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## *Editor's desk.....*



Dear Readers,

Advances in nanotechnology have revolutionized many aspects of medical treatments and electronics, and huge potential exists for similar benefits to be realized in the oils and fats sector as well. Several industry technology developers, in cooperation with academia, are investigating the potential applications of nanotechnology and examining the scope for crossover technologies that have been developed for other industries. The range of applications is enormous and has the potential of developments in emulsions, cosmetics and vegetable oil processing fields. Nanotechnology has enormous potential in the oil industry, with numerous applications currently under investigation. These include, for example, improvements in material design for enhanced resistance to corrosion or erosion, improved and enhanced oil recovery, improved understanding of reservoirs through use of nanosensors.

Studies have concluded the high efficiency of nanotechnology in bleaching of vegetable oils. The mechanical alloying process succeeded to obtain nanosized bleaching earth powders after 10 h of milling and the morphology appeared rod with 46.6 nm in length and 4.46 nm in diameter. The milled nano-sized bleaching powder was used in bleaching of oils under study. Comparing color indices, and peroxide values for the resulted bleached oils with the unbleached and control ones bleached with raw bleaching earth. It was concluded that using nano-particles bleaching earth had 30% high bleaching efficiency and peroxide values for oils bleached with raw bleaching earth were reduced to 30 % more for oils bleached with nano bleaching earth.

Nanotechnology plays a major role in the vegetable oil sector through the quality oil production ends with advanced processing, packaging, and long-term storage, provided enormous growth in industry through enhancement in quality. The nanomaterials and nanosensors help the consumers providing information on the state of the oil/cosmetic/derivative inside and its nutritional status with enhanced security through pathogen detection. Most of the bioactives against various diseases are hydrophobic in nature having least bioavailability and stability; thus, the nanotechnology-based delivery systems provided an enhanced bioavailability and targeted delivery of bioactive compounds. Active utilization of nanocolloidal particles in different branches of specialty chemical industry, such as quality, safety, nutrition, processing, and packaging, has been widely reported recently.

Nanoparticles are manufactured all over the world, though very few countries possess the standard regulatory rules for the utilization of nanotechnology in vegetable oil-food products. The applications of nanoparticles in food packaging are less harmful than the utilization of nanoparticles as a process additive, food ingredient. So far, very few in vivo studies have been conducted on the effects of nanofoods in human and animal health. There should be appropriate labeling and regulations advised for marketing of nano particle based products which can help to increase consumer acceptability. Thus, utilization of these nanotechnologies, if managed and regulated correctly, can play a significant role in improving processing and product quality which will benefit human health and well-being.

A handwritten signature in black ink, appearing to be 'R. Trivedi', written in a cursive style.

**Prof. Rakesh Kumar Trivedi**

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# आपका विश्वास भारत का No.1



## पहले इस्तेमाल करें फिर विश्वास करें

\* As per Nielsen Retail Index data for MAT March 2021.  
All India (Urban +Rural) market in Washing Powder Category.



# DEVELOPMENT AND NUTRITIONAL EVALUATION OF VALUE-ADDED READY-MIX POWDER INCORPORATED WITH FLAXSEEDS AND SPICES

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## ABSTRACT:

The aim of the present study was to process oats, pulses and flaxseeds by using them to create Value-Added Ready-Mix Powder. The method for making ready-mix powder was standardized, and the finalized product's mixture of different qualities were evaluated. In the field of functional foods, flaxseed is quickly becoming one of the most important sources of nutrients and phytochemicals because of its high concentrations of  $\alpha$ -linolenic acid (ALA), lignans, and fiber. It is a prime source of alpha linolenic acid. The seed offers digestible high-quality protein, soluble fiber, lignans, and oil with a high omega-3 content and, one of the richest sources of  $\alpha$ -linolenic acid oil. It also has significant promise as a source of phenolic compounds. It seems that lignans are anti-carcinogenic substances. Focus is being placed on the health advantages of flaxseed's omega-3 and lignan phytoestrogens, which may have chemo-protective qualities in both humans and animals. A grain with a high concentration of soluble fiber and potent minerals, oats are known to be both healthy and nutritious. Pigeon peas is one of the most significant perennial food legume crops in the semi-arid tropics, provide a significant source of protein, dietary fiber, vitamins, and minerals.

Spices like, Cinnamon's powder structure contains beneficial phytochemicals as well as a per

centage of water - soluble bioactive polyphenols. Fenugreek seeds and/or seed extracts increased glucose uptake, decreased levels of glycosylated hemoglobin, proinflammatory cytokines, and pancreatic enzymes. The dried clove bud is made up of minerals, cellulose, essential oil helps to treat numerous pains, such as those in the joints, muscles, or sinewy tissue, especially rheumatoid arthritis. In addition to having a lot of vitamin C, chili also has a lot of vitamin A, vitamin B6, vitamin K, and other minerals. Due to its low levels of salt, cholesterol, and saturated fat, it is an excellent nutritious alternative to meat. In the study three types of mixture were using oats, green gram, pigeon peas and flaxseeds in all the three mixtures but with different types of spice combinations and processed for sensory evaluation immediately after development. It was concluded that the mixtures contained substantial amount of carbohydrates, proteins, fats, dietary fiber, and energy contents. They were also good antioxidants and contained high amounts of polyphenols in them. Easy preparative methods using the ready mixes were organoleptically well accepted among the panel members who consumed the ready mixes preparations.

**Keyword:** Ready-mix powder, value added, flaxseeds, oats, pulses, cinnamon, fenugreek, clove, chili, chemical compositions, phenolic compounds, vitamins and health benefits.

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## 1. INTRODUCTION

Food-based strategies seek to improve nutrition by increasing access to and consumption of a diet high in micronutrients and appropriate in macronutrients made up of a variety of readily available foods. People's lives have altered significantly in this period of industrialization and technological advancement. The demand for various sorts of convenience food has significantly expanded as living in the modern "cash-rich, time-poor" culture gets busier.<sup>1</sup> Since the dawn of time, convenience meals have been essential to human existence since they eliminate several processes typically needed in food preparation and cooking. Unused crops that can be combined or supplemented to give complete nutrition can be used to create affordable, nutrient-dense ready-to-cook food products.<sup>2</sup> Foods with added value significantly relate together diet, health, and happiness. Around the world, the term "functional food" is used in many different contexts, although none of these definitions is recognized or officially recognized. The current goal was to create value-added Ready-Mix Powder from oats, pulses, and flaxseeds. The method of making ready-mix powder was standardized, and the final product's mixture of several qualities was evaluated. These grains and flaxseed can be added using a variety of methods, including extrusion, flaking, popping, and combining with other grains.<sup>3</sup>

Flaxseed is rapidly rising to prominence as one of the most significant sources of minerals and phytochemicals in the realm of functional foods due to its distinctive nutrient profile. The key bioactive components of flaxseed, are lignans, Omega-3 fatty acids, and dietary fiber, which can be utilized through the preparation of value-added products. It has a lipid content of around 40%, of which 47–57% is alpha-linolenic acid (ALA), as well as other advantageous components such dietary fiber (28%), lignan, and secoisolariciresinol diglucoside (SDG).<sup>4</sup> Flaxseeds are similar to soy proteins in terms of their nutritional value and amino acid composition. While lysine is scarce and arginine, aspartic acid, and glutamic acid

are abundant in flaxseed protein, respectively. A high cysteine and methionine content raises antioxidant levels and lowers the risk of cancer. It is an excellent source of minerals, particularly phosphorus (650 mg/100 g), magnesium (350–431 mg/100 g), calcium (235–250 mg/100 g), and salt (27 mg/100 g). It has the most potassium (5600–9200 mg/kg) of any meal, and a high consumption of potassium is inversely correlated with blood platelet aggregation, blood free radicals, and the risk of stroke. Small amounts of both fat-soluble and water-soluble vitamins are present in flaxseed. The quantity of vitamin E in each 100 grams is 39.5 mg of  $\gamma$ -tocopherol. Antioxidant  $\gamma$ -tocopherol safeguards cellular proteins and lipids from oxidation. It also encourages sodium excretion in urine, which may reduce blood pressure, heart disease risk, and Alzheimer's disease risk. Dietary flaxseed consumption aids individuals in avoiding serious ailments such as cancer, diabetes, obesity, renal, bone, and gastrointestinal disorders. Additionally, it enhances platelet aggregation and immunological function.<sup>5</sup>

In addition to being a significant source of protein and calories, pulses have a number of nutritional advantages. 60 to 65 percent of the carbohydrates in pulses come from starch, which is their main source. Because the starch in pulses digests slowly, they have a low glycemic index (GI). With both soluble and insoluble fibers, pulses are extraordinary source of fiber.<sup>6</sup> The red gram, often referred to as pigeon pea, is a grain legume crop produced in semi-arid and subtropical regions of the world and is the third-ranked crop in Asia. Pigeon pea is said to contain 21–26% protein, 1.2–% fat, 65–% carbs, and 3.8–% ash, according to various estimations. Because of the significant amount of dietary fiber they contain, in addition to energy, protein, minerals and vitamins, which may benefit a variety of health conditions, pigeon peas might be considered a functional food.<sup>7</sup>

*Avena sativa* L., or oats, are well known for their high levels of dietary fiber, phytochemicals, and nutritional value. Oat eating is thought to provide a



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number of health advantages, including anti-inflammatory and hypo-cholesterolemic effects. It is a great source of complex carbohydrates, protein (11–15%) with a well-balanced amino acid profile, lipids (5–9%), including significant unsaturated fatty acids, minerals, vitamins, and phytochemicals. Additionally, a huge proportion of phenolic compounds, antioxidants, and the functional ingredient glucan (2.3–8.5%) are found. Consumption of oats or oat fiber has been demonstrated to lower postprandial glucose and insulin concentrations, and the decrease in insulin concentration may offer a method through which blood pressure may decrease in response to oat consumption. Oat intake has grown due to the health advantages of dietary fibers such beta-glucan, functional proteins, lipid and carbohydrate components, and phytochemicals found in oat fiber. Tocols, sometimes referred to as Vitamin E, is another advantageous substance present in oats. These natural anti-oxidants found in grains may be advantageous to both human and animal health. Both tocotrienols and tocopherols are present. They contribute to decreasing cholesterol levels by scavenging free radicals. Oat grain contains trace components such flavonoids, saponins, lignans, and sterols, albeit in very small amounts. These substances have antioxidant characteristics and are bioactive.<sup>8</sup>

Ready to mix powders are appealing and can be high in demand because it helps to conserve highly valuable resources including time and energy, and also require no skill in preparation. In such convenience meals, nutrition is the most significant consideration. A variety of Ready-Mix Powders were created, each with a control, using varying proportions of various kinds of spices. Utilizing black pepper powder, cumin powder, fenugreek powder, cinnamon powder, fennel seed powder, powdered cloves, chili flakes, asafoetida, salt, and powdered sugar, the composition of ready-mix-powders was standardized. Due to high dietary fiber content and other health advantages, it has the

potential to contribute as nutrient-dense foods. Spices add an essence, and flavour to food and an extra antioxidant to body. These are also being used as a remedy for health in various diseases, including fenugreek, coriander, turmeric, cinnamon, cumin, clove, and others. Generally, spices as part of the diets, have holistic effects on human health. Since ancient times, all spices have been incorporated in traditional ways and in everyday cuisine.<sup>9</sup>

Among all the spices, cinnamon is one which is most frequently used. In addition to having a pleasant taste, cinnamon is rich in antioxidants that guard against cell deterioration and even decrease cholesterol levels. Additionally, experts think that it lowers the risk of heart disease and helps maintain healthy levels of HDL in the body.<sup>10</sup> Cinnamon's powder structure contains beneficial phytochemicals as well as a percentage of water-soluble bioactive polyphenols that can be successfully used in people who consume a network of soy flour that is similarly high in protein. While lowering blood cholesterol, it promotes the body's natural generation of insulin. Flavonoids and polyphenols found in cinnamon have antioxidant and cancer-prevention abilities by scavenging free radicals.<sup>11</sup>

Fenugreek is being offered as a nutraceutical with assertions to lower hyperglycemia. The effects of fenugreek on biological and pharmacology has connected to a number of its constituents, including steroids, N-compounds, polyphenolic chemicals, volatile ingredients, amino acids, etc. It was discovered that fenugreek seeds and/or seed extracts increased glucose uptake, decreased levels of glycosylated hemoglobin, proinflammatory cytokines, and pancreatic enzymes, dose-dependently restored glycogen levels in muscle and liver, inhibited lipid peroxidation, and restored some antioxidant enzymes, including glutathione (GSH) and superoxide dismutase (SOD) activities in the liver and pancreas. Fenugreek is also used to treat a number of health conditions, including diabetes, ulcers, bronchitis, menopausal symptoms, TB

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infection, fevers, sore throats, wound healing, arthritis, abscesses, swollen glands, and other skin irritations. Additionally, it resolves issues with lowering blood pressure and blood sugar levels.<sup>12</sup>

Cumin is a staple spice because its seeds contain a volatile oil that gives them a distinct scent. In addition to helping with several illnesses like tooth pain, dyspepsia, and jaundice, spices are also useful. It is a rich source of iron and supports a strong immune system. Cumin is a significant spice that is used in cooking and has antibacterial properties in its essential oil. In the presence of the procarcinogen 1,2-dimethylhydrazine (DMH), cumin inhibits the development of colon cancer. Cumin seeds contain flavonoids, which are known to enhance the antioxidant system and have antioxidant action. Cumin seeds in the diet have been shown to improve metabolic abnormalities associated with diabetes. It has been used as a carminative and eupeptic in the treatment of mild stomach-related issues, as an astringent in broncho-pulmonary disorders, as a hack remedy, or as a pain reliever, and has also been regarded as a hunger stimulant.<sup>13</sup>

Black pepper (*Piper nigrum* L.) is known as the “king of spices” since it is such a valuable medicinal herb. It contains antibiotics and tannins that help prevent hypercholesterolemia. Black pepper comprises piperine (piperinoyl-piperidine), a spicily hot nitrogenous compound. Piperine, a member of the pyridine group and the primary active component of pepper, is also used as an anti-inflammatory, an anti-malaria, and a cough suppressant. The typical pepper has a piperine level of 6%, however oleoresin has a piperine percentage ranging from 25.74 to 48.32 %. An antibacterial, antioxidant, anti-inflammatory, anti-In addition to having a substantial number of antioxidants, black pepper also has an active component that may be useful in reducing inflammation brought on by diseases like diabetes and arthritis. Additionally, black pepper improves digestion, especially when ingested raw, and some people have reported that it helps them with constipation.<sup>14</sup>

Cloves are the flower buds of the evergreen *Syzygium aromaticum* clove tree. Although cloves are most famous for being a pleasant and fragrant spice, they have also been utilized in folk medicine. Utilizing whole or ground cloves to season your meal may provide taste while also adding fiber, vitamins, and minerals. Eugenol, a substance found in cloves, has been demonstrated to have antioxidant properties.<sup>15</sup> The dried clove bud is made up of minerals, cellulose, essential oil, steam-volatile oil, resins, tannins, proteins, fixed oil, fixed oil, fixed oil, and fixed oil. The clove also has antibacterial, antiviral, antimicrobial, anti-diabetic, anti-inflammatory, antithrombotic, anti-anxiety and anti-remembering-in-agony. Numerous pains, such as those in the joints, muscles, or sinewy tissue, especially rheumatoid arthritis, are treated with clove oil.<sup>16</sup>

Red chilli is a ubiquitous spice found in most dishes. The red chilli, a plant belonging to the genus *Capsicum*, is one of the most widely used spices in the world. Through the Spanish term “chile,” this word, chile or chilli, is derived from the Nahuatl word “chilli.” In addition to having a lot of vitamin C, chili also has a lot of vitamin A, vitamin B6, vitamin K, calcium, magnesium, folate, potassium, thiamin, iron, copper, and other minerals. Chilli contains antioxidants that assist the body dealing with cholesterol. Additionally, it aids in calorie burning. The primary bioactive ingredient in chili, capsaicin, is what gives it its hot flavor and many health advantages. Capsaicin is used in a wide range of medicinal products thanks to its analgesic, anti-arthritic, anti-bacterial, anti-inflammatory, and anti-rhinitis qualities.

