reaction with different amines and the resulting compounds were characterized and screened for anticancer activity where two derivatives showed excellent activity towards breast cancer cell lines, while rest of the compounds exhibited moderate activity specifically breast cancer cell lines. Only two compounds showed moderate activity towards cervical cancer cell lines. None of the compounds exhibited anti cancer acitivity towards A549 (Alveolar) cell lines.

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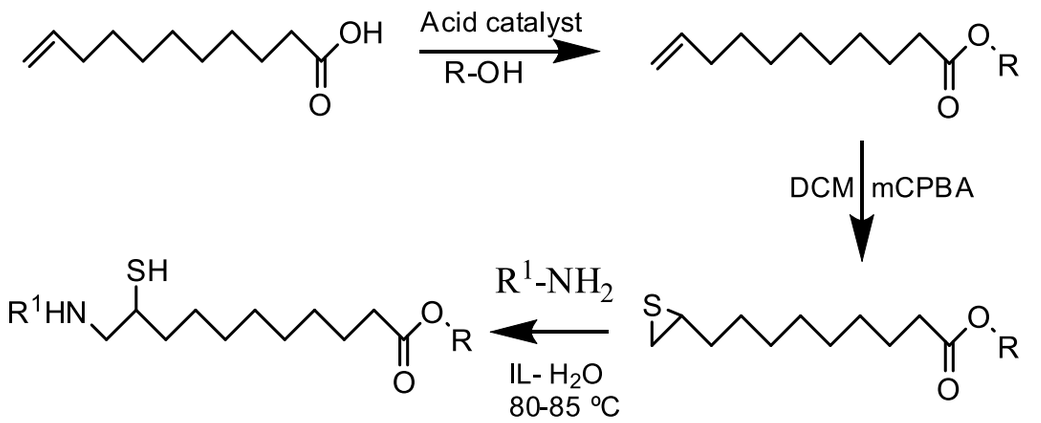

Where R= Methyl, butyl

R^1= Phenyl, p-nitrophenyl, p- toluidine, pyridin-2-ylamine

Scheme 1: Synthesis of alkyl11-amino-10-mercapto undecanoates

Comparative study of catalytic activity from different waste sources for Biodiesel synthesis

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Key words: Snail shell; Marble slurry; Transesterification; Reusability.

Abstract

This paper aims to compare the catalytic activity of differentsolid catalyst derived from calcined snail shell (CSS), KOH loaded snail shell (KOH/SS), Calcined Marble slurry (CMS) and Marble slurry derived Hydroxyapatite (HAP) for biodiesel synthesis from soybean oil by the transesterification reaction.Material characterization such as XRD and FTIR was done for the catalysts. Catalytic activities of developed catalysts were also tested by Hammet indicator method. The experimental outcome revealed that among all catalyst, 96% highest biodiesel yields found by using KOH/SS under reaction parameters such as reaction time of 3.5 h, methanol to oil molar ratio of 9:1 reaction temperature of 65ºC and catalyst loading of 4wt%. Reusability of the developed catalysts was also investigated and found that catalytic activity changes up to 5 times for all the catalysts.These results concluded that all developed catalysts from waste sources have the promising ability for biodiesel production by the transesterification reaction. Among all the used catalyst in this study, the KOH/SS catalyst exhibits stronger basicity and catalytic activity than another catalyst for biodiesel synthesis.

1. Introduction

Recently renewable energy source like biodiesel has been gaining much attention worldwide due to its renewability, biodegradability, nontoxic and environment friendly nature (Saluja et al., 2016; Rodoinova et al., 2016). Biodiesel is produced from edible and nonedible oils by the transesterification reaction in the presence of the homogeneous or heterogeneous catalyst and alcohol. A homogeneous catalyst such as potassium hydroxide, sodium hydroxide, sulphuric acid gives high biodiesel yield with shorter reaction time (Gupta et al., 2016) but the use of homogeneous catalyst increases the cost of biodiesel in purification step with water and also very difficult to separation. So heterogeneous catalyst was found to be the better catalyst with many advantages such as noncorrosive, reusable, high rate of reaction and no need of water washing step over a homogenous catalyst (Islam et al., 2013). Among the various solid heterogeneous catalysts, CaO is one of the best heterogeneous catalysts with superior catalytic activity in transesterification reaction (Gupta and Agarwal 2016(a); Colombo et al., 2017). But the use of commercial CaO increases the cost of overall biodiesel synthesis and also required special skill to operate it (Marinković et al., 2016). Therefore many researchers have been gaining much attention to exploring CaO as a heterogeneous catalyst from waste sources such as shells, rice husk, dolomite, sea shell sand and marble slurry(Gupta and Agarwal 2016(b)). Various studies have been carried out for biodiesel production from different waste sources such as shells, rice husk, dolomite, sea shell sand and marble slurry (Gupta and Agarwal, 2016; Muciño et al., 2014; Gupta et al., 2017), but nobody yet tried to do comparative study on biodiesel production from different waste sources. Hence the purpose of this work was to evaluate various waste sources such as snail shell (SS), KOH impregnated snail shell (KOH/SS), marble slurry (MS) and hydroxyapatite (HAP) from MS as solid base catalysts for transesterification of soybean oil. Comparative studies of different developed catalysts for biodiesel production were further explained.

2. Materials and method

2.1 Materials

Soybean oil was obtained from local market of Jaipur (India).Snail shells and Marble slurry was collected

from Ram Ganga shore in Bareilly and Jaipur (India) respectively. Potassium hydroxide (KOH), Potassium dihydrogen orthophosphate [KH_2PO_4] and anhydrous methanol with 99% purity were of analytical reagent grade purchased from Merck, Germany.

2.2Catalyst Preparation

Firstly, the collected SS and MS were cleaned with water 2-3 times for separating dirt and undesired material and subsequently dried at 120ºC for 10 h. After drying, shells and MS were smashed in a grinder to the small size and calcined at the temperature of 850ºC for 3 h in the muffle furnace. The Calcined material named as CSS (calcined snail shell) and CMS (calcined marble slurry) was then cooled in a desiccator and then utilized as a heterogeneous base catalyst for biodiesel production. Afterward, KOH/SS catalyst was prepared by wet impregnation method (Gupta *et al.,* 2017). Typically 4 g of calcined SS powder (4 g), distilled water (40ml) and potassium salt compound (1 g) was mixed properly by continuous stirring for 1-2 h. The prepared solution was separated from water using a filter paper and dried in a hot air oven at 106ºC and recalcined at 600ºC for 2 h.

The basic heterogeneous catalyst synthesized from MS, i.e., HAP, was developed by a process explained in the literature (Ibrahim et al., 2015). Typically, a suitable amount of MS and concentrated nitric acid was added with continuous stirring which produces calcium nitrate. The chemical reaction is as follows:

$$CaCO_3 + 2HNO_3 \longrightarrow Ca(NO_3)_2 + H_2O + CO_2 \quad (1)$$

MS Nitric Acid Calcium Nitrate

Then, 50 mL of 0.06 M dilute phosphate solution was prepared and added at the rate of 200 mL/h and made to react with 50 mL of 0.1 M calcium nitrate solution under continuous stirring as follows:

$$5Ca^{2+} + 3PO_4^{3-} + OH^- \longrightarrow Ca_5(PO_4)_3OH\ (HAP) \quad (2)$$

Before the start of each reaction, the pH (10.5) of the solution was maintained by using NH_4OH solution. The final product was washed with distilled water and then after with ethanol to remove traces of water. Then dried in a microwave oven (at 80 °C) for 12 h and sintered (at 700 and 950 °C) in an oven for 2h.

2.3 Catalyst Characterization

XRD (X-ray diffraction) technique was used to observe the crystalline characteristics of all catalysts using X-Pert pro powder panalytical instrument. The FT-IR (Fourier Transform Infrared Spectroscopy) spectra were detected on FT-IR spectrometer (Perkin Elmer spectra two) in the range of 400–4500 cm^{-1} by KBr pellet method (for the sample preparation). The Hammett indicator was used to know the basicity and the basic strength (H_) of the catalysts respectively.

2.4 Transesterification reaction

The transesterification reaction was performed in two neck round bottom flask connected with the thermometer, a reflux condenser, and magnetic stirrer. The preheated soybean oil (100ml) was added into the flask which already containing methanol and catalyst under continuously stirring. The transesterification reaction was taken out at the constant temperature of 65ºC with a reaction time (3.5 hours), methanol to oil molar ratio (9:1) and catalyst concentration (4wt%).After Completion of the reaction, the cooled product mixture was transferred to the separating funnel for phases separation. The solid base catalyst was removed from the reaction product via centrifugation at 5000 rpm for 20 min. The upper phase(biodiesel) was separated and stored for further transesterification reaction, and lower part consisted of glycerol and solid catalyst.

Biodiesel yield was characterized by GC analysis using the European regulated procedure EN-14103 by dissolving the ester layer (250mg) in n-hexane(5 ml) solution of internal standard methyl heptadeconate (10gm/L of C_{17} ester in hexane). The yield of biodiesel was calculated by the equation (3).

$$\text{Yield\%} = \frac{\sum A - AMH}{AMH} \times \frac{CMH - VMH}{m} \times 100\% \quad (3)$$

where A is the total peak area from methyl ester, AMH the area of methyl heptadeconate, which response factor is equal to those of ester; CMH the concentration in mg/ml of the methyl heptadeconate (10 mg/ml); VMH the volume in ml of the methyl heptadeconate solution (5 ml); m is the weigh in mg of the sample (250 mg) (Suryaputra *et al.* 2013).

3.Results and Discussion

3.1 Soybean Oil analysis

The properties of soybean oil (Table I) were analyzed as per ASTMD6751 Standards.

Table I Properties of soybean oil.

Soybean oilproperties	Measured Value
Density (gm/ml)	0.89
Kinematic viscosity at 40^{0}C (Cst)	16.45
Acid value(mg of KOH/gm of oil)	1.15
FFA	0.57
Flash point $^\circ$C	150
Fire point $^\circ$C	180
Saponification value(mg of KOH/gm of oil)	196
Molecule weight (gm/mol)	863.60
Ester value (mg)	194.88
% of glycerol	10.65

3.2 Characterization of the developed catalysts

3.2.1 XRD and FTIR analysis

Figure 1(a) and 1(b) shows XRD pattern of uncalcined SS, calcined SS, KOH/SS, uncalcined MS, calcined MS and HAP. The uncalcined SS and MS mainly consist of calcium carbonate as indicated by different peaks at 2θ around 27.22°,31.06°,33.10°,36.09°,37.82° and 45.82°(Gupta and Agarwal 2016(a)).After thermal treatment of SS and MS, most of these peaks disappeared and intense sharp peaks of CaO were observed at 2θaround 32.12°,37.280° and 53.76° (Yin *et al.,* 2016).In XRD plot of KOH/SS, the peaks at 32.13°, 37.29° and 53.78°observed which indicate the occurrences of reaction between KOH and CaO during the calcination.XRD patterns of HAP shows the two highest peaks at 31.02°and 50.61°which is characteristics of HAP and closed to HAP standard compared with literature (Pujiyanto *et al.,* 2014).These result indicated that the SS and MS had been successfully synthesized into active basic catalyst before transesterification reaction.

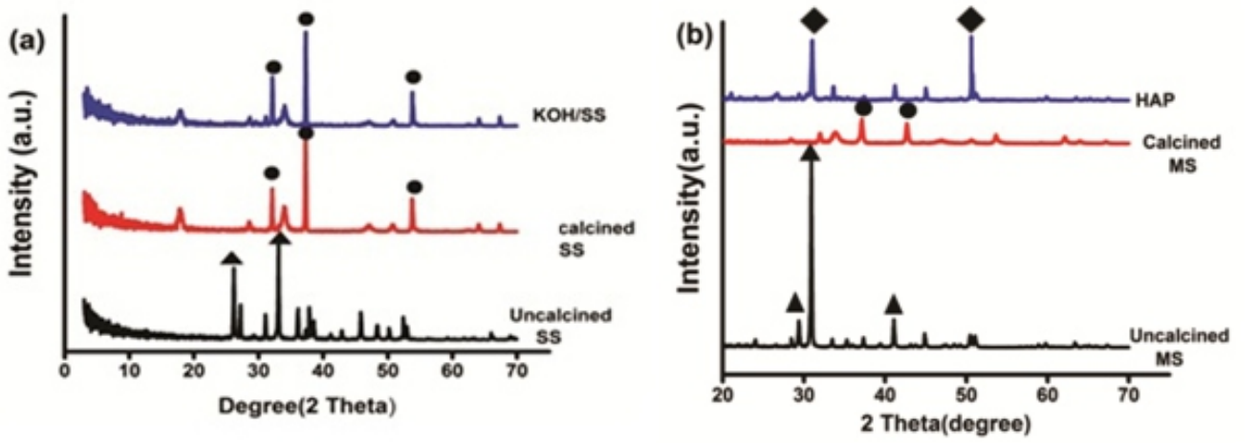

Figure 1 XRD curve for (a) uncalcined SS, calcined SS, KOH/SS (b) uncalcined MS, calcined MS and HAP Symbols:

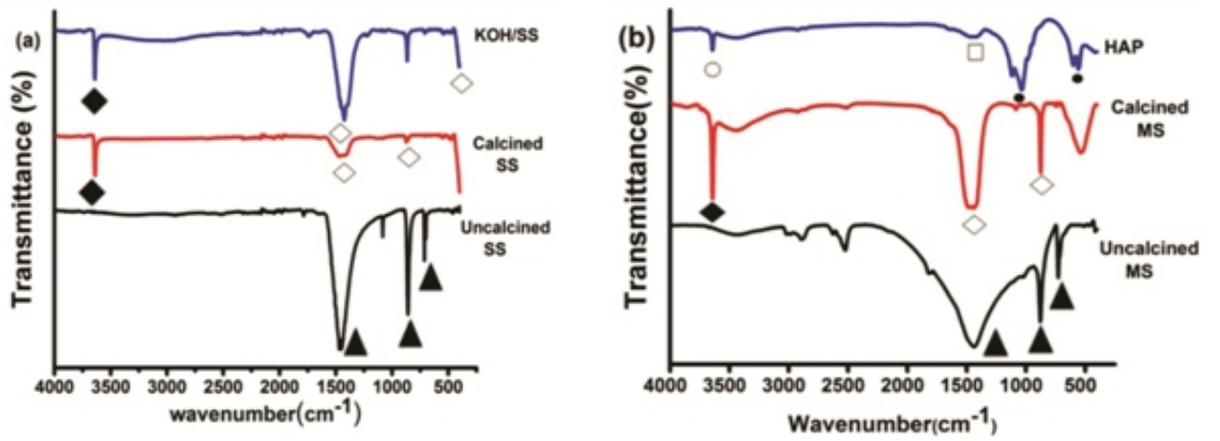

Figure 2 FTIR curve for (a) uncalcined SS, calcined SS, KOH/SS (b) uncalcined MS, calcined MS and HAP Symbols: (= CaCO3, ◇=CaO, =Ca(OH)2, ●= PO43- , ❑ =CO32- , ○= OH-1)

Figure (2) shows the FTIR spectra of uncalcined SS, calcined SS, KOH/SS,uncalcined MS, calcined MS and HAP respectively. The strong peaks at 1454 cm^{-1} were attributed to carbonate minerals in SS and MS. Other two peaks at 712 cm^{-1} and 855.99 cm^{-1} also shows the mode of carbonate groups. After calcination of SS and MS at 850°C, the intensity of these bands decreases due to thermal decomposition of $CaCO_3$ and formation of CaO and $Ca(OH)_2$. One sharp band appears 3640cm^{-1} due to formation of basic OH group attached to the calcium atoms i.e. $Ca(OH)_2$ and band at 1471 cm^{-1} shows CaO group (Gupta *et al.,* 2017). The band at 3640cm^{-1} in the spectrum of KOH/SS catalyst was assigned to molecular CO_2 adsorbed by the basic hydroxyl groups in the catalyst. Other peak appears in the spectrum of KOH/SS indicated that basic hydroxyl groups was generated via the reaction of KOH and $CaCO_3$ by calcination(Laio *et al.,* 2013). In FTIR plot of HAP, Hydroxyl stretch is observed at 3643 cm^{-1} in the spectra of hydroxyaptite as shown in figure 2(b). There were also bands at 103.04 cm^{-1} and 1420 cm^{-1}, which were assigned to the phosphate group and the carbonate, group (Pujiyanto *et al.,* 2014).

3.2.2 Basicity measurement

Tanabe and Yamaguche *et al.,* 1963 reported the Hammett process to know the basic strength of the catalyst. The base strength of the catalyst and total basicity of the catalysts has been presented in Table II. After the investigation, the pure SS and pure MS were found to be the weaker base than CSS and CMS. Subsequent modification in snail shell and marble slurry, basic strength of KOH/SS and HAP increase with total basicity and KOH/SS found to be the better base catalyst than another catalyst.

Table II Basicity determination of catalysts

Catalyst	Basic strength(H_)	Total basicity (mmoles/gm)
SS	9.8< H_<12.0	3.23
CSS	9.8< H_<17.2	9.70
KOH/SS	9.8< H_<18.4	12.6
MS	7.2< H_<9.8	2.90
CMS	9.8< H_<15.0	8.73
HAP	9.8< H_<17.2	11.23

Table III shows the comparative study of the waste derived solid base catalyst concerning reaction conditions for transesterification reaction. It can be justified that small amount of catalyst(4wt%), less reaction time(3.5 h) and lower methanol: oil(9:1) is sufficient for better biodiesel yield as compared to the other waste catalyst.

Table III Comparative study of various heterogeneous solid catalysts from waste sources.

Catalyst	Experimental conditions			Conversion (%) Or Yield (%)	References
	Catalyst loading (wt%)	Oil :methanol	Time (h)		
Waste marble derived Ba/CaO	3	1:9	3	88	Balakrishnan *et al.,* 2013
K/HAP	8	1:9	1.5	96.4	Wei *et al.,* 2009
Waste animal bone based HAP	20	1:18	4	96.78	Gandure, *et al.,* 2017
CaO from Egg shells	3	1:9	65	>95	Joshi *et al.,* 2015
CaO from Egg shells	10	1:18	60	95	Viriya-Empikul *et al.,* 2010
KI loaded oyster shell	3.5	1:6	60	85	Jairam *et al.,* 2012
KBR laoded eggshell	3	1:12	65	82.48	Mahesh *et al.,* 2015
CaO from Scallop shell	5	1:6	65	86	Sirisomboonchai *et al.,* 2015
CaO from Snail shell(SS)	4	1:9	3.5	90	Present study
KOH/SS	4	1:9	3	96	Present study
Marble slurry	4	1:9	3	88	Present study
Marble slurry based HAP	4	1:9	3	93	Present study

3.3 Biodiesel characterization

The Yield of biodiesel was analyzed by using GC analysis as shown in Figure 3 which shows the typical chromatograms for the biodiesel produced via soybean oil and developed catalyst. This chromatograph represent the different ester peaks such as palmitic acid (C16:0) at 19.5 minute, stearic acid (C18:0) at 24.6 minute, oleic acid (C18:1) at 25.2 minute, linoleic acid (C18:2) at 26.5 minute, and linolenic acid (C18:3) at 20 minute.