Additionally, it has a significant role in the management of cardiovascular illnesses, type 2 diabetes, obesity and inhibits the progression of prostate cancer. Acute tonsillitis is one more condition for which red chili is utilized as an alternative medication, along with inflammation, diabetes, and low back pain.<sup>17</sup>

Fennel is a magical cure for issues with the respiratory, endocrine, and digestive systems. Fennel includes volatile chemicals, flavonoids, phenolic compounds, fatty acids, and amino acids. It has been used for cough, flatulence, diarrhea, and constipation since ancient times. It exhibits a number of pharmacological properties, including anti-microbial, anti-pyretic, anti-spasmodic, anti-thrombotic, apoptotic, anti-viral, anti-inflammatory, anti-mutagenic, anti-nociceptive, cardiovascular, chemo-modulatory, anti-tumor, hepatoprotective, hypoglycemic, hypolipidemic, and memory-enhancing properties.<sup>18</sup>

In many cultures across the world, asafoetida is utilized both as a culinary flavoring ingredient and as a folk remedy for a wide range of illnesses including influenza, whooping cough, asthma, ulcer, epilepsy, stomachache, flatulence, bronchitis, intestinal parasites, and antispasmodic medications. Asafoetida has therapeutic purposes in addition to being used to season food, particularly snacks. Crude extracts of asafetida were tested for their antibacterial efficacy against a variety of bacterial and fungal species. An effective treatment for stomachaches brought on by gas and whooping cough.<sup>19</sup>

Utilizing the spices in the preparation of value-added ready-mix powder may offer the beneficial effects to the powder, in addition to the beautiful gustatory perception. The aim of the present study is to design value added ready mix with the above added nutrients taking into account the positive effects of the aforementioned ingredients on health.

## 2. MATERIALS AND METHODS

### 2.1 Acquisition of Materials

Flaxseeds, Oat, pulses and other ingredients (spices) were procured from local market.

### 2.2 Preparation of Ready-Mix-Powder

The approach for developing the control and

value-added Ready-Mix-Powder was standardized.

#### 2.2.1 Roasting

Oats, green gram and pigeon peas were cleaned and free from broken seeds, dust and other foreign materials manually. After that all the ingredients were roasted in iron vessel for 5 to 6 minutes at 70 °C. In an electric grinder, roasted ingredients were crushed to a coarse powder. The sample was stored in glass containers for future use at refrigerated temperature.

#### 2.2.2 Processing of flaxseed

The seeds were physically inspected to ensure they were free of debris, broken seeds, and other foreign particles. Seeds were roasted in iron vessel for 3 to 4 minutes at 70 °C. Using an electric grinder, roasted seeds were crushed into a coarse powder and kept in glass jars for later use.

#### 2.2.3 Preparation of Ready-Mix- Powder

The processed oats, green gram, pigeon peas and flaxseeds were blended with the ratio of 40%, 20%, 20% and 10% respectively in a 100g of Ready-Mix-Powder. Along with a control, various types of Ready-Mix-Powders were developed with variable combinations of different sorts of spices, which was added in a 10% ratio. The formulation of Ready-Mix-Powders were standardized by using black pepper powder, cumin powder, fenugreek powder, cinnamon powder, fennel seed powder, powdered cloves, chili flakes, salt and powdered sugar. In order to conduct an organoleptic analysis, the prepared Ready-Mix-Powder was served at room temperature.

#### ❖ Treatments

T1 -Ready-Mix-Powder prepared using 5 g of cumin and fenugreek seed powder

T2 -Ready-Mix-Powder prepared using 5 g of black pepper and cinnamon powder

T3 -Ready-Mix-Powder prepared using 5 g of cloves and fennel seed powder



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### ❖ *Formulating Recipes*

40 g of Oats + 20 g of Green Gram Dal + 20 g of Pigeon Pea Dal + 10 g of Flaxseeds+

3 g of Red-chili flakes (only for T1 & T2)

↓  
Roasted all the ingredients

↓  
It ought to be ground into a fine powder.



Add all spices per the amount of incorporation (10 g of it for each Ready-Mix-Powder)



**For T1-** Add powdered Cumin, Fenugreek seed powder, Asafetida & Salt to it and mix well

**For T2-** Add Black Pepper and Cinnamon Powder along with Asafetida & Salt to it and mix well

**For T3-** Add Cloves and Fennel Seed Powder along with granulated Sugar & Salt to it and mix well



Finally Store it in air tight vessel



T1



T2



T3

## 2.3 Proximate analysis

The products were evaluated for protein, fat, ash, crude fiber, carbohydrate and moisture content (AOAC, 2000).<sup>20</sup>

### 2.3.1 Moisture Content

1gm of sample was taken in a clean and tarred moisture dish. Then the sample was spread uniformly in the dish and the dish was placed in the hot air oven maintained at 100<sup>0</sup> C with the open lid for about 5hrs. After that, the dish was removed from oven and lid closed and cooled in a desiccator. Cooled dish was then weighed repeatedly for a constant weight and lastly the moisture content was calculated.<sup>21</sup>

### 2.3.2 Ash content

For measuring the ash content 1gm of sample was taken into a tared crucible. Predry if the sample is very moist. Place the crucibles in a cool muffle furnace and Ignite 6 hour at about 550°C. After that, turn off muffle furnace and quickly transfer crucibles to a desiccator with a porcelain plate and desiccant by using safety tong. Lastly the ash is measured and calculated.<sup>21</sup>

### 2.3.3 Fat content

2gm of predried sample was taken into a glass beaker, 20ml of petroleum ether was place into the beaker and put beaker on heater of apparatus for 30 minutes at 100°C. After that, the beaker was cooled in desiccator and weigh.<sup>21</sup>

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### 2.3.4 Protein Analysis

A 1-ml aliquot of protein solution (1–10 mg protein/ml) is combined with a 5-ml biuret reagent. Copper sulfate, NaOH, and potassium sodium tartrate are all components of the reagent, which is used to stabilize the cupric ion in the alkaline solution. The absorbance is measured at 540 nm against a reagent blank after allowing the reaction mixture to remain at room temperature for 15 or 30 minutes. If the reaction mixture is not clear, filtration or centrifugation are needed before assessing the absorbance. Bovine serum albumin (BSA) is used to create a standard curve of concentration versus absorbance.<sup>21</sup>

### 2.3.5 Determination of Total Carbohydrate

0.01gm and 0.05gm of sample was taken into a boiling tube and hydrolyze by keeping it in a boiling water bath for three hours with 5 ml of 2.5 N HCl and cool to room temperature. Solid sodium carbonate was used to neutralize it until the effervescence stops. Prepare a 100 ml volume and centrifuge it. 1 ml aliquots of the supernatant were taken for analysis. The standards were prepared by taking 0, 0.2, 0.4, 0.6, 0.8 and 1 ml of the working standard. Where 'O' serves as blank. After that 4ml of the anthrone reagent was added to it and heated for 8 minutes in a boiling water bath.<sup>20</sup>

### 2.3.6 Crude Fiber

0.5 gm of ground material was extracted with ether or petroleum ether to remove fat (initial boiling temperature 35-38°C and final temperature 52°C). Extraction can be skipped if the fat content is less than 1%. After extraction with ether, the dried extracted material was boiled with 50 ml of sulfuric acid for 30 min with glass beads. Then, filter it through filter paper and wash with boiling water until washings are no longer acidic. After that, the filtrate was boiled with 200 ml of sodium hydroxide solution for 30 min and filter it through filter paper and wash with boiling water. The residue was removed and transfer to silica crucible (preweighed) and dry it for 2hour minutes at 130 +2°C. Lastly the sample

was Ignite for 30 min at 600 ± 15°C. Cool in a desiccator and reweigh.<sup>20</sup>

### 2.3.7 Free fatty acid (FFA)

To determine the free fatty acid content, the sample (20 g) was refluxed with petroleum ether in a water bath for 14 h to extract fat. To 5 g of fat, 10 ml of warm neutral alcohol and 10 ml of petroleum ether were added along with few drops of phenolphthalein indicator and titrated against 0.1 N NaOH and the results were expressed in terms of oleic acid equivalents (AOAC 2000).<sup>20</sup>

### 2.3.8 Antioxidant Content

For the analysis of antioxidant content, 0.1 gm of sample was taken in a conical flask (100ml) and 25 ml of 1-Butanol was added. After that, the 5ml of TBA solution was added to it. The TBA was prepared by using 0.2 gm of solid TBA placed in 100ml of 1-Butanol. Then the solution was heated at 90°C for 10 minutes and leave it to cool. Finally, the absorbance was taken at 530nm and the value was calculated.<sup>20</sup>

### 2.3.9 Polyphenol Content

1gm of weighed sample was taken in a conical flask (100ml) and by adding distilled water made a 20 ml solution. From this solution 1ml of sample solution was taken and 1 ml of Folin-Ciocalteu Reagent was added. Then, the solution was made to 15ml by adding some more distilled water and kept it in a dark place for 30 minutes. Lastly, the absorbance was taken at 765nm and the value was calculated.<sup>20</sup>

### 2.3.10 Determination of fatty acid composition

Fatty acid composition of the ready-mix powders were analyzed by GC. Fatty acid ethyl esters (FAME) were prepared by the method described by Metcalfe and the compositions were determined by GC analysis. The GC (make: Agilent, model: 6890N) instrument used was equipped with an FID detector and capillary DB-Wax column (30mL, 0.32mm I.D, 0.25µm FT). N<sub>2</sub>, H<sub>2</sub> and airflow rate were maintained

at 1ml/min, 30 ml/min, and 300ml/min, respectively. Inlet & detector temperature was kept at 250°C, and the oven temperature was programmed as 150-190-230°C with an increased rate of 15°C/min and 5 min hold up to 150 °C and 4°C/min with 10 min hold up to 230°C. The percentage proportions of fatty acids were calculated.<sup>21</sup>

## 2.4 Physicochemical characteristics

### 2.4.1 Bulk density

After tapping the measuring cylinder (100 ml) on a wooden board until there was no discernible volume drop, the volume of 10 g of the product was calculated using the measuring cylinder. The apparent (bulk) density was computed using the weight and volume (Jones et al. 2000).<sup>22</sup>

### 2.4.2 Swelling power and solubility index

1 gram of the ground sample was placed in a graduated centrifuge tube together with 10 ml of water. After 30 minutes of shaking in a water bath set at 30 °C, the tube was centrifuged. The supernatant was transferred to a weighted Petri plate, evaporated to dryness over a water bath, and then dried for three hours in a hot air oven with the temperature held at 105 °C. To determine swelling power and solubility index, the remnant in the centrifuge tube and the dry Petri plates were weighed (Bello-Perez et al. 2000).<sup>23</sup>

### 2.4.3 Viscosity

In a Brookfield viscometer (model), a 10% (w/v) slurry of the sample was made by mixing 5g of material with 45 ml of distilled water (model RV; Brookfield Engineering Inc., Stoughton, USA) by using suitable spindles. The samples were heated tender in a boiling water bath, after that chilled to 30 °C and then the viscosity of the cooked paste was measured (Brandtzaeg et al. 1981).<sup>24</sup>

### 2.4.4 Dispersibility

In 100 ml of water, 5 g of the product were

evenly distributed. It was decided to filter an aliquot (10 ml) of the dispersion using filter paper that had already been pre-weighed. Separately weighted after drying, the filtrate. The number of dispersed solids was determined by solids still on the filter paper, while the number of soluble solids was determined by dried filtrates (Kulkarni et al. 1991).<sup>25</sup>

## 2.5 Sensory/ organoleptic evaluation

The method for making ready-mix powder was standardized, and the finalized three types of product's mixture of different qualities were evaluated. Among them T1 mixture contains oats, green gram dal, pigeon pea dal, flaxseeds, red-chili flakes and powdered Cumin, fenugreek seed powder, asafetida & salt mixed as spices, where T2 contains oats, green gram dal, pigeon pea dal, flaxseeds, red-chili flakes and black pepper and cinnamon powder along with asafetida & salt as to add spice to it and T3 contains oats, green gram dal, pigeon pea dal, flaxseeds, red-chili flakes and cloves and fennel seed powder along with granulated sugar & salt to taste. All the Value-Added Ready-Mix Powder was processed for sensory evaluation immediately after development. The experimental Value-Added Ready-Mix Powders were organoleptically evaluated by panel of 100 members who were familiar with the major sensory attributes of food products. They were asked to evaluate the products using 5 point hedonic



**Figure 1: Cooked Ready-mix powder**



scale with score 5 as excellent and 1 as disliking, on the basis of five parameters-

- Taste/Flavour
- TextureRating
- Aroma/Smell
- OverallAcceptability.