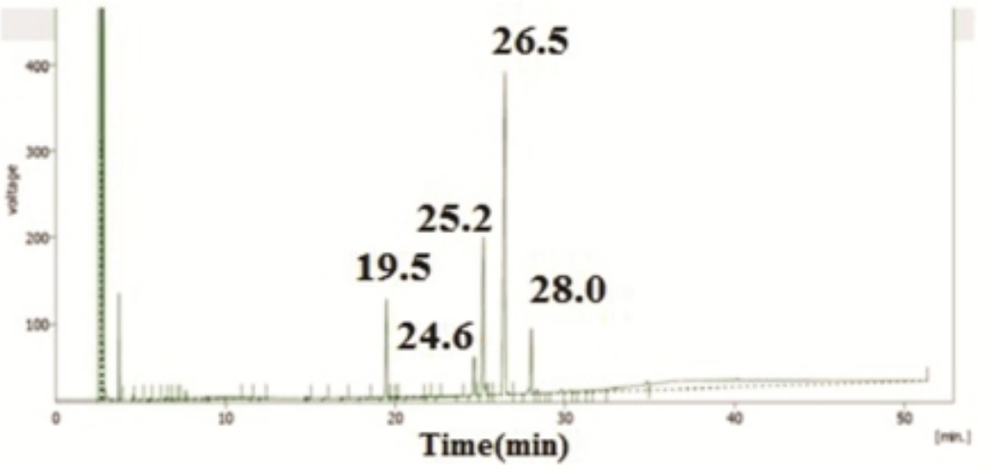

Figure 3 GC analysis of biodiesel from soybean oil by using KOH/SS.

Soybean oil biodiesel propertieswere analyzed and were compared as per ASTM D6751 Standard which has shown in Table IV.

Table IV. Properties of soybean biodiesel

Properties	Soybean oil biodiesel from CSS	Soybean oil biodiesel from KOH/SS	Soybean oil biodiesel from CMS	Soybean oil biodiesel from HAP	ASTM Standard
Yield (%)	90	96	89	93	-
Density(gm/ml)	0.878	0.889	0.878	0.889	0.86-0.90
Kinematic viscosity at 40°C	5.2	4.721	5.5	4.9	1.9-6.0
Cloud point, °C	6	3	6.6	4.2	-3 to 12
Flash point, °C	160	145	169	150	130-170

4. Reusability study of the catalyst

The reusability study of the catalyst is significant for the industrial use. To evaluate the reusability of all the catalyst, the transesterification reaction mixture was separated by using filter paper to obtain the used solid catalyst and cleaned with methanol 2-3 times to remove glycerol traces on it. Subsequently, the catalyst was calcined at the high temperature in the muffle furnace for 3 h to activate the catalyst. The activated catalyst was utilized for more study of reusability with similar reaction conditions.The catalytic activity of CSS, KOH/SS, CMS, and HAP decreased clearly with in 5 runs as shown in Figure 4. The decrease biodiesel yield may be due to the reduced surface area or pore blockage and also the structural change of the catalyst.

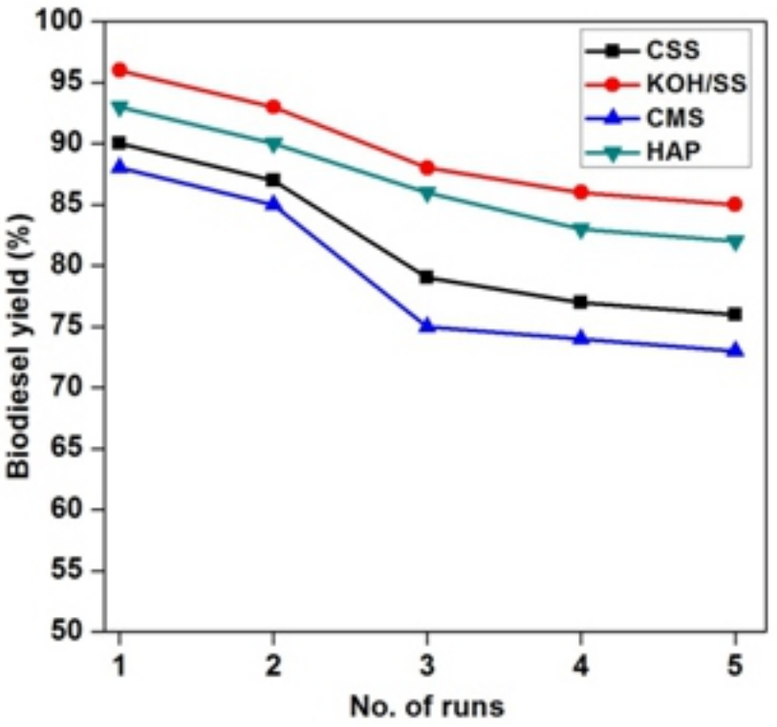

Figure 4 Reusability study for catalyst.

5. Conclusion

This study revealed that snail shell and marble slurry derived catalyst could be a promising catalyst for biodiesel production.KOH/SS exhibited stronger catalytic activity and basicity as compared with the other developed catalyst by characterization results. KOH/SS showed high biodiesel yield (96%) and better biodiesel properties under reaction conditions such as 3.5 h reaction, 9:1 methanol to oil molar ratio and 4wt% catalyst loading among all the developed catalysts. The KOH/SS catalyst from snail shell exhibits excellent catalytic activity and stability than marble slurry derived catalyst in the transesterification reaction.

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FOOD GRADE LUBRICANTS- A TECHNO COMMERCIAL REVIEW (Part I)

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INTRODUCTION

Food grade lubricants refer to lubricants that are used to maintain the food processing machinery, i.e. it protects the tools and machines from wear and tear. However, since many of them come into direct contact with food, these lubricants used in these machineries need to be different from the usual machine lubricants. In other words, they should not render the food unfit for consumption. Pharmaceuticals, food, beverages, meat processing and production consists of a vast array of equipment, compactor, pumps, mixers, oven chain drives, conveyor systems, compressors, and many other units and systems. All of it needs to be lubricated properly to keep working. With growing concern over the food and pharmaceutical products, processors need to comply with various regulatory frameworks like the Hazard Analysis & Critical Control Points (HACCP). The standards vary from country to country. However, these are internationally accepted standards which are followed by food grade lubricant manufacturers. For instance, the non-profit public health and safety company, NSF International, along with USDA, has set standards for various food grade lubricants.

As food safety laws are proliferating, the demand for food grade lubricants is increasing with the growth of processed food industry worldwide. Also drug authorities are insisting on incidental contact safety. All the major companies have to adhere to safety laws in order to avoid product recalls and penalty fees. The trend is more prominent in developed countries such as the U.S., U.K, Canada, Norway, Germany, France and Italy. The food and drug authorities in developing countries such as China, Brazil, India and South Africa etc are tightening the safety norms which will result in higher demands for food grade lubricants in days to come.

A) INDIAN LUBRICANT INDUSTRY OUTLOOK

India occupies third position in the global lubricants market after US and China, accounts for about 5.5% of the global automotive lubricants and 4% of the industrial lubricants market by volumes.

The domestic lubricant industry was a Rs 44,750 crore (US$ 7.1 billion) market in the year 2016-17 with volumes of 3.4 million tons, growing at 2.5 to 3.5 % CAGR in the past few years. Where as global lubricant was 46.5 million tons, valued at US$ 151.5 where as Asia Pacific lube market was at 10.8 million tons. It is projected that in year 2020-21 that Indian Lube market would be 3.75 million tons, valued at Rs.49,150; (US$ 7.8 billion) and global market would be around 48.5 million tons valued at (US$ 170). The lube market has been summarized in table 1

Table 1
GLOBAL AND INDIAN LUBE MARKET

	2016-17		2020-21	
	VOLUME MMT	VALUE BILLION US $	VOLUME MMT	VALUE BILLION US $
GLOBAL	46.5	151.5	48.5	170.0
INDIA	3.4	7.1	3.75	7.8

The automotive lubricants segment, currently comprising oil PSUs, multinational companies and a number of small and regional players, is highly fragmented.

Over the recent years, consumer awareness about usage of lubricants has improved leading to an increased demand for high performance products. The domestic lubricants industry is witnessing stiff competition among players leading to an overall shift in perception of lubricants market from a volume driven market to a value driven market.

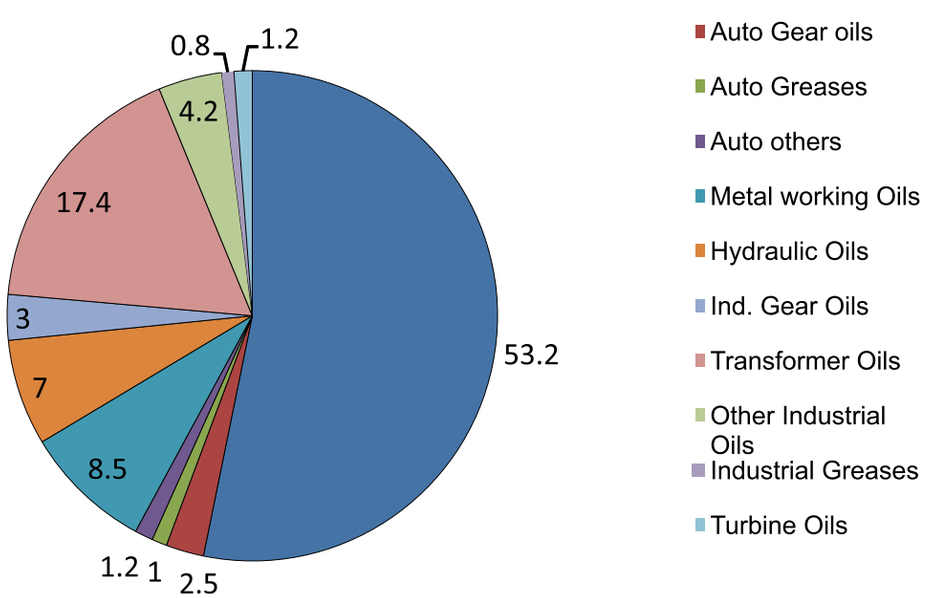
Based on applications, lubricants are broadly classified as automotive lubricants and industrial lubricants. Engine oil, gear oil, greases and hydraulic and transmission fluid are the products under automotive lubricant segment. Industrial lubricants comprise process oil, general industrial oil, metalworking fluids, industrial engine oil and greases. Table 2 gives segment wise India Lube market.