The degree, to which a product was liked, was expressed for appearance as like extremely attractive, moderately attractive, attractive/ matches photo, unappetizing, unattractive, for taste as like tasted great, flavorful, acceptable, off flavor, flavor did not appeal to me, for texture as like wonderful, good texture, acceptable texture, off texture, inappropriate texture/flat/runny, for aroma/ smell rating as like wonderful aroma, appealing aroma, acceptable aroma, aroma is not appealing, unappetizing aroma, for overall acceptability extremely acceptable, moderately acceptable, acceptable, moderately unacceptable, unacceptable.

## 2.6 Statistical Techniques

Per 100 g of dry substance, the values were

determined. Three separate trials were used to conduct the studies, and the findings are reported as mean standard deviation (SD).

## 3. RESULT AND DISCUSSION

### 3.1 Changes in Proximate Analysis Data of the Ready-Mix Powders

The ready-mix-powder was a homogeneous powder with light cream color and desirable aroma. The moisture content of T1, T2 and T3 are respectively 2.58g/100 g, 7g/100g and 6.98g/100g, which helped to extend its shelf life because the majority of the materials used in its manufacture were popped. A quality source of protein (For T1, T2 and T3 are respectively 37g/100g, 34.2g/100g & 28g/100g) was formed by the product.

The carbohydrate content in these different types of ready-mix-powders are T1- 36.489 %, T2-37.79 % and T3- 36.72 % which represents it as a good source of carbohydrate as shown in the Table 1. As well as it is low in fat (For T1, T2 and T3 are respectively 19.03mg/100g, 19.21mg/100g and 32.77mg/100g), will help in weight management.

**Table 1: Proximate Analysis of Ready-Mix-Powder (g/100 g)**

Content	Value for T1	Value for T2	Value for T3
Moist ure	2. 58± 0.01	3.7 6± 0.8	6 6 .98 ± 1.75
Ash	4.4± 0.10	3.7 0±0.06	3.20±0.11
Crude Fiber	2.11±0.05	3.33±0.07	1.98±0.04
Carbohydrate	36.49±0.18	37.79±0.17	36.72±0.11
Protein	37±0.31	34.20±0.72	28.35±0.06
Fat	17.42±1.4	17.22±2.66	22.77±2.05
Energy*	294.89±13.08	375.74±18.31	419.73±22.42
Calcium (mg/100 g)	234.5±1.22	163.6±1.30	191.43±0.99
Iron (mg/100 g)	7.48±0.27	6.81±0.12	6.43±0.22
Zinc (mg/100 g)	2.21±0.09	1.43±0.27	1.18±0.04
Magnesium (mg/100 g)	64.88±0.56	107.1±0.67	62.43±1.23

\*Energy= (Protein×4) + (Carbohydrate×4) + (Fat×9); Values are mean ± SD; Values are taken in triplicate

The little variations in the different nutrient concentration could be attributed to the difference in the ingredients used for the formulations of this different types of ready-mix- powders. The ash contents of these 3 types of Ready-Mix-Powder are T1-4 . 4gm/100g, T2-3 . 7gm/100g and 2.2gm/100g. Ash contents were connected to the mineral content of the formulation ingredients. The energy values for T1, T2 and T3 are respectively 294. 89Kcal/100g, 375. 74Kcal/100g and 419.73Kcal/100g which indicates it as a good energy sourced product.

The Ready-Mix-Powders also formed a good source of minerals such as calcium (For T1, T2 and T3 are re spectively 234.5mg/100gm, 163.6mg/100gm and 191.43mg/100gm), magnesium (For T1, T2 and T3 are respectively 64.88mg/100g, 107.1mg

/100g and 62.43mg/100g), iron (For T1, T2 and T3 are respectively 7.48mg/100g, 6.81mg/100g and 6.43mg/100g) and zinc (For T1, T2 and T3 are respectively 2.21mg/100g, 1.43mg/100g and 1.18mg/100g).

### 3.2 Changes in polyphenol content

The polyphenol content of these 3 types of Ready-Mix-Powder were 1.1g/100g, 0.91g/100g and 0.69 g/100g of T1, T2 and T3 respectively as shows in the Table 2 and Figure 6. Polyphenols are naturally found in foods made from plants. Studies have shown that polyphenols are essential in preventing a number of disorders, including hyper cholesterolemia, hyperglycemia, hyperlipidemia, and the resurgence of cancer.<sup>26</sup>

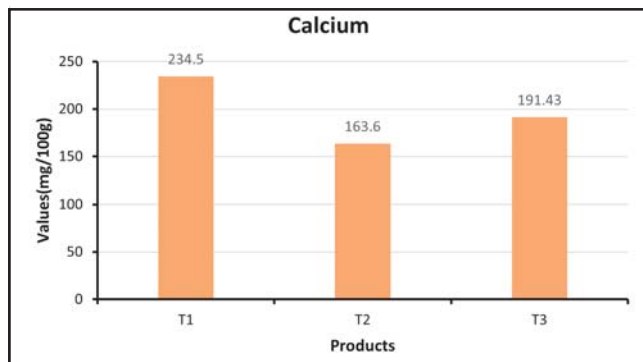


Figure 2: Bar Diagram of Calcium Content

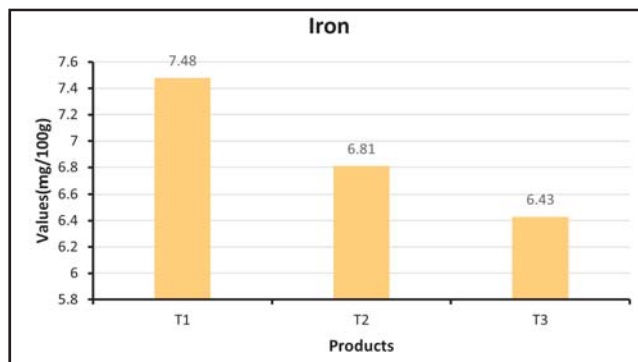


Figure 3: Bar Diagram of Iron Content

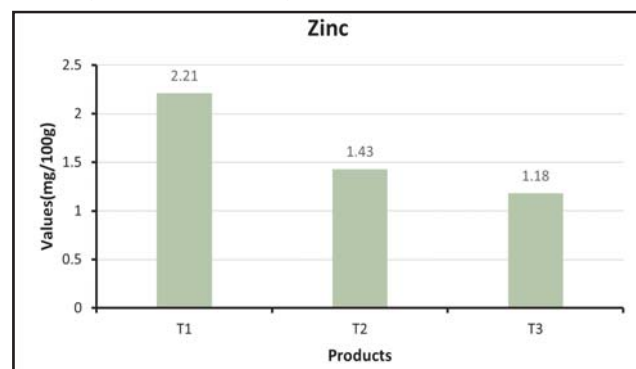


Figure 4: Bar Diagram of Zinc Content

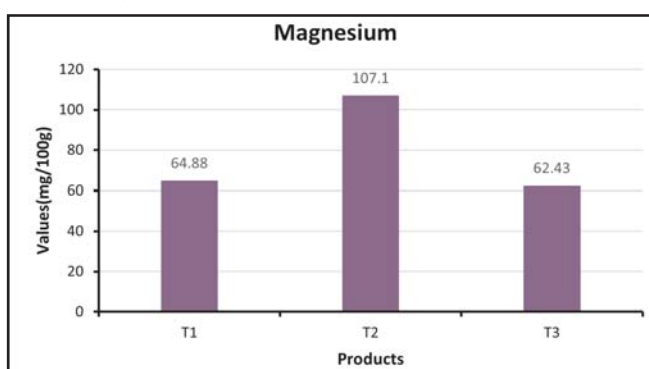


Figure 5: Bar Diagram of Magnesium Content

Table 2: Nutrient Composition of Ready-Mix-Powder (g/100 g)

Nutrients	Value for T1	Value for T2	Value for T3
Polyphenols	1.1±0.42	0.91±0.12	0.69±0.10
Anti-oxidant	0.66±0.03	0.85±0.03	0.98±0.10

Values are mean ± SD; Values are taken in triplicate

### 3.3 Changes in antioxidant content

The antioxidant content of the powders was 0.66g/100g, 0.85g/100g and 0.98g/100g of T1, T2 and T3 respectively as shows in the Table 2 and Figure 7. As the T3 ready-mix-powder content clove it has higher antioxidant content than other 2 powders. Antioxidants have received a lot of interest as food stabilizers, dietary supplements, and natural health products because they play a significant role in preventing or delaying oxidation.<sup>27</sup>

### 3.4 Changes in fatty acid composition

Table 3 depicts fatty acid composition of the ready mix powders. The fatty acid composition of these three types of ready-mix powders is lower at  $C_{18:0}$ , which is T1-3.536, T2-2.857 and T3-1.842, where the higher point is found at  $C_{18:1}$ , which is T1-36.63, T2-33.315 and T3-35.029. This indicates that the fatty acid content of these formulation in all storage conditions accelerated and normal. This was also seen by the lack of an off flavor in the product during storage, which was most likely caused by the inactivation of lipase during HTST treatment.

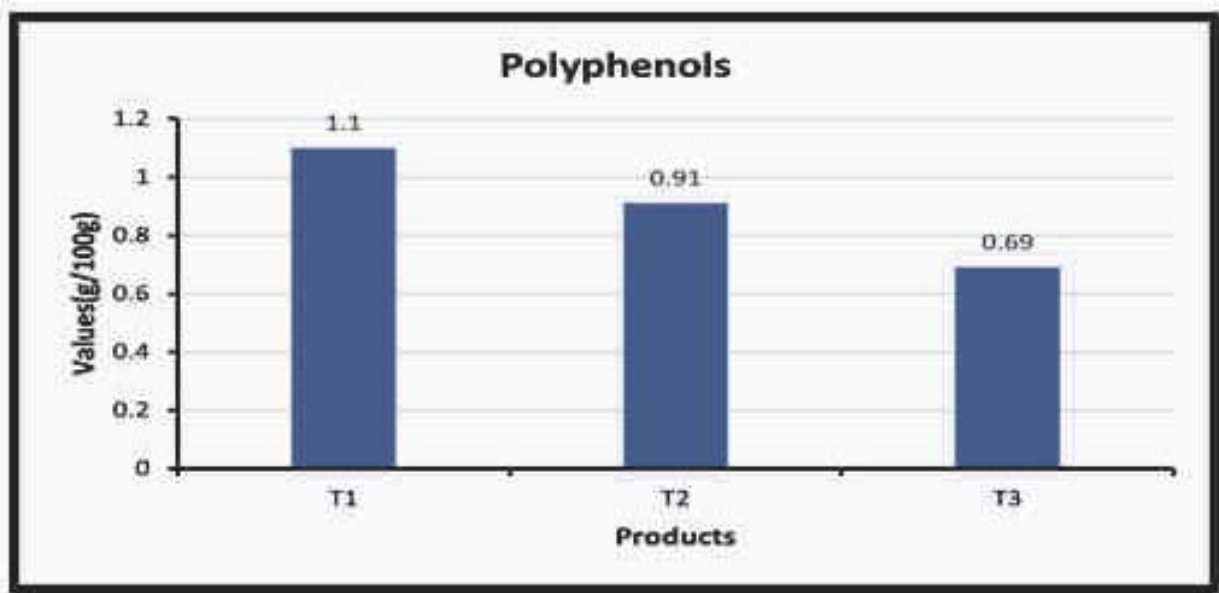


Figure 6: Bar Diagram of Polyphenols content

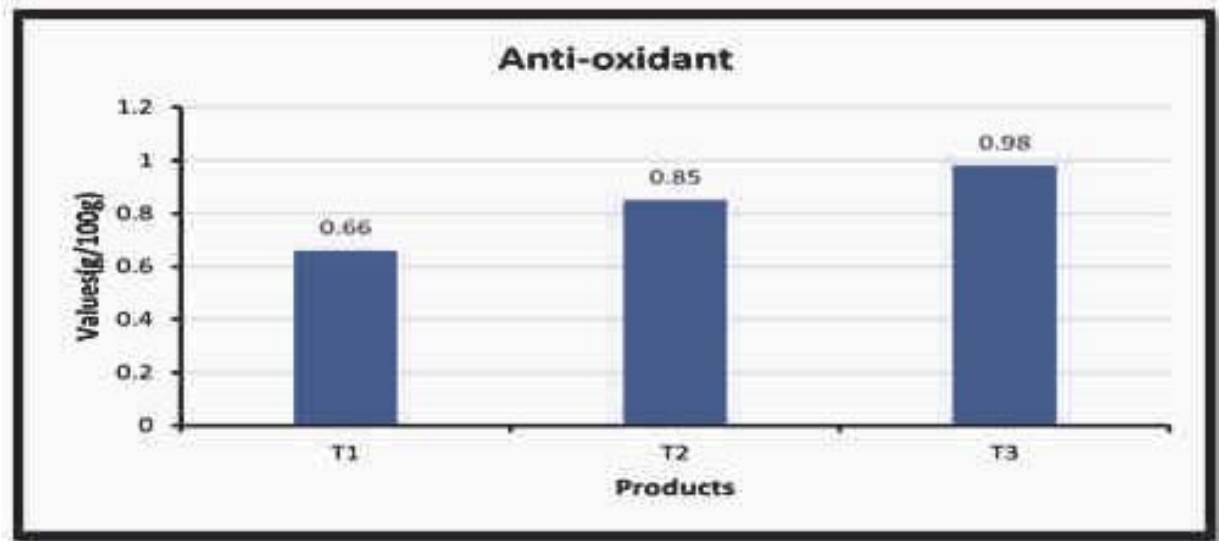


Figure 7: Bar Diagram of Antioxidant Content



### 3.5 Changes in Functional Properties of Ready-Mix Powders

The functional properties of the products is presented in Table 4. Data indicates that the product was cream colored powder. Bulk Density of the product were T1- 0.645g/ml, T2-0.60g/ml and T3-

0.60g/ml, indicating its fluffy nature. The swelling power of the products at 30°C were T1-53.5g/100g, T2- 62g/100g and T3-63g/100g and the solubility index values of the product did not differ significantly at 30°C. The product exhibited about 23.5% dispersible solids and 1.58% soluble solids for T1, 20.92% dispersible solids and 2.56% for T2, 13.78% dispersible solids and 3.62% for T3.