Table 2

INDIAN LUBE MARKET; APPLICATION WISE

END USE SEGMENT	% SHARE
Auto Engine oils	53.2
Auto Gear oils	2.5
Auto Greases	1.0
Auto others	1.2
Metal working Oils	8.5
Hydraulic Oils	7.0
Ind. Gear Oils	3.0
Transformer Oils	17.4
Other Industrial Oils	4.2
Industrial Greases	0.8
Turbine Oils	1.2

% SHARE OF VARIOUS TYPES OF LUBRICANTS



B) NEED FOR FOOD GRADE LUBRICANTS

In many manufacturing and packing operations, food and pharmaceutical products there are chances of incidental contact, some time there may be direct contact with the lubricants. There are numerous instances of contamination and product re-calls.
Some of the documented recalls are as follows;

- 1996, 3 tons of turkey sausages were recalled because of grease contamination.
- I998, almost 500,000 pounds of smoked ham were recalled because of gear oil contact.
- 2002, a US-based soft drink producer recalled over 3,000 cases of product that contained gear lubricant.
- 2002, Australian authorities recalled soft drinks because of mineral oil contamination
- 2003, A Danish milk powder manufacturer recalled 1,100 tons of product that was contaminated by lubricant leaking through a worn axle in a gearbox.

Indian food and pharmaceutical industries are aware of the possible hazard. Especially for multinational companies fulfilling HACCP (Hazard Analysis and Critical Control Points) requirements is top priority.

At present there are no regulations set up either by Drug authorities or FSSAI. But both the bodies are aware of hazard of possible incidental contact with lubricants. It is expected that shortly some regulations will be introduced. BIS is in process of framing standards for food grade lubricants.

C) END USER INDUSTRY FOR FOOD GRADE LUBRICANTS

- Food processing
- Edible oil manufacturing and processing
- Confectionary
- Fruit & vegetable processing
- Sugar
- Dairy
- Bakery
- Beverages
- Cosmetics
- Pharmaceuticals; (Finished dosage forms & Active ingredients)
- Animal feed
- Tobacco, et

D) CLASSIFICATION OF FOOD GARDE LUBRICANTS BASED ON DELIVERY SYSTEM

The broad classification of Food grade lubricants is;

1. Lubricating greases
2. Lubricating oils
3. Lubricating sprays.

E) EXAMPLES OF APPLICATIONS OF FOOD GRADE LUBRICANTS

GREASES	LUBRICATING OILS	SPRAYS
Roller & plain bearings	Circulating lubrications	General lubrications
Hinges	Hydraulics	Ball & Roller bearings
Slide ways, slide rolls	Planetary/ Spur/ Bevel gears	Transport & drive chains
Roller	Transport & drive chain	Rust dissolvers
Cam disk	Vacuum pumps	Cleaners
Gear Rack	Screw compressors	
Central lubrication	Tablet compacting	
Threaded spindle		
Small gears		

F) INTERNATIONAL REGUALTION FOR FOOD GRADE LUBRICANTS

United States

- Heavily regulated by USFDA with National Sanitation Foundation (NSF), an international product certification body.
- Products must be formulated in accordance with Title 21 Code of Federal regulations (21CFR) 178.3570.
- Ingredients in the lubricant must be listed as safe in (21CFR) 178.3570 where there is a list of acceptable components including oils, antioxidants, surfactants, etc., along with use limitations.
- Another option to comply with the regulation could be through an Approved contact notification from the FDA.

European Union

- No formal regulation for lubricants, Typically the U.S. compliant requirement is accepted.
- Publishes / Provides information on food safety & ingredients

Canada

- The Canadian Food Inspection Agency (CFIA) enforces that country's regulations. It maintains a list of CFIA-approved chemical compounds , approves & accepts contact material

China

- The Food Safety authority; "Food Hygiene Law of the People's Republic of China" This law doesn't cover products but instead covers the premises, processing facilities and Environments produced. It doesn't specify hygiene standards for lubricants, it focuses more on potential harmful substances being added into the products that could contaminate the final lubricant.

Australia

In the past the Australian Quarantine and Inspection Service (AQIS) operated a program similar to the one in Canada, reviewing and listing products as compliant and mirroring the U.S. criteria for ingredient review. Lubricants with incidental contact are termed Lubricants Type A. Two years ago they also suspended their acceptance review program.

Asia-Pacific

- Several countries are strengthening their food safety regulations and expanding laws for food processing and focus further on the lubricant sector, but no regulations exist now.

India

- Neither Food Safety & Standards Authority of India (FSSAI) nor Central Drugs Standard Control Organization have formulated any regulations / guidelines on Food Grade Lubricants
- FSSAI , under Section 10, 16 (1)(i) (c) and 18(1) (2) (b)(c), has established a Risk Assessment Cell for assessment and mitigation on food safety
- CDSCO issues notifications / circulars / publications on various issues including risk assessment, but rarely on incidental contact.

G) REGISTRATION CATEGORY CODES FOR LUBRICANTS AND INGREDIENTS

H1 : Main category, as food grade lubricants for incidental contact. Not intended for direct, intentional contact with food. Lubricants and anti-rust agents, or as release agents on gaskets or seals of tank closures, where there is possibility of incidental food contact must be formulated in compliance with CFR, Title 21, Section 178.3570 and other sections. (Limit 10 ppm)

HX-1: Category for the ingredients that goes into forming an H1 lubricants, pre-screened components and additives meeting the requirements for H1 lubricants. 470 registered HX-1 ingredients by more than 80 companies.

H2 : Category for noncontact lubricants, those that should have absolutely no contact with food. These lubricants are intended to be used somewhere else in a food production facility but not on the line that processes food. The registration process performs a general overview to make sure there are no

carcinogens in the product and other general health and safety areas. When manufacturers register these lubricants, food manufacturers have added reassurance that a third party has reviewed them.

HX-2: Category for the ingredients that goes into forming an H2 lubricants, pre-screened components and additives meeting the requirements for H2 lubricants

3H: Products are used on grills, loaf pans, cutters, boning benches, chopping boards, or other hard surfaces in contact with meat and poultry food products to prevent food from adhering during processing. Products containing edible oils such as corn oil, cottonseed oil and soybean oil, mineral oil complying with 21 CFR, Part 172, Section 172.878, and other GRAS substances may be acceptable upon review by NSF. In addition, de-foaming agents complying with 21 CFR Section 173.340 (a) (1) and (a) (2) may be acceptable.

H3: Soluble oils that are used to treat hooks, trolleys and other similar equipment that may contact food or edible products, used to prevent rust on hooks, trolleys and similar equipment. Treated equipment which contacts edible products should be cleaned by washing or wiping before putting the equipment back into service. Products may be composed of any of the following:

- Edible oils (corn, cottonseed, soybean) complying with 21 CFR, Section 172.860;
- Mineral oil complying with 21 CFR Section 172.878;
- GRAS substances complying with 21 CFR Parts 182 (multi-purpose only) or 184.

HT-1: Heat transfer fluids in primary and secondary heating and cooling systems in food processing facilities. Preparations permitted for use as heat transfer fluids, where there is possibility of incidental food contact must be formulated in compliance with CFR, Title 21, Section 178.3570 and other sections referenced therein; ingredients may also comply with CFR Title 21 part 172.

National Sanitation Foundation (NSF) International operates a product registration & certification program and works with clients in 48 countries around the world

H) IDENTIFICATION BASED ON RELIGIOUS REQUIREMENTS

- **Halal**: Identification of products which are classified as "Halal" (legal) according to the Islamic dietary regulations.
- **Kosher**: Identification of products which are classified as "Kosher" (suitable, clean) according to the Jewish dietary laws.

I) ISO 21469 CERTIFICATION, AN INTERNATIONAL STANDARD FOR FOOD GRADE LUBRICANTS

ISO 21469:2006 is a voluntary product certification for lubricants that is internationally accepted. It specifies hygiene requirements for the formulation, manufacture, use and handling of lubricants which, during manufacture and processing, can come into incidental contact (e.g. through lubrication, heat transfer, load transmission, or the corrosion protection of machinery) with products and packaging used in the food, food-processing, cosmetics, pharmaceutical, tobacco, beverages, edible oil processing animal feed industries. It is not applicable to substances used as product additives or to those in direct product contact, but confines itself to hygiene without addressing occupational health and safety matters

The lubricants which are certified by NSF can display the official NSF ISO 21469 certification mark on their packaging and labels.

PRODUCT REGISTRATION PROGRAM

NSF International operates a product registration program. At present, It works with clients in 48 countries around the world. Audits are performed initially for certification and then annually to maintain certification

FORMULATION REVIEW STEP

All product formulations are reviewed to verify that ingredients are acceptable. The label is reviewed to confirm that the end use of product is specified, there is traceability on the packaging, to verify correct use of the certification body mark, the shelf life is indicated and there are no false claims made..

RISK ASSESSMENT STEP

The certification is end-use specific, so both the

involved in the risk assessment step. The manufacture has to identify potential points of risk within the product and has to offer risk mitigate plan.

FACILITY AUDIT STEP

The products are evaluated for potential chemical, physical and biological hazards from a risk assessment standpoint. The facilities are using good manufacturing practices and haven't missed any points brought up during the risk assessment and risk mitigation. Facility is evaluated for hygienic facility environment and there are raw material testing, release and quarantine procedures in place. The audit also verifies that the facility maintains a standardized quality procedure.

H) GLOBAL & INDIAN MARKET FOR FOOD GRADE LUBRICANTS

GLOBAL MARKET SIZE

The global market size in year 2016 for food grade lubricants was 46,000 tons. It is projected that it will reach 65,000 tons by 2021.. In terms of value, the market for food grade lubricants is projected to grow at 7.0% from USD 183 Million in 2016 to USD 266 Million by 2021.

YEAR	QUANTITY, TONS	REVENUE, Million USD
2016	46,000	183
2021 (Projected)	65,000	266

INDIAN MARKET FOR FOOD LUBRICANT

Based on import data as well production by local food grade lubricant manufacturers, by conservative estimation, the 2016 Indian market was 3400 tons. It is projected it will reach 5400 tons by 2021 and by value terms it would be Rs. 2970 million rupees, thus will grow by almost 10%. .

YEAR	QUANTITY, TONS	REVENUE, Million Rs.
2016	3400	1750
2021 (Projected)	5400	2970

The higher growth rate for Indian market is attributed to following growth drivers.

GROWTH DRIVERS FOR INDIAN MARKET

- Growing pharmaceutical market
- Growing food processing industry
- Growing meat processing industry
- Growing confectionary industry
- Presence of number of multinational food companies, especially US based
- Awareness in user industry
- Proposed Regulations

End of Part I

Part II covers, formulations, various base oils and additives used, patents and other published research work

Research Roundup For October –December 2017

Physicochemical and thermal characterization of seed oil from Mexican mamey sapote(Pouteria sapota)

Hernández-Santos et al. performed physicochemical characterization of native mamey sapote seed oil [*Pouteria sapota* (Jacq.) This oil showed good oxidative stability as it had low peroxide, free fatty acid, and *p*-anisidine values. The main fatty acids present in the oil were palmitic, stearic and oleic acid, constituting five major triacylglycerides families: PLP, POP, StOO, POSt and StOSt. Crystallization and melting points of the oil were −37.7 and 23.84 °C, respectively [J. Amer. Oil Chem. Soc. 94, 1269–1277 (2017)]. The oil had higher SFC when the temperature was lower than 10 °C. X-ray diffraction patterns showed that prolonged storage times lead to the formation of β crystals. Micrographs showed granular crystals (91–105 μm), with needle edges similar to cocoa butter. In addition, mamey sapote seed oil can be used in confectionery products or as a possible substitute for cocoa butter to improve and obtain good-quality products.

Green Synthesis of Double Long-Chain Diglycerol Diacetal and Its Application as Lubricating Base Oil

Dehydration acetalization of diglycerol with lauraldehyde in the presence of homogeneous acid catalysts using water as solvent has been studied by Li et al. The prepared double long-chain diglycerol diacetal was identified as a mixture of six regioisomers by GC and GC-MS. *p*-Dodecylbenzenesulfonic acid exhibited excellent activity for this acetalization reaction [J. Amer. Oil Chem. Soc. 94,1301–1311(2017)]. The reaction parameters with *p*-dodecylbenzenesulfonic acid catalyst were subsequently optimized in terms of the yield and isomer distribution of diglycerol diacetal. Under the optimum operating conditions, the yield of diglycerol diacetal reached 93.9%, and the selectivity in terms of the distribution of the six regioisomers was found to be in proportions of 24:36:18:11:3:8. Furthermore, the tribological performance of diglycerol diacetal has been evaluated as a potential lubricant base oil by means of an Optimol SRV-V oscillating reciprocating friction and wear tester. The tribological test results revealed that diglycerol diacetal displayed superior lubrication properties compared to the commonly used poly-alpha-olefin (PAO 8).

Adulteration and Presence of Polycyclic Aromatic Hydrocarbons in Extra Virgin Olive Oil Sold on the Brazilian Market

Seventy samples sold in the Brazilian market as extra virgin olive oil (EVOO) were evaluated by Tfouni et al. for the presence of the 13 polycyclic aromatic hydrocarbons (PAH) classified as carcinogenic and genotoxic by the Joint FAO/WHO Expert Committee on Food Additives (JECFA), to verify if the products were adulterated and to evaluate if there is a correlation between PAH presence and adulteration. PAH were detected in 93% of the samples, with summed levels varying from not detected to 41.10 μg/kg. Five samples showed BaP concentration above acceptable levels set by European legislation and by Brazilian regulation (2.0 μg/kg) and 7 presented PAH4 levels above the limit set by European legislation (10.0 μg/kg). The levels of fatty acid composition, sterols content, stigmastadiene and specific extinction did not comply with both Brazilian and International Olive Council (IOC) standards in 18, 31, 30 and 21% of the samples, respectively [J. Amer. Oil Chem. Soc. 94,1351–1359 (2017)]. The tolerance levels for these analyses in the Brazilian standards are 55.0–83.0 g/100 g (oleic acid), 3.5–21.0 g/100 g (linoleic acid), ≤0.05 g/100 g (*trans*-oleic acid), ≤ 0.05 g/100 g (*trans*-linoleic + *trans*-linolenic acid), ≤0.15 mg/kg (stigmastadiene), ≤2.50 (K232), ≤0.22 (K270), ≤0.01 (ΔK), 1000–1600 mg/kg (Σ sterols). Results indicate that 19 samples were adulterated. According to principal component analysis, samples were distinguished as: (1) EVOO with addition of vegetable oil from another source, (2) EVOO with addition of refined oil and (3) samples possibly not adulterated. The variable ΣPAH was related mainly to samples of EVOO with addition of vegetable oil from another source.

Geographical Differentiation of Greek Extra Virgin Olive Oil from Late-Harvested Koroneiki Cultivar Fruits

Seventy-four monovarietal olive oil samples belonging to the Koroneiki cultivar were collected by Kosma et al. from four selected olive oil-producing regions of Greece (Messinia, Lakonia, Irakleio and Etoloakarnania), during two harvesting periods (2012/2013 and 2013/2014) at the stage of full maturation (maturation index 5–6). Determination of volatile compounds (VC), fatty acid (FA) composition, total phenolic content (TPC) and color parameters was carried out in an effort to classify Koroneiki olive oil samples according to geographical origin, while conventional quality parameters (CQP) were used to characterize the samples respectively [J. Amer. Oil Chem. Soc. 94,1373–1384 (2017)]. The analytical data were then subjected to statistical analysis using multivariate analysis of variance (MANOVA) and linear discriminant analysis (LDA). The results showed a correct classification rate of 79.7% based on VC analysis, 81.1% based on the combination of VC analysis and FA composition, and 87.8% based on the combination of VC analysis and color parameters

Influence of Geographical Origins on the Physicochemical Properties of Hass Avocado Oil

A study was conducted to compare the physicochemical properties of Hass avocado oil from different geographical locations (Mexico, Australia, United States and New Zealand) by Tan et al. Regardless of geographical origins, Hass avocado pulp was characterized by high lipid content (61.27–62.66%). Among Hass avocados of different origins examined, avocado oil of New Zealand origin exhibited the lowest saponification value. The *L**, *a** and *b** values for avocados of New Zealand origin were higher than others, translating into the oil being the lightest in color and containing more red and yellow pigments [J. Amer. Oil Chem. Soc. 94,1431–1437 (2017)]. The predominant fatty acids in the Hass avocado oil were oleic (42.59–50.97%) and palmitic (20.61–25.63%) acids, whereas the predominant triacylglycerols (TAGs) were OOO (21.41–34.69%) and POO (19.65–24.68%), where O and P denote oleic and palmitic acids, respectively. The melting curves of Hass avocado oil displayed three endothermic peaks, whereas the crystallization curves displayed two endothermic peaks. Hass avocado oil of New Zealand origin contained a significant amount of natural pigments and unsaturated compounds (unsaturated fatty acids and tri-unsaturated TAGs) than Mexico, Australia and United States origins.

Sustainable Oxidative Cleavage of Vegetable Oils into Diacids by Organo-Modified Molybdenum Oxide Heterogeneous Catalysts

Exploiting vegetable oils to produce industrially valuable diacids via an eco-friendly process requires an efficient and recyclable catalyst. In this work reported by Ello et al., a novel catalytic system based on organo-modified molybdenum trioxide was synthesized by a green hydrothermal method in one simple step, using Mo powder as precursor, hydrogen peroxide, and amphiphilic surfactants cetyltrimethylammonium bromide (CTAB) and tetramethylammonium bromide (TMAB) as capping agents [J. Amer. Oil Chem. Soc. 94,1451–1461 (2017)]. The synthesized materials were first characterized by different techniques including XRD, SEM, TGA, and FT-IR. Interestingly, various morphologies were obtained depending on the nature of the surfactants and synthetic conditions. The synthesized catalysts were employed in oxidative cleavage of oleic acid, the most abundant unsaturated fatty acid, to produce azelaic and pelargonic acids with a benign oxidant, H_2O_2. Excellent catalytic activities resulting in full conversion of initial oleic acid were obtained, particularly for CTAB-capped molybdenum oxide (CTAB/Mo molar ratio of 1:3) that gave 83 and 68% yields of production of azelaic and pelargonic acids, respectively. These are the highest yields that have been obtained for this reaction by heterogeneous catalysts up to now. Moreover, the CTAB-capped catalyst could be conveniently separated from the reaction mixture by simple centrifugation and reused without significant loss of activity up to at least four cycles

Liquid Phase Selective Catalytic Oxidation of Oleic Acid to Azelaic Acid Using Air and Transition Metal Acetate Bromide Complex

Industrially important di-carboxylic acids are synthesized from mono-carboxylic unsaturated and unsaturated fatty acids. In this study by Hajra et al. aimed to perform the simultaneous catalytic oxidative C=C cleavage of oleic acid (OA) to azelaic acid and pelargonic acid, and oxidation of the terminal methyl group in pelargonic acid to azelaic acid using cobalt- and manganese-acetate as catalyst, hydrogen bromide as co-catalyst and air in acetic acid at elevated pressure (2.8–5.8 barg) and temperature (353–383 K). Oxygen solubility is determined under varying pressure, temperature and OA loading [J. Amer. Oil Chem. Soc. 94, 1463–1480 (2017)]. The effect of OA loading, pressure and temperature on OA conversion and azelaic acid selectivity is studied by varying one variable at a time; however, the presence of the synergistic effect of the catalyst and co-catalyst is investigated by central composite design assisted response surface methodology. Oxidation of terminal methyl group in saturated fatty acid is also confirmed by the oxidation of stearic acid to octadecanedioic acid using identical oxidation conditions of OA. Oxidation products of fatty acids are quantified by gas chromatographic analysis. The innovation of the work is thus the ability of the catalytic system to perform a total oxidation of a terminal methyl group of the hydrocarbon chain. OA oxidation kinetics relating to catalyst and co-catalyst concentration along with oxygen solubility at elevated temperature and pressure is established. The frequency factor and activation energy for OA oxidation is determined using the Arrhenius equation.