**Table 3: Fatty acid composition of ready-mix powder**

<div style="display: flex; align-items: center; justify-content: center;"> <div style="text-align: center; margin-right: 10px;"> ↓ Sample </div> <div style="text-align: center;"> Fatty acid → </div> </div>	Fatty Acid (% w/w)				
	C16:0	C18:0	C18:1	C18:2	C18:3
<b>T1</b>	12.86±0.22	3.53±0.18	36.63±0.22	25.03±0.43	21.93±0.66
<b>T2</b>	11.97±0.38	2.86±0.08	33.31±0.17	25.22±0.11	26.64±0.30
<b>T3</b>	11.73±0.45	1.84±0.55	35.03±0.43	23.41±0.28	27.98±0.16

Values are Mean±S.D. Values are taken in triplicate

soluble solids for T2 and 13.78% dispersible solids and 1.58% soluble solids for T3. Dispersibility is the ease with which the food powder disperses as a particle in the liquid phase. Higher numbers indicate better dispersion. It was shown that the substance in powder form could be easily dissolved in water without the creation of any lumps.<sup>28</sup> The

T1, T2 and T3 product exhibited a cold paste viscosity of 4.69CPS, 6 CPS and 4.34 CPS, which increased to 270 CPS, 240 CPS and 240 CPS respectively. The product's increased viscosity and swelling ability may be caused by the inclusion of pectin and guar gum, which expand significantly during heating and produce a comparably thick slurry.

**Table 4: Functional Properties of Ready-Mix-Powder (g/100 g)**

Parameters	Value for T1	Value for T2	Value for T3
Bulk density (g/ml)	0.645±0.028	0.60 ±0.02	0.60±0.02
Dispersibility (g/100 g)			
Dispersed solids			
Soluble solids	1.58±0.59	2.56±0.22	3.62±0.19
Water absorption capacity (g/100 g) 30°C	53.5±14.84	62±8.48	63±11.31
Water solubility index (g/100 g) at 30°C	94.25±0.21	92.55±1.2	94.4±2.82
Viscosity 10 % slurry (CPS) Cold paste	4.69±0.09	6.0±0.44	4.34±0.20
Cooked paste	270.00±1.89	240.00±2.34	240.00±3.20

Values are mean ± SD; Values are taken in triplicate

### 3.6 The comparison of Organoleptic Evaluation of Ready-Mix-Powders

Organoleptic tests of the Ready-Mix-Powders depend on its first Appearance, Taste/ Flavour, Texture Rating, Aroma/ Smell, Overall Acceptability of the product. Table 5 shows the comparison among the Ready-Mix-Powders of their organoleptic quality factors.

#### 3.6.1 Appearance Acceptability

Table 5 shows that T2 obtained lower score  $3.05 \pm 0.16$  for its appearance. Appearances of Ready-Mix-Powders depend on appearance of ingredients and on processing technique. In the appearance acceptability test, Hedonic scale showed that T3 liked very much ( $>4$ ) by the panelists.

#### 3.6.2 Taste/ Flavour Acceptability

The volatile components of the source material determine how the products taste and smell. The scores for flavor were  $3.73 \pm 0.65$ ,  $4.13 \pm 0.56$  and  $3.53 \pm 0.70$  of T1, T2 and T3 respectively shows in the Table 4. In this section showed that T2 like by the panelists.

#### 3.6.3 Texture Acceptability

The texture of the Ready-Mix-Powders depends upon the rate of the proportion of ingredients used. The mean score of texture were  $3.25 \pm 0.75$ ,  $4.05 \pm 0.14$  and  $4.20 \pm 0.56$  of T1, T2 and T3 respectively shows in Table 5. The Texture of T3 was very much liked.

#### 3.6.4 Aroma/ Smell Acceptability

As shows in the Table 5, in the section of aroma or smell acceptability T3 Ready-Mix-Powder scored high. Also, the aroma of the T2 liked by the panelists.

#### 3.6.5 Overall Acceptability

The overall acceptability of the product is also influenced by the quality of the raw materials used in the processing. The mean overall acceptability scores were  $4.14 \pm 0.57$ ,  $4.03 \pm 0.43$  and  $4.43 \pm 0.53$  of T1, T2 and T3 respectively as shows in the Table 5. In acceptability test, Hedonic scale showed that the T3 Ready-Mix-Powder was more acceptable comparing with all quality characteristics by the panelists.

**Table 5: Sensory Qualities of the Developed Product**

Ready-Mix-Powder	Appearance	Taste/Flavor	Texture Rating	Aroma/Smell	Overall Acceptability
T1	$3.08 \pm 0.12$	$3.73 \pm 0.65$	$3.25 \pm 0.75$	$3.17 \pm 0.57$	$4.14 \pm 0.57$
T2	$3.05 \pm 0.16$	$4.13 \pm 0.56$	$4.05 \pm 0.14$	$4.05 \pm 0.40$	$4.03 \pm 0.43$
T3	$4.08 \pm 0.17$	$3.53 \pm 0.70$	$4.20 \pm 0.56$	$4.17 \pm 0.05$	$4.43 \pm 0.53$

Values are mean  $\pm$  SD; Values are taken in triplicate

## 4. CONCLUSION

It can be concluded that flaxseeds can be better utilized to develop value added products.

- The proximate, other nutrient analysis and analysis of functional properties displayed the

features of three different types of Ready-Mix-Powders.

- The findings revealed that the powders provided substantial amount of carbohydrates, proteins, fats, dietary fiber, and energy contents that were of good nutritional values. The lipid, ash

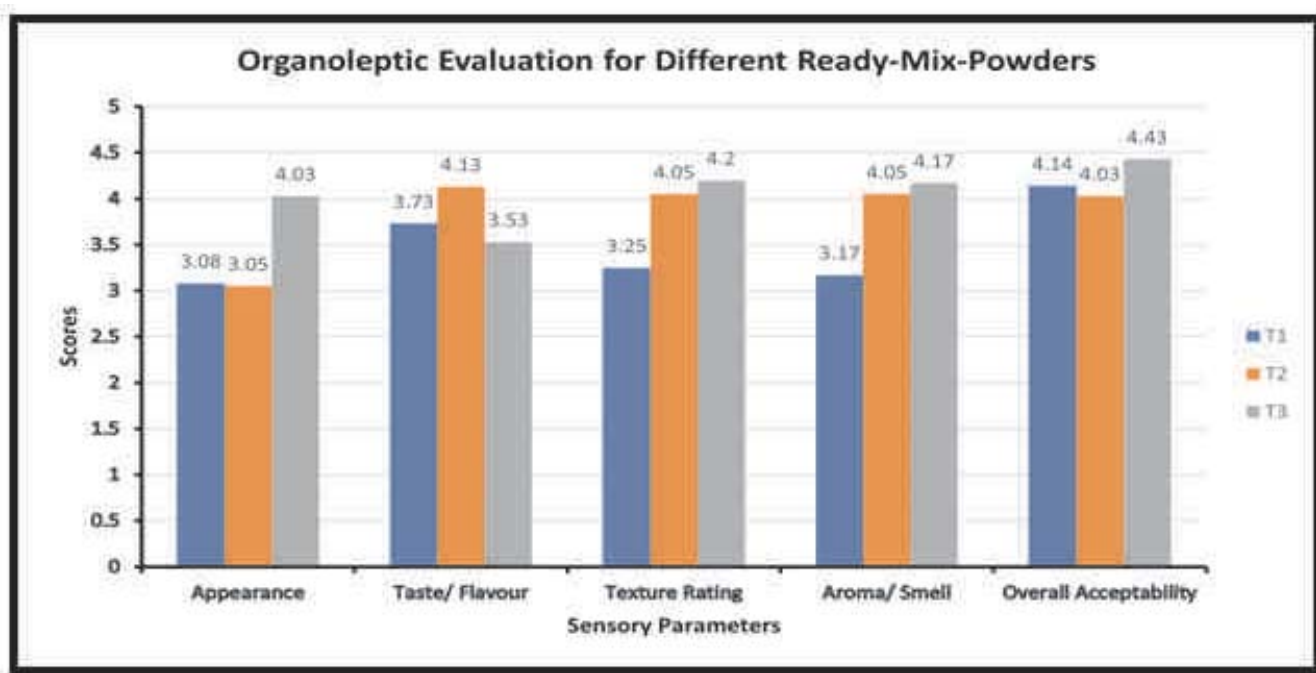


Figure 8: Bar Diagram of Sensory Evaluation

moisture, fiber, carbohydrate, and calorie levels were all within the range of those that earlier research have validated.

- Furthermore, the protein level needs to be increased to provide Ready-Mix-Powder a comprehensive nutritional value.

Sensory evaluation of Ready-Mix-Powders reveals that the products were very well accepted and can be easily formulated with less preparation. Among the three types of powders T3 - Ready-Mix-Powder prepared using 5 g of cloves and fennel seed powder was very much liked by panelists and its acceptability is higher than other two products. The Ready-Mix-Powder was determined to be acceptable for feeding youngsters as well as individuals of all ages because it had an adequate amount of protein and minerals.

Results clearly revealed high potential of the Ready-Mix-Powders prepared by incorporating the flaxseeds and spices. The production process uses locally accessible, underutilized grains, is affordable,

and is simple to implement on a small-scale to large-scale.

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# OXIDATIVE STABILITY STUDIES OF SFA, MUFA AND PUFA RICH OILS OVER A SPAN OF ONE YEAR STORAGE AT ROOM TEMPERATURE

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## ABSTRACT:

The aim of the present study was to examine and compare the oxidative stability of selected SFA, PUFA and MUFA rich filtered and refined oils. The oils were tested by using Rancimat at 120 °C. and their oxidative stability was compared in a span of one year of storage. Changes in Acid value, Peroxide value, Anisidine and Totox value were also observed. After the proper storage at room temperature for one year the best results were obtained in case of coconut and olive oil. The induction times for the peanut and rice bran oils were similar from 6.09 h to 5.73 h in the twelfth month. The shortest induction times were determined for grapeseed oil: 2.4 h and 1.6 h, respectively. A decrease of oxidative stability of about 30% was found in all the oils after 12 months of storage.

**Key Words:** Lipid Oxidation, Oxidative Stability, Edible Oil, Rancimat

## INTRODUCTION

Lipid oxidation is responsible for deterioration, spoilage of important qualities and rancidity of the vegetable oil and reduces the organoleptic characteristics of the fried food which have negative effects on the taste, colour and odour of vegetable oil. (Rossell, 2001a; Tabee, 2008; Ellen, 2008; Dutta, 2002). It adversely affects the nutritional value of oils due to the production of potentially very toxic compounds which has a direct impact on consumer

acceptance. (Abdullah, 2007). Oxidative stability is an important parameter in evaluating the quality of oils and fats, as it gives an accurate estimation of their susceptibility to oxidative degeneration. (Barmak, 2011 & Johnson 2015). Oxidation is a complex process initiated by free radical reactions at the double bonds of unsaturated fatty acids. Therefore, greater the number of double bonds or degree of unsaturation of the fatty acids, the greater the susceptibility to oxidation. (Griffiths, 2019) These reactions starts with hydroperoxides, which are rapidly decomposed to higher oxidation products as aldehydes, ketones, alcohols, hydrocarbons, esters, furans and lactones ((Choe & Min, 2006; Adetola et al., 2016)

A thorough knowledge of the ability to oxidation of vegetable oils has become a topic of prime importance, because these oils may be used in food, pharmaceutical, and industrial applications (paints, rubber, base lubricants, cosmetics, etc.).

Different experimental Techniques have been developed and used to assess the resistance of fats and oils to oxidation (Amir, 2013). Chemical determination of Acid Value (AV), Peroxide value (PV) p-Anisidine value (AnV) and Totox value are being used to determine the quality of oil since long Among the available Instrumental techniques, the Rancimat test is the most commonly used, due to its simplicity and repeatability and accuracy. Moreover, this method may be seen as a green technique as organic solvents are not required in this technique.





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The method of Rancimat accelerates the oxidation process by a high temperature and a continuous stream of air that passed through the sample. The fatty acid molecules are then oxidized in the sample. The first oxidized products to develop are peroxides. After a certain time, the fatty acids are completely destroyed and secondary oxidation products appear. These include, volatile organic acids with low molecular weight, such as acetic acid and formic acid will be driven by the current air in a second container, which contains distilled water and allows continuous measurement of conductivity. The presence of volatile acids in the measuring vessel results in an increase in conductivity. The time that elapses until the appearance of the product of secondary reaction is the induction time, the period Induction or Oil Stability Index (OSI). This value characterizes the resistance of the sample to oxidation (Farhoosh, 2008) The longer the induction time, the longer the sample will be stable. The induction time measured according to the Rancimat method is a standard parameter of quality control in manufacturing,. It is used in production plants and food processing. In addition to oils and vegetable fats, the Rancimat naturally allows determining the oxidation stability fats of animal or vegetable origin. Foods usually contain antioxidants, which slow down the oxidative decomposition of oils or fats. These antioxidants can be original natural or artificially added.

The primary goal of this study is to estimate the Oxidative Stability of different SFA, PUFA and MUFA rich edible oils stored at room temperature over a span of one year to get an approximate idea about oxidation products formation. The induction time reflects the interaction of bound and free fatty acids, the number of double bonds, existing antioxidants and existing pro-oxidants at elevated temperatures. This gives an overall picture of the quality of an oil and how it will act over time Therefore, measuring induction time on the Rancimat allows us to get as much value from the oil as possible by determining the shelf life and deciding if further actions are required.

## Materials and Methods:

A total number of 9 samples of edible oils were chosen for the study, Coconut, Palm, Olive, Rice bran, Sunflower, and Mustard, Peanut, Soybean and Sesame oil. Out of 9 oils were 5 were filtered and rest were refined. The oils selected were within similar manufacture month (within one month of manufacturing date) and expiry period. All the samples were subjected to room temperature.