Transformation of Methyl Linoleate to its Conjugated Derivatives with Simple $Pd(OAc)_2$/Lewis Acid Catalyst

With the rapid depletion of fossil resources, the exploitation of biomass to partly replace fossil resources as the source of carbon in the chemical industry constitutes a promising alternative for the near future. This work reported by Senan et al. introduces catalytic transformation of vegetable oil, i.e., methyl linoleate, to its conjugated esters by a simple $Pd(OAc)_2/Sc(OTf)_3$ catalyst, which has extensive applications in industry [J. Amer. Oil Chem. Soc. 94,1481–1489 (2017)]. It was found that adding non-redox metal ions like Sc(III) to a simple $Pd(OAc)_2$ catalyst can effectively improve its isomerization activity in toluene/*t*-BuOH solvent, whereas $Pd(OAc)_2$ alone is inactive. Preliminary mechanistic investigations together with previous studies suggested that the in situ-generated heterobimetallic Pd(II)/Sc(III) dimer serves as the key species for methyl linoleate isomerization, and the reaction proceeds by [1,3]-hydrogen shift mechanism involving a formal Pd(II)/Pd(IV) cycle.

Optimization of an Aqueous Extraction Process for Pomegranate Seed Oil

Response surface methodology employing a five-level, four-variable central composite rotatable design was applied to study the effects of extraction time, extraction temperature, pH and water/solid ratio on the extraction yield of pomegranate seed oil using an aqueous extraction approach by Ghorbanzadeh et al. In addition, quality indices, fatty acid composition and antioxidant activity of the obtained oil were studied and compared with those of typical hexane-, cold press- and hot press-extracted oil [J. Amer. Oil Chem. Soc. 94,1491–1501 (2017)]. Aqueous extraction resulted in the maximum oil recovery of 19.3% (w/w), obtained under the following critical values: water/solid ratio (2.2:1.0, mL/g), pH 5.0, extraction temperature = 63 °C and extraction time = 375 min. This yield is lower than that obtained via hexane extraction (26.8%, w/w) and higher than the yields from cold press (7.0%, w/w) and hot press (8.6%, w/w) extraction. A comparison of the characteristics of the oils based on extraction method revealed that the unsaturated fatty acid content was highest for the oil obtained by aqueous extraction. In addition, higher levels of iodine and peroxide and lower levels of acid, *p*-anisidine and unsaponifiable matter were observed. The oil obtained with aqueous extraction also exhibited higher antioxidant activity than oils obtained by hexane or hot press extraction.

Combined Short-Path Distillation and Solvent-Assisted Crystallization of Beef Fatty Acid Methyl Esters

Solvent-assisted crystallization has previously been employed to remove long-chain saturated fatty acids (≥ 18 carbons) from animal fat to improve its cold temperature biofuel properties. The same technology can be used for removing long-chain saturated fatty acids (SFA) from animal fats for human consumption, but SFA remaining (i.e., 14:0 and 16:0) are more atherogenic than longer chain SFA. In the present study, an easy and efficient method was developed by Dugan et al. using short-path distillation prior to solvent-assisted crystallization for the more complete removal of SFA from beef tallow, and for the first time reports the distillation and crystallization behaviour of polyunsaturated fatty acid biohydrogenation products (PUFA-BHP). Shorter chain SFA methyl esters (i.e., 14:0 and 16:0) were efficiently removed at 90 °C, 9.3 Pa, with a rotor speed of 70 rpm and either two cycles of distillation at 90 drops/min or three cycles at 110 drops/min. Stearic acid (18:0) was then effectively removed by crystallization at −20 °C using a sample to methanol ratio of 1:10 [J. Amer. Oil Chem. Soc. 94, 1503–1508 (2017)]. The remaining fraction enriched with PUFA-BHP (i.e., rumenic acid, *c*9,*t*11-18:2, and its precursor vaccenic acid, *t*11-18:1) have potential use in disease model (i.e., cell culture and animal) studies to help further elucidate their bioactivity and mode of action, and may in the future have functional food or nutraceutical potential.

Synthesis of Biolubricants by the Esterification of Free Ftty Acids from Castor Oil with Branched Alcohols using Cationic Exchange Resins as Catalysts Saboya et. al. have evaluated three cationic exchange resins (PD206, CT269DR and CT275DR), supplied by Purolite®, with different physicochemical properties in the esterification reaction of the free fatty acid from the castor oil with 2-ethylhexanol in order to obtain products with interesting properties to be used as biolubricant basestock [Ind. Crops & Products 104, 52-61 (2017)]. These resins have been characterized by SEM, elemental analysis (CNHS), TG/DTA and XPS. Likewise, these catalysts have been analyzed after the catalytic reaction to evaluate changes in the active phase during the esterification reaction. The catalytic data reveal that the macroreticular-type resins (CT269DR and CT275DR) show higher conversion values, about 95% after 4 h, than a gel-type resin (PD206) due to several factors, such as the higher amount of available acid sites or the denser divinylbenze matrix, which provides higher thermal and mechanical resistance and minimizes sulfur leaching. The deactivation of the active phase has been ascribed to the strong adsorption of reagents and products on the sulfonic groups located at the surface, as indicated in the XPS spectra of the spent catalysts. The products characterization showed that 2-ethylhexyl ricinoleate exhibited excellent properties at low temperature (pour point −39 °C), viscosity index value compatible with commercial synthetic basestock oils, higher biodegradability than mineral oil and better oxidation stability than the original feedstock.

Rice Bran Oil Extraction using Alcoholic Solvents: Physicochemical Characterization of Oil and Protein Fraction Functionality

Rice bran, an underutilized rice processing by-product, is a promising source for food and biodiesel oil production and can also be used to produce protein for use in human food products. The main objective of this study by Capellini et al. was to assess the feasibility of replacing hexane, which is traditionally used to extract vegetable oils, with safer solvents, i.e., ethanol and isopropanol, in rice bran oil (RBO) extraction [Ind. Crops & Products 104, 133-143 (2017)]. Thus, the effects of the solvent type on the physicochemical characteristics of the oil and defatted bran products were studied. The results showed that the presence of water in the alcoholic solvents negatively affected the oil extraction; however, using absolute solvents in single-stage batch extractions at 80 °C resulted in oil yields of up to approximately 80%. The solvent water content and process temperature strongly impacted the properties of the protein

fraction; the nitrogen solubility index (NSI) decreased from approximately 40% for the absolute solvents to 17 and 15% for the aqueous ethanol and isopropanol, respectively, when the extraction was performed at 80 °C. More of the minor nutraceutical compounds were transferred from the oleaginous matrix to the oil by aqueous ethanol than by hexane, yielding RBO with 1.53% γ-oryzanol and 769 mg/kg tocotrienols. On the other hand, absolute isopropanol exhibited a higher tocopherol extraction capacity; RBO with a tocopherol content of 98.1 mg/kg was obtained with this solvent. Based on these results, short-chain alcohols are promising alternatives to the conventional extraction solvent, because they enable high-quality protein fractions and oils to be obtained and add value to the rice production chain.

Acylation of Epoxidized Soybean Biodiesel Catalyzed by SnO/Al_2O_3 and Evaluation of Physical Chemical and Biological Activity of the Product

Biodiesel has emerged as an alternative to partially replace diesel. However, the presence of polyunsaturated fatty acid chains, such as when using soybean oil as starting materials, is associated with several issues due to its low oxidative stability. Other issues, such as the formation of biofilms in fuel storage tanks and unsatisfactory cold properties are also major concerns during the use of biodiesel. Hence, this work by Oliveira et al. is aimed at modification of the chemical structure of soybean biodiesel in two steps: (i) epoxidation of soybean biodiesel double bonds and (ii) acetylation of the soybean biodiesel epoxide with acetic anhydride, using a heterogeneous catalytic system [Ind. Crops & Products 104, 201-209 (2017)]. The catalyst used for acetylation step was tin oxide supported on alumina. The catalytic system activity was evaluated using different temperature, catalyst/substrate ratio and different anhydrides. Oxidative stability, thermal analysis and anti-microbial activity were evaluated. The products structures were confirmed by infrared and nuclear magnetic resonance. The results show yields up to 98% of the desired product − di-acetyl biodiesel in 10 h and 15% catalyst (w/w). The catalyst could be recycled up to four times with minor activity loss. After acetylation, it was observed an increase in both oxidative stability and biostatic activity. However, the freezing point did not change.

Derivatization of Castor Oil based Estolide Esters: Preparation of Epoxides and Cyclic Carbonates

Estolides that are based on castor oil and oleic acid are versatile starting points for the production of industrial fluids with new properties. Doll et al. derivatized a variety of unsaturated estolides by epoxidation with hydrogen peroxide. The epoxidized estolides were further modified using supercritical carbon dioxide and tetrabutylammonium bromide to chemically incorporate carbon dioxide into the material yielding a 5-membered cyclic carbonate structure [Ind. Crops & Products 104, 269-277 (2017)]. These new epoxides and cyclic carbonates exhibited higher pour points, oxidation onset temperatures, and viscosities, compared to the corresponding unsaturated precursors. One derivative had a dynamic viscosity of ~9000 mPas at 40 °C, demonstrating potential for use in industrial applications.

Plasticization Effect of Epoxidized Cottonseed Oil (ECSO) onPoly(lactic acid)

In this work by Carbonell-Verdu et al. the use of an environmentally friendly plasticizer derived epoxidized cottonseed oil (ECSO) for poly(lactic acid) (PLA) is attempted. Melt extrusion was used to plasticize PLA formulations with different ECSO contents in the 0–10 wt.%. PLA formulation with 10 wt.% showed a remarkable increase in mechanical ductile properties with a percentage increase in elongation at break of more than 1100% and a noticeable increase in the impact absorbed energy [Ind. Crops & Products 104, 278-286 (2017)]. Differential scanning calorimetry (DSC) and dynamic mechanical thermal analysis (DMTA) revealed a clear decrease in the glass transition temperature of neat PLA as the ECSO content increased. Field emission scanning electron

microscopy (FESEM) of fractured surfaces from impact tests showed an improvement of ductility with typical rough and porous topographies. Migration tests in *n*-hexane at different temperatures revealed very low migration properties thus leading to new interesting plasticizers for improved PLA industrial formulations.

Lipid Characterization of *Eryngium maritimum* Seeds grown in Tunisia

Fatty acid, triacylglycerol, phospholipid, tocol and sterol compositions of *E. maritimum* seeds collected from plants grown in ten different Tunisian littoral locations were determined by BenLajnef et al [Ind. Crops & Products 105, 47-52 (2017)]. Constitutes from different locations are significantly different. Seeds presented high oil content, ranging from ca. 22% to 34% on a dry weight basis, and were characterized by a constant fatty acid profile among the growing locations. Gas chromatographic analysis revealed an important level of unsaturated fatty acids (ca. 90% of total fatty acid content), with a prevalence of oleic (ca. 63%) and linoleic (ca. 25%) acids. Results obtained by GC-FID showed that the carbon number (CN) values of the TAGs in all samples were 50 (1.5%), 52 (25.6%) and 54 (73%). Phosphatidylcholine was the most common phospholipid, accounting for more than 60% of the total content, followed by phosphatidic acid (about 30%). Different tocols concentration was reported in seeds with β-tocotrienol as the most abundant one in all samples. Stigmasterol was the most abundant sterol in all seeds, representing nearly 70% of the total content, followed by β-sitosterol and Δ^7-sitosterol. In all, the findings of this work highlight the valuable levels of minor constituents from *Eryngium maritimum* storage lipids which can be therefore a prospective source of useful natural bioactive molecules that may replace synthetic antioxidants.

Application of *Calophyllum inophyllum oil* as Antifungal Fat-liquor for Leather Industry

Calophyllum inophyllum oil which contains mixed oleic, linoleic acid, stearic acid and palmitic acid is well known for its antimicrobial properties. Presence of unsaturation leads to provide better opportunity for its modification for fat liquoring. Transesterified emulsion of *Calophyllum inophyllum oil* shows good antifungal property. To enhance its antifungal activity against more fungal species the blending of trans esterified emulsion with water extract of *Bishop's weed* leads to the formation of an antifungal fat liquor. This blended product mitigates most of the disadvantages associated with conventional tanning. Sahu et al. investigated the possibility of using *Calophyllum inophyllum oil* as antifungal fat liquor for leather industry [Ind. Crops & Products 105, 104-112 (2017)]. They collected fresh mature cultures of fungal species such as, *Aspergillus Niger, Penicillium notatum, Aspergillus flavus*and *Paecilomyces variotii* and used as sources of inoculum for the antifungal activity of the fat liquor . Gas chromatograph with a mass detector (GC/MS) analysis of both *Calophyllum inophyllum oil* and *Bishop's weed* were carried out separately. The presence of unsaturated acids like oleic acid and linoleic acid provide the platform for its modification by transesterification process and the presence of two compounds namely *thymol* and *carvacrol* in the *Bishop's* weed enhances the antifungal activity of fat liquor. The antifungal activity of fat liquor is confirmed by Agar well diffusion method.

Camelina (*Camelina Sativa*) Oil Polyols as an Alternative to Castor

Castor oil is a natural polyol and widely used in many applications. Although castor oil is a renewable biobased product, its production is geographically limited to tropical regions, resulting in fluctuations in supply and price stability. Omonov et al. This work explored the possibility of the production of castor oil alternatives from camelina oil grown in temperate regions as an industrial crop and its subsequent use in polyurethane formulations in a detailed study. Castor oil replacement polyols were produced from camelina oil via controlled epoxidation using *in-situ* generated performic acid and a subsequent

hydroxylation via acid catalyzed epoxy ring opening with alcohol [Ind. Crops & Products 107, 378-385(2017)]. The viscosity and hydroxyl functionality of camelina oil polyol was targeted to match castor oil since these parameters dictate the performance of these polyols as drop-in replacements. The reaction of camelina oil polyols and castor oil with polymeric diphenylmethane diisocyanate to produce polyurethane was studied via their rheological behavior during curing, which indicated their relative reactivities. In polyurethane applications, it was found that whilst camelina oil polyols showed lower reactivity towards isocyanates, reactivity can be readily adjusted to match castor oil by the addition of small amount of common catalysts. The thermo-mechanical properties of cured polyurethanes from camelina oil polyols (expressed via glass transition temperature) were evaluated using differential scanning calorimetry and found to significantly exceed those made using castor oil. Overall, this study demonstrates that polyols from camelina oil can be produced with a range of viscosities and hydroxyl functionalities and are suitable to replace castor oil in polyurethane applications. The ability to adjust these parameters offers a significant advantage over castor oil.

Synthesis and Thermal Study of Polymers from Soybean, Sunflower, and Grape seed Maleinated Oil

Alarcon et al. synthesized some polymers from maleinated vegetable oils (soybean, sunflower, and grape seed) and glycerol. First, all the vegetable oils were previously maleinated with maleic anhydride, then, the maleinated oils were esterified with glycerol to obtain the polymers; the synthetic route is simple without the use of organic solvents, the results indicate that the synthesis was effortless using inexpensive reagents, such as vegetable oils, maleic anhydride, and glycerol [Eur. J. Lipid Sci. Technol.119, 10.1002/ejlt.201600515 (2017)]. These polymers could be applied in the area of adhesives and waterproofing materials. The results obtained by TGA-DTA and DSC data showed that these polymers had different thermal behaviors, which is related to the different quantity of unsaturated fatty acids in each oil structure. Finally, the FTIR and 1H-NMR analysis help to clarify the polymers structure and the reaction sites: hence, it was possible to propose a reaction mechanism and the structure.

Algerian *Moringa oleifera* whole Seeds and Kernels Oils: Characterization, Oxidative Stability, and Antioxidant Capacity

The rising of global per capita food consumption has driven scientists and manufacturers into a search for new or renewed edible oil sources, while also improving their production towards maximum yield, quality, and economic feasibility. Hence, a new origin (Algerian) of *Moringa oleifera* seed oil was characterized in the present study by Boukandoul et al. in terms of its physical and chemical features on two consecutive crops. Moreover, aiming to ascertain if the industrial solvent extraction could be enhanced, extracted oils from shelled and unshelled seeds were compared [Eur. J. Lipid Sci. Technol.119, 10.1002/ejlt.201600410 (2017)]. All oils under study presented a low oxidation status and an excellent resistance to oxidation. Additionally to the characteristic high average content of unsaturated fatty acids (71% oleic acid), microcomponent analyses revealed interesting amounts of phytosterols (4–5 g/kg), tocopherols (287–327 mg/kg), carotenoids (4–8 mg/kg), and phenolic compounds (11–17 mg/kg). These outcomes were within or better (stability and oxidative status) than reported data for *M. Oleifer a*seed oil from other origins, and without significant differences between crops. Apart from a slight color change and increased sterol and wax contents when extracted with shells, no other significant compositional changes were observed, including oil extraction yield, both equivalent to 37% on a kernel basis. This work sustains the high quality of Algerian *M. oleifera* seed oil, a still underutilized crop in this country. Also, it provides an insight on the potential use of grinded whole seeds directly for solvent extraction, reducing labor demand and cost for the industries, without significant variation on

compositional and stability parameters.

Pomegranate Seed Oil Organogels structured by Propolis Wax, Beeswax, and their Mixture

The capability of propolis wax, beeswax, and their mixture to gel pomegranate seed oil (PSO) was studied by Fayaz et al. Samples were prepared increasing the concentration of waxes in pomegranate seed oil from 5 to 15% w/w. After propolis wax, beeswax, and PSO chemical characterization, thermal, and structural properties of wax-based organogels were studied [Eur. J. Lipid Sci. Technol.119, 10.1002/ejlt.201700032 (2017)]. Both propolis wax and beeswax formed organogel due to the presence of crystal networks made of needle-like β′-form crystals. Based on FTIR spectroscopy, mainly vander waals interactions were formed in the systems. Propolis wax organogels displayed lower firmness and G′ and G″ modulus in comparison to the corresponding beeswax samples. This was due to the presence of larger crystals with a less organized network. The observed behaviors were attributed to differences in chemical composition between these two types of waxes. The combined use of propolis wax and beeswax led to organogels with behavior in between the organogels with single waxes, probably as a consequence of the formation of mixed crystals.