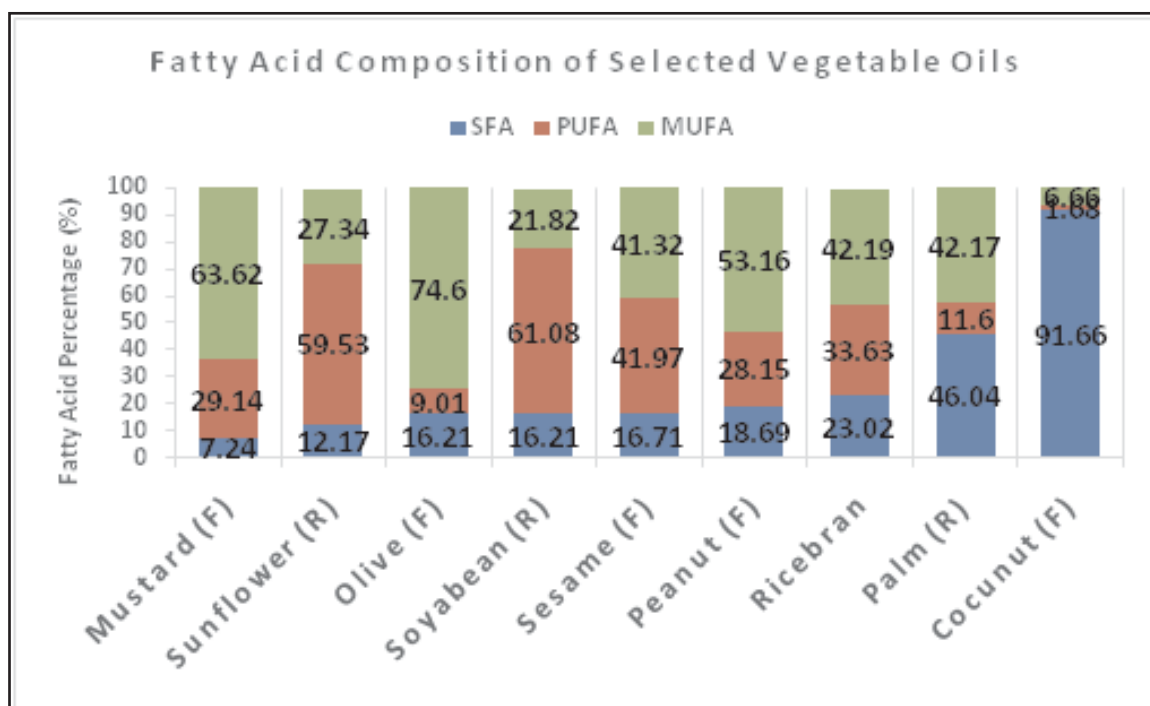
The fatty acid composition of the oil samples was determined according to the AOAC Official Method 996.06/969.33, with minor modifications. Fatty acid methyl esters (1 mL), prepared by AOAC standard method, were separated on a GC-FID system (8890, Agilent, Santa Clara, CA, USA) equipped with a SP-2560 capillary column (length 100 m, i.d. 0.25 mm, film thickness 0.2 µm). Helium was used as a carrier gas. A split/split less injector was operated at a temperature of 230 °C with a split rate set to 100:1, and the detector was the GC-FID. The GC's oven temperature was programmed as follows: 80 °C hold for 2 min, ramped to 230 °C at a rate of 2.5 °C/min, hold for 5 min. Fatty acids were identified by comparing their retention times with authentic standards (Sigma-Aldrich, SUPELCO 37 Component FAME Mix, catalog # CRM47885) and the results were reported as weight percentages.

Alterations in chemical properties and primary and secondary oxidation products were studied and statistically analysed at the beginning of the study and after one year of storage all the oils were studied for chemical properties; to measure primary and secondary oxidation products. Acid value and Peroxide value (PV) (as a measure of primary oxidation product) were determined by IS method (IS 548 P-1). Acid value was calculated in mg of KOH per gm of oil. Peroxide value was calculated in mill equivalent of peroxide per kg of oil sample. Anisidine value (AnV) and Totox value (TV) as a measure of secondary oxidation products. Anisidine value was determined by AOCS Cd 18-90. method. Totox value was calculated from peroxide value and p-Anisidine value as  $\text{Totox} = 2 * \text{PV} + \text{AnV}$ .

The induction period (time in hours) of the oil samples was measured using Metrohm 892 professional Rancimat equipment in which air (20 ml/min) was bubbled through 2.5 g of sample at 120 °C. The change in conductivity of 60 ml distilled water versus time was observed and the induction period (IP) was calculated using Metrohm software Stabnet 1.1.

## Results & Discussion:

Fatty acid composition of the studied oils is presented in Figure 1. The share of saturated fatty acids ranged from 7.24% in rapeseed oil to 91.66 % in coconut oil the highest percentage of monounsaturated acids were found in Olive oil Mustard oil and Peanut oil as 74.60%, 63.62 % and 53.16 percentage respectively. The largest share of polyene acids were found in Soyabean and Sunflower oil 61.08 % and 59.53 %.



**Figure 1: Shows the fatty acid composition selected of edible oils. Coconut oil was found to be most saturated, and soya bean oil was most unsaturated having highest PUFA Content**

(F) Filtered (R) Refined

SFA: Saturated Fatty Acid

MUFA: Monounsaturated Fatty Acid

PUFA: Polyunsaturated Fatty Acid

## Chemical properties:

Primary and secondary oxidation products were measured for selected vegetable oils in first month and 12<sup>th</sup> month of study and were statistically analysed and compared. The increase in Peroxide value indicates that storage for longer time potentiate

the formation of lipid peroxide molecules (Nkpa et al., 1990). It was observed that initially the peroxide value was in the stipulated limits defining the good quality of oil. After the 12-month storage period, the Peroxide value didn't increase much, but variations were observed in Anisidine value and Totox value because of the secondary oxidation products.

**Table: 2 Acid Value, Peroxide Value p-Anisidine value and Totox value of Selected Vegetable Oils**

Oil	AV		PV		AnV		TOTOX	
	1 Month	12Month	1 Month	12 Month	1 Month	12 Month	1 Month	12 Month
Coconut	0.42	0.78	0.29	0.62	1.13	4.35	2.81	5.59
Palm	0.12	0.27	0.49	0.86	2.45	4.12	3.43	5.84
Olive	0.24	0.79	0.46	0.75	2.61	4.65	3.53	6.15
Peanut	1.21	2.42	1.19	2.57	3.52	5.52	6.54	10.66
Mustard	0.86	1.29	0.89	1.51	3.19	4.61	5.17	7.63
Sunflower	0.12	0.26	0.69	2.23	2.51	5.49	3.88	9.95
Soyabean	0.10	0.32	0.62	2.34	2.03	5.82	3.27	10.26
Rice bran	0.16	0.35	0.82	1.49	33.01	54.12	34.65	57.10
Sesame	4.86	5.76	1.49	2.59	5.89	9.54	9.17	14.72

AV: Acid Value

PV: Peroxide Value

p-AnV : Para Anisidine Value

### Rancimat Analysis:

The oxidative stability of the SFA, MUFA and PUFA rich oils as calculated by Stabnet 1.1 software was in the range of 2.99 to 30.58 hrs. Induction Period in hours by the Rancimat method is presented in Table 3 and Figure 2.

**Table: 3 Induction Period of selected oils at 120 °C**

Induction period of Selected oils at 120 °C			
S/No	Oil	Induction Time 1s Month (hrs)	Induction time 12th Month (hrs)
1	Coconut	30.58	28.9
2	Peanut	8.73	6.09
3	Mustard	5.13	4.18
4	Oilive	7.84	5.13
5	Sunflower	6.78	3.86
6	Soyabean	6.53	4.82
7	Ricebran	6.57	5.73
8	Sesame	3.65	2.99



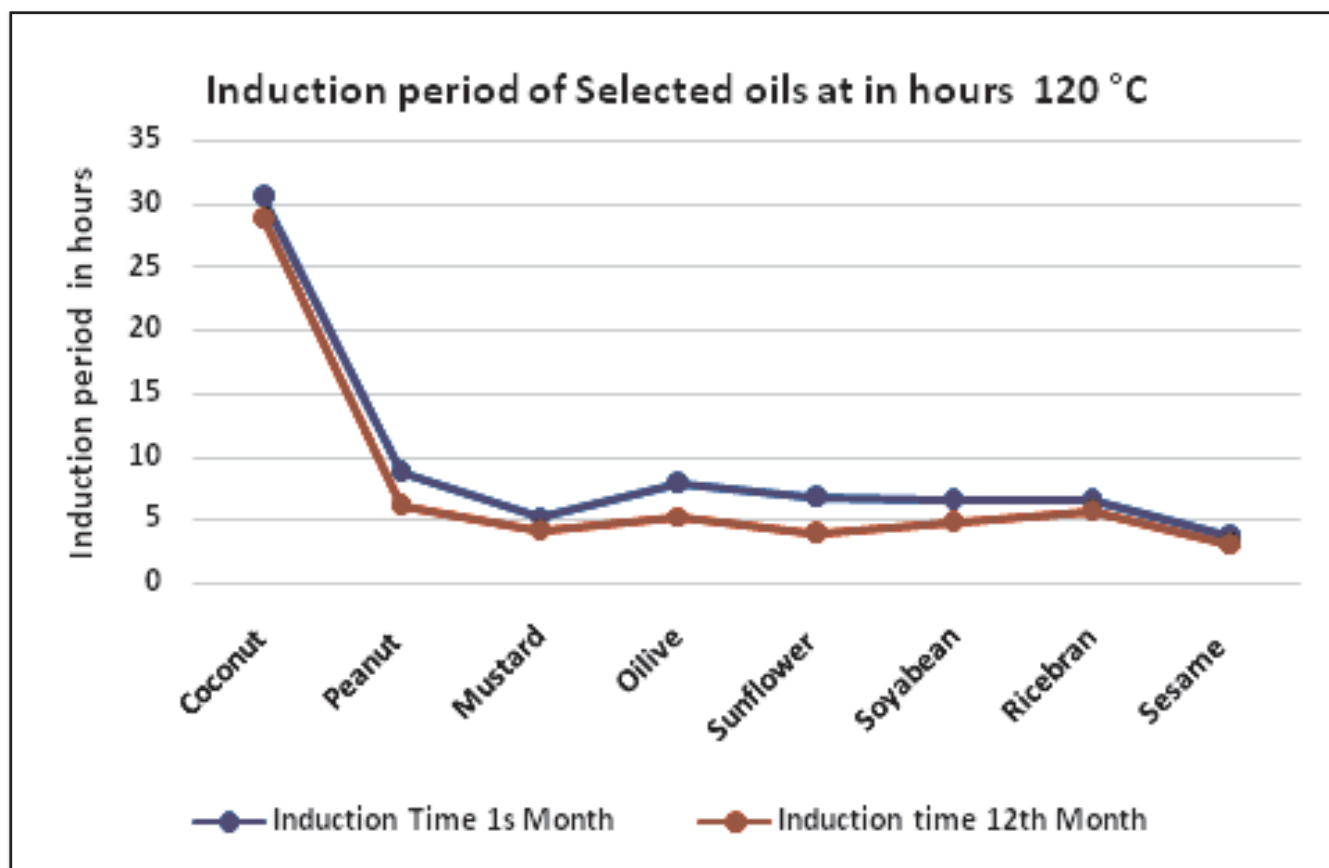


Figure: 2 Induction Time of selected oils in hrs at 120 °C

## Conclusion:

**In the present study it is concluded that based on the result obtained** Storage at room temperature and exposure to light intensify the oxidative reactions. These were characterized by an increase in free fatty acids and peroxide values, and a reduction in the induction period. It was found that among all vegetable oil Soyabean and Sunflower Oil are prone to oxidative deterioration during one year storage period. Coconut Palm and Olive showed better results after in first month and after 12<sup>th</sup> month of storage because of saturated fatty acid content. Natural antioxidants is effective in ensuring oxidative stability of vegetable oils like Rice bran and sesame oil.. Further studies on different oils and under the effect of different temperatures and should be done to confirm these findings.

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## RESEARCH ROUNDUP

### Biodiesel production from wild mustard (*Sinapis Arvensis*) seed oil using a novel heterogeneous catalyst of LaTiO<sub>3</sub> nanoparticles

**Shahabaldin Rezanian *et al*** synthesized a novel heterogeneous catalyst using lanthanum titanium dioxide (LaTiO<sub>3</sub>) nanoparticles via co-precipitation and calcination processes [**Fuel**, **307**, Article 121759,(2022)]. The proposed LaTiO<sub>3</sub> NPs were used for biodiesel production from wild mustard seed oil in the presence of methanol. The solid catalyst material was characterized by Fourier-transform infrared spectroscopy (FT-IR), X-ray diffractometry (XRD), and scanning electron microscopy (SEM). The process of trans-esterification was affected by the reaction conditions, such as the molar ratio of alcohol to oil (4:1), amount of catalyst (100 mg), reaction time (60 min), and temperature (80 °C). Under optimized conditions, the highest rate of biodiesel was 92.21% at 80 °C for 60 min in the presence of LaTiO<sub>3</sub> NPs. Finally, the catalyst showed a maintained performance for at least five runs and up to eight runs. This result implied that the reported catalyst has a high potential to produce biodiesel at a reasonable cost and appropriate reaction conditions.

### Principles, Advances, and Perspectives of Anaerobic Digestion of Lipids

Several problems associated with the presence of lipids in wastewater treatment plants are usually overcome by removing them ahead of the biological treatment. However, because of their high energy content, waste lipids are interesting yet challenging pollutants in anaerobic wastewater treatment and codigestion processes. The maximal amount of waste lipids that can be sustainably accommodated, and effectively converted to methane in anaerobic reactors, is limited by several problems including adsorption, sludge flotation, washout, and inhibition. These difficulties can be circumvented by appropriate feeding, mixing, and solids separation strategies, provided by

suitable reactor technology and operation. In recent years, membrane bioreactors and flotation-based bioreactors have been developed to treat lipid-rich wastewater. In parallel, the increasing knowledge on the diversity of complex microbial communities in anaerobic sludge, and on interspecies microbial interactions, contributed to extend the knowledge and to understand more precisely the limits and constraints in influencing the anaerobic biodegradation of lipids in anaerobic reactors. This critical review discusses the most important principles underpinning the degradation process and recent key discoveries and outlines the current knowledge coupling fundamental and applied aspects. A critical assessment of knowledge gaps in the field is also presented by **B. Conall Holohan *et al*** integrating sectorial perspectives of academic researchers and of prominent developers of anaerobic technology [**Environmental Science & Technology** **56**, 4749-477,(2022)].

### Reflectance spectroscopy with operator difference for determination of behenic acid in edible vegetable oils by using convolutional neural network and polynomial correction

A novel polynomial correction method, order-adaptive polynomial correction (OAPC), was proposed by **Shizhuang Weng *et al*** to correct reflectance spectra with operator differences, and convolutional neural network (CNN) was used to develop analysis model to predict behenic acid in edible oils [**Food Chemistry**, **367**, Article 130668 (2022)]. With application of OAPC, CNN performed well with coefficient of determination<sup>2</sup> of correction ( $R_{cor}$ ) of 0.8843 and root mean square error of correction ( $RMSE_{cor}$ ) of 0.1182, outperforming partial least squares regression, support vector regression and random forest with OAPC, as well as the cases without OAPC. Based on 16 effective wavelengths selected by combination of bootstrapping soft shrinkage, random frog and Pearson's correlation, CNN and 2

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OAPC exhibited excellent performance with  $R_{cor}$  of 0.9560 and  $RMSE_{cor}$  of 0.0730. Meanwhile, only 5% correction samples were selected by Kennard–Stone for OAPC. Overall, the proposed method could alleviate the impact of operator differences on spectral analysis, thereby providing potential to correct differences from measurement instruments or environments.