Synthesis and Evaluation of Cytotoxic Activity of Conjugated Linoleic Acid Derivatives (esters, alcohols, and their acetates) toward Cancer Cell Lines

Conjugated linoleic acid (CLA) is natural unsaturated fatty acid known for the wide biological properties therefore is available in functional food and cosmetics and as a dietary supplements. In this study by Niezgoda et al., a series of simple derivatives of *cis*-9,*trans*-11 and *trans*-10,*cis*-12 CLA and a mixture of those isomers have been evaluated as alternative for free CLA preparations [Eur. J. Lipid Sci. Technol.119, 10.1002/ejlt.201600470 (2017)]. Obtained in high yields (>72%) ethyl esters, alcohols, and their acetates have been subjected to in vitro cytotoxic assay against human tumor cells (human promyelocytic leukemia, breast cancer, and colon cancer) and compared with stearic and linoleic derivatives. Studies showed that the growth inhibitory activities depended on the degree of unsaturation. All CLA derivatives except ethyl esters showed similar activities compared to free CLA isomers. Moreover, this study showed that *trans*-10,*cis*-12 CLA derivatives have better anticancer properties against all tested tumor cells than derivatives of *cis*-9,*trans*-11 isomer and a mixture of both isomers.

Synthesis and Characterization of a New Biobased Poly(urethane-ester) from Ricinoleic Acid and its use as Biopolymeric Matrix for Magnetic Nanocomposites

This work reported by Peres et al. is aimed at preparation of a new biobased poly(urethane-ester) synthesized from ricinoleic acid (RA), 1,6-diisocyanatehexane, and glycerol through bulk polymerization. RA-modified magnetic iron oxide nanoparticles, previously obtained through coprecipitation technique, were introduced in situ to produce a magnetic nanocomposite. DSC analyses revealed that the glass transition (Tg) of the biobased poly(urethane-ester) was equal to −75°C, and for the magnetic biobased poly(urethane-ester) the Tg was reduced in approximately 3°C [Eur. J. Lipid Sci. Technol.119, 10.1002/ejlt.201600451 (2017)]. DTG analyses were used to measure the thermal stability and the amount of magnetic nanoparticle dispersed into the polymeric matrix, indicating that the polymeric materials exhibit good thermal stability and the fraction of nanoparticles dispersed into the biobased poly(urethane-ester) matrix was equal to 2.19 wt%. Infrared spectroscopy and NMR were used to recognize functional groups in structures and follow the reaction evolution. Magnetic force, hysteresis loops and XRD essays used to provide magnetic features of the materials, showed that crystallite size of the mono domains was equal to 7.6 nm and that magnetic materials exhibit superparamagnetic behavior. GPC was used to evaluate average molar mass of polymer, determined equal to 2569 Da and molar mass

dispersity equal to 2.4.The polymer is characterized by FTIR, ^{1}H NMR, TG, and DSC. The composite is studied by magnetic force exhibiting superparamagnetic behavior.

Characterization of Pomegranate (*Punica granatum* L.) Seed and Oils

Juhaimi et al. characterized pomegranate (*Punica granatum* L.) seed and oils with a view to study their potential as a source of phytochemical, and bioactive compounds [Eur. J. Lipid Sci. Technol.119, 10.1002/ejlt.201700074 (2017)]. While total phenol contents of seed extracts vary between 23.6 mg gallic acid equivalent/g and 28.8 mg gallic acid equivalent/g, their antioxidant activities were changed between 17.6 and 22.9% ($p < 0.05$). The seeds of pomegranate varieties contained 347–647 mg/kg Ca, 2076–3846 mg/kg K, 1572–2327 mg/kg P, and 1357–1781 mg/kg Mg. While the total phenol contents of seed oils change between 7.8 mg gallic acid equivalent/g and 19.2mg GAE/g, antioxidant activity of oil samples varied between 6.8 and 8.8% ($p < 0.05$). The predominant fatty acid of oils is punicic acid, and punicic acid contents of oil samples were found between 71.2 and 77.6%. Oleic acid contents of seed oils were determined between 7.6 and 9.1%. In addition, **γ**-tocopherol contents of pomegranate oil ranged from 236 mg/100 g to 389 mg/100 g. Pomegranate seeds are sources of bioactive and phytochemical such as fatty acid, **γ**-tocopherol, total phenolic content, and flavonoids with potentially high antioxidant activities.

A Simple Enzymatic Process to Produce Functional Lipids From Vegetable and Fish Oil Mixtures

Functional lipids (e.g., those triacylglyerols containing EPA and DHA, besides others) are related to the prevention and treatment of many cardiovascular diseases. Furthermore, fatty acids located at the central bond (*sn*-2) of triacylglycerols are more efficiently absorbed via lymphatic route. Marin-Suarez et al. with an aim to increase the Omega-3 content at the *sn*-2 position of vegetable and fish oils (high oleic sunflower and sardine oil), conducted random and 1,3-specific lipase enzymatic interesterification [Eur. J. Lipid Sci. Technol.119, 10.1002/ejlt.201700233 (2017)]. Direct interesterification and a two-step treatment (in situ hydrolysis and re-esterification) were evaluated using Lipozyme TL IM and Novozyme 435. For both lipases, direct esterification led to a minor production of by-products, that is, free fatty acids and diacylglycerols (<10 mol%), and to an increase of EPA and oleic acid at the *sn*-2 position, regardless of the oil mixture composition. The best result was obtained using pure sardine oil and Lipozyme TL IM, (at *sn*-2 position, the content of EPA and oleic acid were doubled, while DHA presented an increase of 20%). This is an easy process by which the composition of the oils used in food fortification can be improved.

Convenient and Environmentally Friendly Production of Isostearic Acid with Protonic Forms of Ammonium Cationic Zeolites

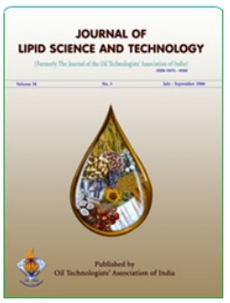
Three ammonium-cationic zeolites (ferrierite, ZSM5, and zeolite BETA) were individually analyzed by Sarker et al. to produce branched-chain fatty acid (i.e., isostearic acid) from unsaturated linear-chain fatty acid (ulc-FA) with up to 98% conversion and 80% selectivity [Eur. J. Lipid Sci. Technol.119, 10.1002/ejlt.201700262 (2017)]. The SiO_2/Al_2O_3 molar ratio of zeolite, amount of water and choice of additive are found to be the key factors for the efficiency of zeolites in this particular reaction. Detailed characterization of zeolites supports the optimization parameters to produce the best results. Large scale production of isostearic acid achieving a 76% selectivity and 96% conversion demonstrates the potential of this system's capability at the pilot scale. Variation in isomeric composition of branched-chain fatty acid (bc-FA) product is observed for the three different catalytic methods. Dimer production due to interaction with the external acidic surface of these zeolites has been observed, but it can be suppressed.

Ultrasound Assisted Synthesis of Hydroxylated Soybean Lecithin from Crude Soybean Lecithin as an Emulsifier

Soybean lecithin is a by-product obtained during degumming step of crude soybean oil refining. Crude soybean lecithin (CSL) contains major amount of phospholipids (PLs) along with minor amount of acylglycerols, bioactive components, etc. Due to the presence of PLs, CSL can be used as an emulsifier. Chiplunkar and Pratap used Crude soybean lecithin (CSL) to synthesize hydroxylated soybean lecithin (HSL) by hydroxylation using hydrogen peroxide and catalytic amount of lactic acid to enhance the hydrophilicity and emulsifying properties of CSL [J. Oleo Science 66, 1101-1108(2017)]. To reduce the reaction time and to increase rate of reaction, HSL was synthesized under ultrasound irradiation. The effect of different operating parameters such as lactic acid, hydrogen peroxide, temperature, ultrasonic power and duty cycle in synthesis of HSL were studied and optimized. The surface tension (SFT), interfacial tension (IFT) and the critical micelle concentration (CMC) of the HSL (26.11 mN/m, 2.67 mN/m, 112 mg/L) were compared to CSL (37.53 mN/m, 6.22 mN/m, 291 mg/L) respectively. The HSL has better emulsion stability and low foaming characteristics as compared to CSL. Therefore, the product as an effective emulsifier can be used in food, pharmacy, lubricant, cosmetics, etc.

Partial Hydrogenation of Sunflower Oil-derived FAMEs Catalyzed by the Efficient and Recyclable Palladium Nanoparticles in Polyethylene Glycol

One approach to improve the oxidative stability of biodiesel is the partial hydrogenation of carbon-carbon double bonds. In the current work, an efficient catalytic system using $Pd(OAc)_2$ dissolved in polyethylene glycol (PEG) which *in situ* generates palladium nanoparticles was developed by Lu and Li in order to promote a selective partial hydrogenation reaction of sunflower oil FAMEs into *mono*-hydrogenated products avoiding the formation of saturated compounds or *trans*-isomers [J.Oleo Science 66, 1161-1168 (2017)]. High content of methyl oleate (85.0±1.4%) was obtained by hydrogenation of sunflower oil biodiesel with only 7.0±0.2% stearic acid. Through evaluating the palladium nanoparticles by TEM analysis, it is observed that 4 nm palladium nanoparticles generated *in situ* in PEG4000 are highly selective for the partial hydrogenation of sunflower oil biodiesel. And the Pd-PEG4000 catalyst can be resued for five times without obvious loss of activity or methyl oleate selectivity.



JOURNAL OF LIPID SCIENCE AND TECHNOLOGY

Disseminating Innovative Technologies

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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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