### Dehydration of fatty acid methyl ester mixtures from enzymatic biodiesel using a modified PVDF membrane

Miquel L. Padula *et al* investigated the efficiency of microfiltration with a polymeric membrane in the dehydration of fatty acid ester mixtures. The dip-coating method was used to modify the surface of polyvinylidene fluoride (PVDF) hydrophilic membranes using silazane to render them hydrophobic [Renewable Energy, 187, pp. 237-247, (2022)]. The membranes were characterized by the contact angle, Fourier-transform infrared spectroscopy (FTIR), zeta potential, field emission scanning electron microscopy (FESEM), membrane water absorption, silazane crosslinking to the membrane surface, and chemical compatibility of membranes to methanol. The microfiltration experiments were carried out in the dead-end and crossflow configurations. Membrane performance was evaluated by permeate flux values, mass reduction ratio, moisture, acidity, and glycerol content in the permeate. Mixtures with different compositions and concentrations of methyl ester, methanol, water, oleic acid, glycerol, and enzyme were used. Silazane deposition time of 60 min at 1 mg silazane/g toluene resulted in the best performance. The modification resulted in chemical and morphological changes in the membrane surface to render it hydrophobic changing its water contact angle from 0 to 94°. For dead-end and crossflow filtration configurations, the modified membranes presented the highest fluxes when compared to the unmodified control membrane, regardless of the composition of the mixtures. All biodiesel mixtures treated with the modified membrane had a decreased water content and an increased oleic acid content in the permeate. This work demonstrated that biocatalyzed fatty acid methyl esters (FAME) can be dehydrated using silazane-

modified membranes, resulting in increased separation efficiency.

### Sulfated - Alumina - Catalyzed Triacetin Synthesis: An Optimization Study of Glycerol Esterification

The catalytic activity of sulfated  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> was evaluated by Felipe Baião Ferreira *et al* for glycerol esterification using response surface methodology (RSM) with a central composite design (CCD), having as response variable triacetin (TAG) yield [Industrial & Engineering Chemistry Research, 61, 4235-4243, (2022)]. Temperature, glycerol/acetic acid molar ratio, and catalyst load were chosen as variables. The analysis of variance (ANOVA) verified that, for the studied range, only the temperature, molar ratio, their interactions, and quadratic terms were significant statistically. Among the adopted conditions for sulfated  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>, the best result attained was 100% glycerol conversion and 39.9% TAG yield at 106.8 °C, 1:8 molar ratio, and 0.75 wt % catalysts. The optimization indicated a maximum yield of 43.6% at 106.8 °C, 1:11.4 molar ratio, and 0.53 wt % SO<sub>4</sub>/γ-Al<sub>2</sub>O<sub>3</sub>. The kinetic study of the reaction rate suggests the first-order dependence on glycerol concentration and activation energy of 59.91 kJ mol<sup>-1</sup>.

### Enhancing oleic acid and oil content in low oil and oleic type Indian safflower (Carthamus tinctorius L.)

Indian safflower (*Carthamus tinctorius* L.), an oilseed crop, is low in oleic acid (14–20%) and oil (26–32%) levels. An attempt was made by Anjani Kammili and Praduman Yadav to improve oleic acid and oil levels in Indian safflower considering the high global demand for oleic oils [Industrial Crops and Products, 175, Article 114254, (2022)]. A parent possessing low oil (30%) and high oleic acid (74%) contents was crossed to two parents having low levels of oil (28% and 30%) and oleic acid (18% and 14%) contents. Progenies possessing high oil and high oleic acid were selected in F<sub>3</sub>-F<sub>4</sub>, and the best were advanced to F<sub>6</sub>. Fifty eight high oil and high oleic F<sub>6</sub> lines were tested for three years for oil and oleic acid levels and two years



for seed yield under unirrigated conditions. Ten inbred lines possessing 36.11–41.01% oil and 77.01–82.56% oleic acid were finally shortlisted. One line, ISF-1264 (Ole) gave seed yield (2480 kg/ha) comparable to the high yielding variety check, A1 (2460 kg/ha) with a 56% increase in oil yield (992 kg/ha) and 6.71 times increase in oleic acid yield (805 kg/ha) over A1 while the remaining nine lines could not compete with A1 for seed yield but gave 13–32% higher oil yield and 4.65–5.7 times higher oleic acid yield than A1. These lines would help in increasing safflower oil and oleic acid production in India, and make safflower cultivation more rewarding to farmers besides increasing the market value of Indian safflower oil once released as varieties for commercial cultivation.

### Oil produced from Ghana cocoa bean for potential industrial applications

Cocoa bean (*Theobroma cacao* L.) oil is a promising intermediate cocoa product with few research demonstrating its potential for the direct production of industrial cocoa products. **Samuel Kofi Tulashie *et al*** highlighted the extraction and characterisations of oil obtained from the Ghana cocoa bean crop to evaluate its suitability for industrial applications. A solvent extraction method was employed to extract the oil at variable extraction temperatures and times [**Industrial Crops and Products**, **177**, Article 114426, (2022)]. Kinetic and thermodynamic models were adopted to study the oil's extraction at varying temperatures and times. The maximum yield of the oil (21.62%) was reached at the highest temperature, 333 K (at 130 min), following first-order kinetics. The mass transfer ( $k_m$ ) and regression coefficient ( $r^2$ ) were  $0.0389 \pm 0.0045 \text{ min}^{-1}$  and  $0.9993 \pm 0.0005$ , respectively. The activation energy ( $E_a$ ), entropy change ( $\Delta S$ ), equilibrium constant ( $K$ ), and enthalpy change ( $\Delta H$ ) were  $15.57 \text{ kJ mol}^{-1}$ ,  $276.13 \text{ J/(mol K)}^{-1}$ , 5.64, and  $78.11 \text{ kJ mol}^{-1}$ , respectively. The activation enthalpy ( $\Delta H^*$ ), entropy ( $\Delta S^*$ ), and Gibb's free energy ( $\Delta G^*$ ) were  $12.87 \pm 0.05 \text{ kJ mol}^{-1}$ ,  $-258.88 \pm 0.17 \text{ J/(mol K)}^{-1}$  and  $97.13 \pm 1.67 \text{ kJ mol}^{-1}$ , respectively, favouring a forward, irreversible, endothermic, and spontaneous extraction. The major

fatty acids identified in the oil were stearic acid (37%), oleic acid (34%), and palmitic acid (26%). The iodine value, peroxide value, saponification value, unsaponification value, and free fatty acids of the oil were below the recommended standards acceptable for industrial applications. Differentials canning calorimetry–thermal gravimetric studies showed that the oil was thermally stable at high temperatures until thermal decomposition occurred around 260 °C. The oil's oxidation was monitored with computational modelling and Fourier infrared red spectroscopy, which found the oil highly stable despite the thermal extraction.

### Etherification of glycerol into short-chain polyglycerols over MgAl LDH/CaCO<sub>3</sub> nanocomposites as heterogeneous catalysts to promote circular bioeconomy

Glycerol is a byproduct from biodiesel production via conventional transesterification processes, representing approximately 10 wt% of the mass of biodiesel produced. Because of increasing biodiesel consumption, the volume of glycerol being produced has grown significantly, leading to a large surplus and, consequently, a dramatic drop in its market value. Thus, the valorization of glycerol into chemicals is a promising pathway toward sustainability in biodiesel industries. This study by **Rujeeluk Khumho *et al*** focused on upgrading biodiesel plant-derived glycerol into short-chain polyglycerols (PG), which are used as intermediates for producing emulsifiers in several consumer products, via catalytic etherification [**Chemosphere**, **291**, Article 133091, (2022)]. To enhance environmental sustainability, solvent-free etherification of glycerol was performed over mixed oxides derived from magnesium–aluminum layered double hydroxides (MgAl LDH). For the first time, natural dolomite, a mixed calcium and magnesium carbonate ( $\text{CaMg}[\text{CO}_3]_2$ ), was used as an Mg source in the preparation of MgAl LDH/CaCO<sub>3</sub> nanocomposites via hydrothermal synthesis. The calcined MgAl LDH/CaCO<sub>3</sub> nanocomposites were characterized by highly dispersed small crystallites of magnesium oxide. Their textural and acid–base

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properties were tuned by varying the Mg:Al molar ratio. The MgAl LDH/CaCO<sub>3</sub> (an Mg:Al molar ratio of 1:1) calcined at 500 °C exhibited a superior catalytic performance to the MgAl LDH available commercially and the one synthesized by conventional co-precipitation. The nanocomposite catalyst displayed selectivity of >99% toward short-chain PG at 52.1 mol% glycerol conversion.

### **Synthesis and catalytic properties of calcium oxide obtained from organic ash over a titanium nanocatalyst for biodiesel production from dairy scum**

The fatty acid methyl ester (FAME) production from dairy effluent scum as a sustainable energy source using CaO obtained from organic ash over titanium dioxide nanoparticles (TNPs) as the transesterification nano-catalyst has been studied by **Walid Nabgan *et al*** [**Chemosphere**, **290**, Article 133296, (2022)]. The physical and chemical properties of the synthesized catalysts were characterized, and the effect of different experimental factors on the biodiesel yield was studied. It was revealed that the CaO–TiO<sub>2</sub> nano-catalyst displayed bifunctional properties, has both basic and acid phases, and leads to various effects on the catalyst activity in the transesterification process. These bifunctional properties are critical for achieving simultaneous transesterification of dairy scum oil feedstock. According to the reaction results, the catalyst without and with a low ratio of TNPs showed a low catalytic activity. In contrast, the 3Ca–3Ti nano-catalyst had the highest catalytic activity and a strong potential for reusability, producing a maximum biodiesel yield of 97.2% for a 3 wt% catalyst, 1:20 oil to methanol molar ratio for the dairy scum, and a reaction temperature of 70 °C for a period of 120 min under a 300 kPa pressure. The physical properties of the produced biodiesel are within the EN14214 standards.

### **Determination of polycyclic aromatic hydrocarbons in commercial olive oils by HPLC/GC/MS – Occurrence, composition and sources**

Polycyclic aromatic hydrocarbons (PAHs) are a large class of organic compounds produced from incomplete combustion. Many PAHs are mutagenic and some are carcinogenic and pose a health risk to humans. Dietary intake of PAHs is a major route of exposure, where fats and edible oils are important contributors to overall dietary PAH exposure. Composed of hundreds of individual compounds as a complex mixture, only 16 PAHs are typically monitored in food and the environment. In this present study, **Holly Ekner *et al*** analyzed 16 commercial olive oil samples from different countries of origin and type (virgin or refined oil) for their content of 45 PAHs using a high-performance liquid chromatograph coupled to a gas chromatograph with a mass spectrometric detector [**Food Control**, **132**, Article 108528, (2022)]. The content of the 45 PAHs varied between 9.17 and 94.7 µg/kg (median: 30.1 µg/kg) in the different olive oil samples. Only one sample didn't meet the regulatory threshold levels for PAHs. The compositional profile of PAHs across the olive oil samples showed a high abundance of PAHs of lower molecular weights, and a large contribution of alkylated PAHs regardless of olive oil type. Direct contact with diesel exhaust emissions from mechanical harvesters has previously shown to affect PAH levels in olive oils. Using diagnostic PAH ratios, biomass/coal combustion and/or petroleum/fossil fuel combustion were indicated as important sources. Source apportionment by Positive Matrix Factorization revealed diesel exhaust emission and biomass combustion as the two major sources of PAHs followed by traffic emissions. This suggests that air quality may have a considerable impact on pollution levels in olive oils and thus indirectly affect dietary exposure.

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## Soybean oil bodies: A review on composition, properties, food applications, and future research aspects

Soybean has small and stable oil bodies that make up 18%–22% of its total mass. These oil bodies are organized droplets of triacylglycerol rich in minor bioactive constituents. They are surrounded by a monolayer of phospholipids in which seven oleosins, two caleosins and one steroleosin are embedded. After standard aqueous extraction, the soybean oil bodies (SOBs) acquire a second protein layer consisting mainly of lipoxygenase, glycinin,  $\beta$ -conglycinin and Bd 30K/P34. As part of the construction of many food products, SOBs have been extensively studied to understand their properties and interactions with other components to replace the traditional oil-in-water emulsions. Nevertheless, SOBs exhibit undesirable behavior under certain conditions that could be overcome by coating them with polysaccharides. This article by **Farah zaaboul *et al*** provides a comprehensive overview of the structure and composition of soybean oil bodies and discusses the use of their properties in the main successful food applications [**Food Hydrocolloids**, **124**, Part A, Article 107296, (2022)].

## Efficient production of biodiesel with electric furnace dust impregnated in Na<sub>2</sub>CO<sub>3</sub> solution

Solid catalyst (Na<sub>2</sub>CO<sub>3</sub>@EFD) was prepared by **Yi-Tong Wang *et al*** by wet impregnation of electric furnace dust (EFD) in aqueous Na<sub>2</sub>CO<sub>3</sub> solution. It had high basicity and acidity of 0.34 and 0.16 mmol/g for biodiesel production [**Journal of Cleaner Production**, **330**, 129772, (2022)]. High biodiesel yield of 99.8 wt% from soybean oil was obtained under the optimized reaction conditions (by Central Composite Design) of 71 °C in 111.36 min with 5.4 wt% catalyst and methanol/oil molar ratio of 11.8/1. After 11 cycles, biodiesel yield still maintained at 90.8 wt% with catalyst recovery rate >90 wt% by magnetic separation of catalyst EFD powders (containing Fe<sub>3</sub>O<sub>4</sub> with magnetism of 59.1 Am /kg). Pure Na<sub>2</sub>CO<sub>3</sub> particles presented poorer recyclability with lower biodiesel yield of 89.5 wt% even at the eighth cycle by centrifugal

separation. Na<sub>2</sub>CO<sub>3</sub>@EFD catalyst had high activity and recyclability because: (i) EFD as support hosted nanoparticles of Na<sub>2</sub>CO<sub>3</sub> (30.3 nm) as main base site for transesterification; (ii) porous EFD support provided acidic sites from metal oxides (e.g., ZnO and Al<sub>2</sub>O<sub>3</sub>) for esterification; (iii) EFD adsorbed active components into its micropores to maintain high recyclability; and (iv) EFD magnetism from magnetic Fe<sub>3</sub>O<sub>4</sub> kept high efficient magnetic separation. Total metals in the blended biodiesel met the National Standard of China and heavy metals were lower than typical petrochemical diesel. The study gave a practical use of industrial solid waste for the green production of biodiesel.

## Review of synthesis, characteristics and technical challenges of biodiesel based drilling fluids

Over the last decades, biodiesel-based drilling fluids have drawn attention of researchers due to their biodegradability, low cost, high flash point compared to other synthetic-based drilling fluids. This review by **Amany A. Aboulrous *et al*** discusses the development of biodiesel-based drilling fluids and analyzes the characteristics of biodiesels (based on the required range of values for their properties) that lead to better understanding in choosing the proper biodiesel for the drilling fluids [**Journal of Cleaner Production**, **6**, 130344, (2022)]. Although, using biodiesels as the base oil in drilling fluids, showed a favourable performance, it still poses some technical challenges such as excessive viscosity, poor pour point, low hydrolytic stability and water-in-biodiesel emulsion stability. In this study, recommended solutions including chemical modification of biodiesel's molecular structure, additives and mixing with other oils are presented. It was highlighted that the modification of biodiesel structure or the use of additives can be expensive and promote incompatibility with the other drilling fluids components. On the other hand, blending biodiesels with other base oils, showed promising solutions for the challenges of high viscosity, poor pour point and hydrolytic stability. It was found that the environmentally-friendly oils to mix with biodiesels, are synthetic esters, paraffins and linear alpha olefins.

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Furthermore, recent studies have suggested the use of nano materials (with hydro phobic characteristics) to stabilize water-in-biodiesel emulsion drilling fluids.

### **Biodiesel production from Custard apple seeds and *Euglena Sanguinea* using CaO nano-catalyst**

**S Sivanesh *et al*** investigated biodiesel production from *Euglena Sanguinea* microalgae and custard apple using nano CaO as a heterogeneous catalyst [**Bioresource Technology**, **344**, Part B, Article 126418(2022)]. Different solvents were used to extract the oil at a fixed speed, time, and temperature for the samples to estimate the optimized oil yield%. The catalyst was synthesized by sol gel method in nano-scale. It was further characterized by FTIR spectroscopy, SEM, and XRD. The algal oil was pre-treated and trans-esterified with a catalyst to produce alkyl esters. The optimized process variables were determined using response surface methodology by varying parameters such as methanol to oil ratio and catalyst weight% for algal bio-oil and MeOH to oil ratio, time, and catalyst weight% for seed oil. The GC–MS was done to characterize the presence of biodiesel. Kinetic studies were done for the optimized condition for the algal oil and seed oil and it follows the pseudo-first order reaction.

### **Production of biosurfactants from agro-industrial waste and waste cooking oil in a circular bioeconomy: An overview**

Waste generation is becoming a global concern owing to its adverse effects on environment and human health. The utilization of waste as a feedstock for production of value-added products has opened new avenues contributing to environmental sustainability. Microorganisms have been employed for production of biosurfactants as secondary metabolites by utilizing waste streams. Utilization of waste as a substrate significantly reduces the cost of overall process. Biosurfactant(s) derived from these processes can be utilized in environmental and different industrial sectors. **Vivek K. Gaur *et al*** focused on global market

of biosurfactants followed by discussion on production of biosurfactants from waste streams such as agro-industrial waste and waste cooking oil [**Bioresource Technology**, **343**, Article 126059, (2022)]. The need for waste stream derived circular bioeconomy and scale up of biosurfactant production have been narrated with applications of biosurfactants in environment and industrial sectors. Road blocks and future directions for research have also been discussed.

### **Evaluation of trans fatty acids, carbonyl compounds and bioactive minor components in commercial linseed oils**

*Trans* fatty acids (TFAs), associated with the risks of coronary heart disease and diabetes, are formed by isomerization of unsaturated fatty acids during refining of linseed oils. In this study, TFAs and the chemical characteristics (acid value, peroxide value, carbonyl compounds, bioactive minor components and fatty acids) in 32 commercial linseed oils were investigated by **Zihan Xu *et al*** and the correlation among them were further analyzed [**Food Chemistry**, **369**, Article 130930, (2022)]. Results showed that C18:3 TFAs were predominant TFAs in linseed oils and about 9% of the samples had TFA contents above 2 g/100 g fat, as well as the average level of TFA in the refined samples was higher than that in the unrefined oils. The correlation analyses suggested C18:3 TFAs exhibited significant negative correlations with acid value, levels of acetone, *trans*-2-nonenal, campesterol and  $\alpha$ -linolenic acid. These results provided a comprehensive insight of TFAs in linseed oil and had important implications for consumers and linseed oil industry.

### **The physicochemical properties of five vegetable oils exposed at high temperature for a short-time-interval**

**Laura Mitrea *et al*** evaluated the physicochemical characteristics of five vegetable oils exposed to high temperatures for a short time interval. Sunflower, rapeseed, maize, palm, and coconut oils, which are largely used for food thermal preparation, were heated for 30 min at 180 °C and analyzed before and after



heating in terms of viscosity, Acid value, Peroxide value, density, and fatty acids profile [**Journal of Food Composition and Analysis**, **106**, Article 104305, (2022)]. The rheological measurements showed raised values for the viscosity, shear rates, and densities after the heat exposure for all investigated samples. Moreover, thermo-oxidative modifications considering the Acid value and Peroxide content showed increased values above the recommended limits (0.6 mg KOH/g oil and 10 mEqO<sub>2</sub>/kg oil) for the investigated oils, excepting the rapeseed oil which was within the limits of the Acid values (0.26 mg KOH/g rapeseed oil). The percentage of the unsaturated fatty acids was influenced by the heat exposure in all samples. The formation of *trans* fatty acids was visible in all samples (by FTIR at 962 cm<sup>-1</sup>) excepting the coconut oil. In conclusion, short-time exposure at high temperatures can impact negatively the physicochemical properties of sunflower, rapeseed, maize, palm, and coconut oils.

### **The application of machine-learning and Raman spectroscopy for the rapid detection of edible oils type and adulteration**

Raman spectroscopy is an emerging technique for the rapid detection of oil qualities. But the spectral analysis is time-consuming and low-throughput, which has limited the broad adoption. To address this issue, nine supervised machine learning (ML) algorithms were integrated by **Hefei Zhao *et al*** into a Raman spectroscopy protocol for achieving the rapid analysis [**Food Chemistry**, **373**, Part B, Article 131471, (2022)]. Raman spectra were obtained for ten commercial edible oils from a variety of brands and the resulting spectral dataset was analyzed with supervised ML algorithms and compared against a principal component analysis (PCA) model. A ML-derived model obtained an accuracy of 96.7% in detecting oil type and an adulteration prediction of 0.984 (*R*<sup>2</sup>). Several ML algorithms also were superior than PCA in classifying edible oils based on fatty acid compositions by gas chromatography, with a faster readout and 100% accuracy. This study provided an exemplar for combining conventional Raman spectroscopy or gas chromatography with ML for the rapid food analysis.

### **Optimization and validation of a simplified methodology for simultaneous extraction of fatty acids and tocopherol homologues in peanuts**

A new sample treatment methodology was developed by **Adriana Juan-Polo *et al*** for the simultaneous determination of oleic, linoleic, palmitic and stearic fatty acids (FA), and  $\alpha$ -,  $\beta$ -,  $\gamma$ - and  $\Delta$ -tocopherol homologues in peanut samples [**Journal of Food Composition and Analysis**, **106**, Article 104287, (2022)]. Usually, the determination of these compounds is carried out after oil extraction with an organic solvent at boiling temperatures of the solvent. The innovative analytical methodology developed in this work was performed in a 20 mL vial in which the FA and tocopherols were simultaneously extracted, and the derivatization of the FA was, afterwards, conducted. The reduction in analysis time from 1 h 30 min (reference methodology) to 20 min (new methodology) increased the sample throughput. Furthermore, the amount of organic solvent used decreased from 40 mL to 6 mL. The new methodology was validated based on the analysis of a certified reference peanut butter sample and the results were compared to those obtained by using the reference methodology and a suitable agreement with the certified values was found. Finally, the new sample treatment approach was used to analyse toasted and fried (with and without tegument) peanuts.

### **Characterization and application of novel composite films based on soy protein isolate and sunflower oil produced using freeze drying method**

Biocomposite films based on soy protein isolate (SPI) and sunflower oil (SO) were fabricated using freeze drying (FDM) as an innovative approach to formulate a fairly easy-to-apply way, moreover, results were compared with the classic film production method (CM). In FDM, SPI edible film solutions were prepared and dried using freeze drying, and then reconstituted to produce the films. The aim by **Burcu Gökkaya Erdem and Sevim Kaya** was to specify the effect of both using FDM and concentration of SO (0.05%,

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0.10% and 0.15% (w/v)) on the characterization of SPI films via thermal, barrier and morphological analyses [**Food Chemistry**, **366**, Article 130709, (2022)]. Reinforced mechanical and good barrier properties were achieved with FDM. By increasing SO content, an improvement of hydrophobic property of the films, a decrease in the swelling values, and a reduction in permeability was observed. The cakes which were wrapped with FDM films showed better textural results than either uncoated cake or the cakes wrapped with CM films.

### **Microalgae biomass as a sustainable source for biofuel, biochemical and biobased value-added products: An integrated biorefinery concept**

Microalgal biomass has been proved to be a sustainable source for biofuels including bio-oil, biodiesel, bioethanol, biomethane, etc. One of the collateral benefits of integrating the use of microalgal technologies in the industry is microalgae's ability to capture carbon dioxide during the application and biomass production process and consequently reducing carbon dioxide emissions. Although microalgae are a feasible source of biofuel, industrial microalgae applications face energy and cost challenges. To overcome these challenges, researchers have been interested in applying the bio-refinery approach to extract the important components encapsulated in microalgae. This review by **Sk. Yasir Arafat Siddiki et al** discusses the key steps of microalgae-based biorefinery including cultivation and harvesting, cell disruption, biofuel and value-added compound extraction along with the detailed technologies associated with each step of biorefinery [**Fuel**, **307** Article 121782, (2022)]. This review found that suitable microalgae species are selected based on their carbohydrate, lipid and protein contents and selecting the suitable species are crucial for high-quality biofuel and value-added products production. Microalgae species contain carbohydrates, proteins and lipids in the range of 8% to 69.7%, 5% to 74% and 7% to 65% respectively which proved their ability to be used as a source of value-added commodities in multiple industries including agriculture, animal husbandry,

medicine, culinary, and cosmetics. This review suggests that lipid and value-added products from microalgae can be made more economically viable by integrating upstream and downstream processes. Therefore, a systematically integrated genome sequencing and process-scale engineering approach for improving the extraction of lipids and co-products is critical in the development of future microalgal biorefineries.

### **Continuous biodiesel production: A review of advances in catalysis, microfluidic and cavitation reactors**

Biodiesel production can be achieved through different processes and different reactor types as well as varying reaction parameters such as catalyst dose, oil: alcohol ratio, reaction time, mixing intensity, free fatty acid content (<3% required) and reaction temperature. The present review by **Jude A. Okolie et al** presents the advances in catalytic biodiesel production with emphasis on the current challenges, and prospects in homogeneous and heterogeneous catalysts [**Fuel**, **307**, Article 121821, (2022)]. Particularly, alkaline earth metal, metal mixed oxides, carbon-based and biomass-derived catalysts, and ionic liquids are discussed in detail. In addition, microfluidic, and acoustic reactors such as ultrasonic and hydrodynamic cavitation reactors are reviewed. The results from conducted studies revealed calcium oxide (CaO)-based catalysts derived from either residual biomass sources or synthetic chemicals are more favorable for biodiesel synthesis. Hydrotalcite catalysts and ionic liquids require harsh conditions such as higher temperature, higher oil: methanol ratio, and longer reaction time to achieve appreciable yield. The micro-reactors and cavitation reactors provide a faster reaction rate compared to conventional reactors because there is a drastic reduction in the time it takes for miscibility and diffusion of the reactant molecules and hence, eliminates the high energy requirement for mixing. The hydrodynamics reactor presents lower erosion challenges and energy input per unit of reactants when compared with the ultrasonic reactor. It is hoped that this study will provide detailed and recent knowledge

on heterogeneous catalysts, microfluidic, and cavitation reactors for biodiesel synthesis.

### Catalytic methanotreating of vegetable oil: A pathway to Second-generation biodiesel

Vegetable oil is one of the most commonly used feedstocks for the production of biodiesel, while the first-generation biodiesel suffers from the disadvantages of considerable instability and corrosivity. Developing second-generation biodiesel is momentous for the sustainable development of global energy, which overcomes the shortcomings of first-generation biodiesel. The methano treating of vegetable oil is a potential new route for the production of second-generation biodiesel, which is comprehensively investigated in this study by **Yimeng Li *et al.*** Throughout the screening of the catalysts, Ga-Ce/TS-1 demonstrates the best overall performances in this methane-incorporated process, leading to 84.23 % of liquid yield, 0.95 % of methane conversion, 72.8 % of oxygen content reduction, and 71% of light hydrocarbon distillates yield [**Fuel**, **311**, Article 122504, (2022)]. The participation of methane promotes deoxygenation performance, optimizes the composition of paraffinic and olefinic hydrocarbons, as well as suppresses coke formation. The catalytic methanotreating of vegetable oil is confirmed to be a promising pathway to second-generation biodiesel.

### Isochoric heat capacity, phase transition and derived key thermodynamic properties of methyl decanoate

**Ilmutdin M. Abdulagatov *et al*** studied single – and two–phase isochoric heat capacity (CV) and liquids-gas phase transition temperature (TS) and density ( $\rho$ ) of methyl decanoate as component of biodiesel derived from coconut oil or babassu oil [**Fuel**, **310**, Part B Article 122251, (2022)]. The 15 liquid and vapor isochores between (152 and 834)  $\text{kg}\cdot\text{m}^{-3}$  at temperatures from (300 to 463) K and at pressures up to 16 MPa were determined using high-temperature and high-pressure nearly constant-volume adiabatic calorimeter. For each experimental isochore the measurements were conducted in the two-phase region

in the immediate vicinity of the phase-transition temperatures (TS) to precisely determine the phase boundary density, one-phase and two-phase isochoric heat capacities ( $\rho$ , CV1, and CV2) using isochoric heat -capacity discontinuity behavior technique. For ten liquid isochores, the TS was experimentally determined using CV discontinuity method (irregular behaviour of CV). For vapor (152.09 and 204.55)  $\text{kg}\cdot\text{m}^{-3}$  and near-critical (235.13 and 300.83)  $\text{kg}\cdot\text{m}^{-3}$  isochores we have never reached isochoric heat-capacity discontinuity temperatures TS due to thermal decomposition of the methyl decanoate at high temperatures (above 473 K). The measured CV2 as a function of specific volume (V) along the various isotherms (below 473 K) were used to accurately estimate the values of the second temperature derivatives of chemical potential and vapour-pressure using theoretically based Yang-Yang relation. The contributions of the vapour-pressure and the chemical potential to the measured total two-phase CV2 of methyl decanoate were estimated. The measured CV2  $\rho$ , and TS were used for determination many key thermodynamic properties of methyl decanoate along the saturation curve as well as the critical property data ( $T_C=669.5 \pm 5$  K and  $\rho_C = 287 \pm 5$   $\text{kg}\cdot\text{m}^{-3}$ ).

### Transesterification of palm kernel oil with ethanol catalyzed by a combination of immobilized lipases with different specificities in continuous two-stage packed-bed reactor

Technically and economically viable production processes are frequently reported, but products often fail to meet EN 14214 specifications. **Rodney H. Miotti Jr *et al*** aimed to investigate the transesterification of palm kernel oil catalyzed by a combination of lipases from different sources and produce biodiesel in accordance with current quality standards [**Fuel**, **310**, Part A Article 122343, (2022)]. Transesterification reactions were first carried out in a stirred batch reactor at 45 °C using an oil:alcohol molar ratio of 1:8. In batch runs, a 50:50 mixture of *Burkholderia cepacia* lipase and *Thermomyces lanuginosus* lipase immobilized onto a silica hydroxyethylcellulose matrix resulted in the highest

fatty acid ethyl ester yield (98%) and a decrease in kinematic viscosity from 30.35 to 4.00 mm<sup>2</sup> s<sup>-1</sup>. The product had undetectable levels of DAG and low content of MAG (1.68 wt%). The enzymes were then packed separately in a continuous two-stage reactor operating at different space times (16 and 14 h) and oil/alcohol molar ratios (1:12, 1:10, and 1:8). The highest volumetric productivity (415.3 ìmol g<sub>cat</sub><sup>-1</sup> h<sup>-1</sup>) was achieved using an oil/alcohol molar ratio of 1:8 and space-time of 16 h. The biodiesel had an alkyl ester content above 98%, a MAG content of less than 0.7 wt%, undetectable DAG content, and kinematic viscosity of 4.22 ± 0.25 mm<sup>2</sup> s<sup>-1</sup>, thereby complying with EN 14214.

**Use of biomass-derived glycerol as an alternative to fossil fuels for aniline production: Energy saving and environmental aspects [Fuel, 310, Part A, Article 122359, (2022)]**

**Mohammad Hasan Khademi and Mohammad Lotfi-Varnoosfaderani**

Catalytic reduction of nitrobenzene is the leading technological step in aniline production. The hydrogen required for this stage, is dominantly produced from fossil fuels through reforming processes, which take much energy and emit large amounts of CO<sub>2</sub>. Biomass-derived glycerol steam reforming is an attractive alternative to traditional reforming to reduce the dependence on hydrocarbon resources and mitigate climate change. This research aims to analyze a mass- and heat-integrated multi-tubular membrane reactor, containing nitrobenzene hydrogenation (exothermic-side) and glycerol steam reforming (endothermic-side) for co-production of aniline and syngas. In this process, hydrogenation reaction acts as a heat source for glycerol reforming, while hydrogen produced in the endothermic-side simultaneously permeates through the membrane, reacts with nitrobenzene to produce aniline and enhances the equilibrium glycerol conversion. Besides, the steam produced in the exothermic-side is continuously recycled to the entrance of the endothermic-side. The role of different parameters on reactor performance is realized using a heterogeneous

model. Numerical results show that by adjusting the adequate operating conditions, glycerol and nitrobenzene conversion above 80% and syngas with H<sub>2</sub>/CO ratio in the range of 2.0–2.5, suitable for Fischer-Tropsch and methanol synthesis processes, can be achieved. In addition, this integrated process is promising in terms of energy saving, environmental pollution mitigation, feasibility and effectiveness for industrial-scale application; however, experimental proof-of-concept is required to ensure the safe operability of this process.

**Thermal cracking of oleic acid promoted by iron species from iron ore tailings for the production of ketones and fuels [Fuel, 310, Part A, Article 122290, (2022)]**

**Vivian A. Luciano *et al***

Iron ore tailings (IOT) are rich in iron oxides and silica and can be used for different applications such as allow the thermal cracking of fatty acids for the production of fuels and products with high added value. Thus, this work aimed to use of IOT as an iron source for the production of high-value products using oleic acid (OA) as carbon sources. These compounds were produced from a thermal decomposition reaction, from the mixture of OA and IOT, in a high pressure reactor (12.5 bar), using the ratio of 1:1 wt (acid:IOT), in temperatures between 250 and 450 °C (3 and 12 h). The results showed that for all reactions, the solid products obtained showed a percentage of less than 6 % of carbon material and different iron phases (Fe<sub>2</sub>O<sub>3</sub>, Fe<sub>3</sub>O<sub>4</sub> and FeOOH). For the reactions carried out at 250 and 350 °C/3h the main fraction obtained was the liquid, however the composition was mainly the starting compound and iron oleate. For the reactions carried out at 400 and 450 °C/3h, the main fraction was gas, mainly hydrogen. For the reaction carried out at 350 °C for 12 h the mass balance showed the formation of similar amounts of liquid and gaseous products. Liquid products formed ketones as the main product, while gaseous products were identified and presented greater selectivity for C3 hydrocarbons.



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### Upgraded methyl oleate to diesel-like hydrocarbons through selective hydrodeoxygenation over Mo-based catalyst [Fuel, 308, Article 122038, (2022)]

Cong Yu *et al*

As an effective substitute for petroleum diesel oil, biodiesel has received much more attention. Moreover, the diesel-like hydrocarbons produced by hydrodeoxygenation can further reduce the oxygen content and have a higher calorific value. Herein, we synthesize MoO<sub>2</sub>/Mo<sub>2</sub>C catalyst and use it for the selective hydrodeoxygenation of methyl oleate to diesel-like hydrocarbons. In this study, the reaction is mainly proceeded through the hydrodeoxygenation route (HDO/DCO is 30.2), the ratio of C<sub>18</sub> hydrocarbons in the products is 96.8%. The higher heating value (HHV) of the liquid products reached 46.09 MJ/kg. Meanwhile, the products contain 41.9% unsaturated hydrocarbons, which is attributed to the selective adsorption of carbon–oxygen bonds by oxygen vacancy in MoO<sub>2</sub>. X-ray photoelectron spectroscopy (XPS) and electron spin resonance spectroscopy (EPR) characterization demonstrate the existence of oxygen vacancies in the catalyst. This system can achieve deep deoxygenation while reducing hydrogen consumption and carbon loss.

### Aqueous phase reforming of biodiesel by product glycerol over mesoporous Ni-Cu/CeO<sub>2</sub> for renewable hydrogen production [Fuel, 308, Article 122014, (2022)]

Kai Wu *et al*

Aqueous phase reforming (APR), regarded as one method for inexpensive H<sub>2</sub> production, was widely studied due to lower temperature and unnecessary to vaporize water and fuels. In this study, the mesoporous CeO<sub>2</sub>, regarded as a metal oxide carrier, was first prepared by colloidal solution combustion, and then the Ni-Cu bimetallic supported on mesoporous CeO<sub>2</sub> was successfully synthesized to investigate the catalytic performance in APR of biodiesel byproduct glycerol. The results show that the Cu element in the catalysts can effectively improve water–gas shift (WGS) reaction

and inhibit the formation of methane, which increases the H<sub>2</sub> production rate from 125.08 to 195.57 μmol·min<sup>-1</sup>·g cat<sup>-1</sup>. The higher reaction temperature is beneficial to the H<sub>2</sub> production rate, but not to H<sub>2</sub> selectivity. Kinetics analysis indicates that catalyst of 1Ni2Cu/CeO<sub>2</sub> + 0.2gCaO has the lowest apparent activation energy (29.86 kJ·mol<sup>-1</sup>). CaO is introduced as an absorbent to improve WGS reaction and reduce methanation reactions through in-situ CO<sub>2</sub> removal and capture. The reaction path and mechanism of APR of glycerol are also discussed in this paper.

### Highly active iron-promoted hexagonal mesoporous silica (HMS) for deoxygenation of triglycerides to green hydrocarbon-like biofuel

The interest in the production of sustainable hydrocarbon-like biofuel is increasing due to the environmental issues such as global warming and uncertainty of crude oil prices. A series of Fe catalysts (5, 10, 40, and 100 wt% Fe) supported on the hexagonal mesoporous silica (HMS) were successfully prepared by Suraya Zulkepli *et al* for the production of biofuel via deoxygenation (DO) reaction [Fuel, 308, Article 121860, (2022)]. In the DO reaction, the diesel range biofuel is produced in the absence of H<sub>2</sub> and solvents. Interestingly, the 40 wt.% Fe/HMS catalyst exhibited the highest conversion and selectivity of 81.4 and 84.8%, respectively toward C<sub>8</sub>-C<sub>20</sub> hydrocarbons-like biofuel at 380 °C for 2 h. This performance is closely related to the well-dispersed Fe in the hematite phase, along with the interaction between Si-OH and Fe-O bonds. The acidity, surface area, and pore size of Fe/HMS have improved the catalytic activity. This result demonstrates that the Fe/HMS catalyst is a potential DO catalyst for the production of renewable hydrocarbon-like biofuel.

### Single-phase determination of calcium and magnesium in biodiesel using smartphone-based digital images

A microanalytical procedure using smartphone-based digital image colorimetry is proposed by Samara Soares *et al* for determining calcium and magnesium concentrations in biodiesel without analyte extraction

[**Fuel**, **307**, Article 121837, (2022)]. The analytical method relies on the discoloration of an alkaline Eriochrome Black T (EBT) solution because of complex formation with the analytes. Ethanol was used as a mediator solvent for dissolving both biodiesel and EBT. The analytical response was based on the measurement of reflected radiation from digital images captured using a smartphone camera. Photometric measurements were based on the RGB color system, taken R channel values as the analytical signal because of complementarity with the color of the EBT solution. The coefficient of variation ( $n = 10$ ) and limit of detection were 1.0% and  $3 \mu\text{mol L}^{-1}$ , respectively. A linear response was observed in the  $10\text{--}75 \mu\text{mol L}^{-1}$  range, described by the equation  $R = 0.612C + 93.1$  ( $r = 0.999$ ). The reagent amounts consumed per determination were as little as  $25 \mu\text{g}$  of EBT and  $120 \mu\text{g}$  of NaOH, with generation of only  $935 \mu\text{L}$  of waste. The procedure was selective for calcium and magnesium, without interference of metal ions ( $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Zn}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Fe}^{2+}$ , and  $\text{Fe}^{3+}$ ) as well as other sample components (glycerol, methanol, and water) in concentrations higher than those expected in biodiesel. Matrix effects for biodiesel obtained from different raw materials were negligible (recoveries from 90 to 104%), making feasible external standard calibration with aqueous/ethanolic reference solutions. The results agreed with those obtained by ICP OES after sample preparation by emulsion breaking, demonstrating the reliability of the proposed approach.

### **A novel *in situ* transesterification of yellow horn seeds to biodiesel using a dual function switchable solvent**

A novel and cyclic *in situ* transesterification of yellow horn (*Xanthoceras sorbifolia* Bunge.) seeds to biodiesel using the switchable solvent, *N, N*-dimethylcyclohexylamine (DMCHA) as both a reusable solvent and catalyst was proposed by **Su Zhang *et al.*** An optimizing study for the reaction conditions has been performed through response surface methodology [**Fuel**, **312**, Article 122974, (2022)]. The maximum yield of fatty acid methyl esters (FAMES) achieved

96.07% under the best conditions of temperature  $63.93^\circ\text{C}$ , 28.05 min, methanol amount of  $10.41 \text{ mL/g}$  (v/w of material) and DMCHA amount of  $16.42 \text{ mL/g}$ . Notably, The results of mechanism study combined with molecular dynamics and quantum chemistry theory showed that DMCHA is compatible with the triglycerides, and the effect of the hydrogen bonds, interaction energy, and radial distribution function on DMCHA performance were investigated for the first time. This new configuration of using switchable solvent for simultaneous extraction and reaction would be an appropriate, efficient and environment-friendly approach for simplify biodiesel production.

### **Green synthesis of glycerol carbonate via transesterification of glycerol using mechanochemically prepared sodium aluminate catalysts**

Glycerol, an important by-product from biodiesel production, has significant potential as platform chemical with several different pathways of value addition being investigated. Herein, **John Keogh *et al.*** reported the synthesis of glycerol carbonate via the transesterification of glycerol with dimethyl carbonate using supported sodium aluminate catalysts [**Fuel**, **310**, Part C, Article 122484, (2022)]. The effects of catalyst preparation method, and different metal oxide supports were investigated. Mechanochemically prepared catalysts were more stable than the corresponding catalysts prepared using a wet impregnation technique. Alumina as a support was found to provide the highest level of stability, amongst the metal oxide supports tested. The mechanochemically prepared catalyst with a sodium aluminate loading of 20 wt% supported on alumina, was found to be the most effective catalyst achieving 96 % yield of glycerol carbonate after 60 min. The catalysts were thoroughly characterized to investigate the effect of the catalyst synthesis method on the catalyst structure, catalytic activity and stability. Mechanochemically prepared 20 wt% sodium aluminate supported on alumina showed facile recovery and good reusability over four times with only a marginal decrease in activity.

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Charnley AK (1992). Mechanisms of fungal pathogenesis in insects with particular reference to locusts. In: Lomer CJ, Prior C (eds) *Biological Controls of Locusts and Grasshoppers: Proceedings of an international workshop held at Cotonou, Benin*. Oxford: CAB International, pp 181-190.

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