K. Subramanian/J. Pharm. Sci. & Res. Vol. 11(9), 2019, 3201-3209

ISSN:0975-1459
Journal of Pharmaceutical Sciences and Research
www.jpsr.pharmainfo.in

A Comprehensive Study on Thermal Degdradation of Selective Edible Vegetable Oils By Simultaneous Thermogravimetric and Differential Thermal Analyses

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Abstract

The oxidative degradability of the unsaturated fat components in edible vegetable oils affect their nutrient value, storage stability, reusability and high temperature culinary applications. In the present investigation thermal and thermooxidative stabilities and decomposition enthalpies of refined and virgin oils of gingely, ground nut, coconut and sunflower in air and N_2 were simultaneously analysed by nonisothermal gravimetry and differential thermal analysis to optimize their process, use and storage conditions for stability and reusability. These oils which registered a negligible mass loss at $\leq 200^oC$ degraded exothermically in air by three step pathway and endothermically in N_2 by a single step. Their thermal stabilities decreased in the order groundnut>gingely> sunflower>coconut and sunflower>groundnut> gingely>coconut in N_2, and groundnut> gingely> coconut>sunflower and gingely>groundnut> coconut> sunflower in air for refined and virgin grades respectively. Their level of saturated and unsaturated fats were also analysed from the recorded thermograms.

Key words: Thermogravimetry; differential thermal analysis; thermooxidative stability; vegetable oils; enthalpy; saturated and unsaturated fats; medium chain fatty acids

INTRODUCTION

Edible Vegetable oils extracted from plant oil seeds, nuts, rice bran etc. are categorized as simple lipids. They contain triacylglycerol (esters of glycerol with long chain C_{12} to C_{18} saturated and unsaturated fatty acids) as their major component along with polyphenols, aldehydes, sterols , antioxidants, vitamins, a variety of volatile compounds and others as minor components. Edible oils are physiologically vital constituents of human diet and life (Eckey 1954; Alfred 2002; Kumar et al.2016; Frank 2011; Orsavova et al. 2015; Manchanda and Jain 2016; Leong et al. 2015) . They provide energy, essential fatty acids necessary to raise high density lipoprotein and serve as a carrier of fat soluble vitamins and antioxidants. They impart taste and platability to food . They constitute 15–20% of total caloric intake by industrialized nations. They protect the organs and body mechanically and thermally and participate in metabolism. Their metabolism generates many bioactive lipid molecules that are fundamental mediators of multiple signaling pathways. Any change in lipid metabolism can result in modification of membrane composition and its permeability, and some pathological states, such as cancer, cardiovascular, neurodegenerative, and metabolic diseases. Dietary factors of the edible oils, play an important role in the causation, treatment, management, and prevention of Coronary heart disease and its incidence is rising rapidly, especially in developing countries, Therefore, right selection of edible oil is extremely important.

The vegetable oil industrialization is one of the important activities of agribusiness . The annual global production of edible oils and fats is around 117 million tonnes and 80% of it is used for food. Consumers prefer vegetable oils over animal fat as they are rich in unsaturated fats and do not contain cholesterol. They find wider uses in pharmaceutical, industrial, food and cosmetic products. These include products such as cooking oils, margarine, salad dressings, coatings, paints, plasticizers, lubricants, hydraulic fluids, thermal fluids in solar cookers, glycerol, synthetic fibres, lecithin, printing inks, medicines, face masks, hand creams, shower gels, soaps, detergents, biofuel and many more too numerous to mention here. They are increasingly used as biolubricants and biodiesel due to their ecofriendly and renewable nature (dos Santos et al.2015; Heikal et al. 2017; Jayadas and Prabhakaran Nair 2006; Li et al. 2015; Ullah et al. 2014; Zhang et al. 2011; Lai et al. 2014 . Vegetable-oil-based biolubricants (dos Santos et al. 2015; Heikal et al. 2017; Jayadas and Prabhakaran Nair 2006) are non-toxic and biodegradable and have a better lubricating performance compared to mineral-oil. These applications and features have increased the race for vegetable oils causing a rapid and significant expansion both in domestic and international market.

The vegetable oils are most liable to oxidative degradation due to their unsaturated fatty acid component leading to their poor oxidative stability in air during their technological processing, storage and use (Takeoka et al. 1997; Warner 2002; Choe and Min 2007; Tyagi and Vasishtha 1996; Rossell 2001; Crnjar and Witchwoot 1981). Free fatty acids, mono- and diacylglycerols, trace metals, chlorophylls, carotenoids, tocopherols, phospholipids, temperature, light, oxygen, oil processing methods, enzymes and fatty acid composition affect their oxidative stability during storage and processing. The oxidation of edible oil produces off-flavor compounds auch as aldehydes, ketones, epoxides, hydroperoxides, hydroxy compounds, etc leading to rancidity and decreased oil quality. Some of these degraded products are potentially carcinogenic, mutagenic and antherogenic and are highly harmful to the heart. To minimize the oxidative degradation of edible oil during processing and storage, it is recommended to use appropriate concentrations of antioxidants such as tocopherols and phenolic compounds (Choe and Min 2006. Since oils are used as the medium

for frying a large number of food products, these oils must be oxidatively stable at the frying temperatures. Prolonged and repeated use of used oil for deep-oil-frying (180^{o}C) in moisture change its physical and/or chemical properties (like shelf life, nutrient value, texture etc) due to various complex reactions like oxidation, free radical formation, hydrolysis, isomerization to trans fatty acids, polymerization etc (Takeoka et al. 1997; Warner 2002; Choe and Min 2006 and 2007; Tyagi and Vasishtha 1996; Rossell 2001; Crnjar and Witchwoot 1981). Prolonged heating also reduces organoleptic and nutritive quality of oils.

Oxidative stress on oils can cause conjugated double bond formation as well as evoluation of trans fatty acids and hazardous components which will result in undesirable influence on nutritional quality, safety and sensory properties (Takeoka et al.1997; Warner 2002; Choe and Min 2006 and 2007; Tyagi and Vasishtha 1996; Rossell 2001; Crnjar and Witchwoot 1981). Hence in view of the aforementioned importance of edible oils, it becomes indispensable to analyse the thermal and thermo-oxidative stability of oils for their processing, storage and use. Thermoanalytical tools such as thermogravimetry(TG), differential thermal analysis(DTA), differential scanning calorimetry (Orsavova et al. 2015; Aluyor et al. 2009; Santos and Souza 2007; Gouveia et al. 2004; Dweck and Sampaio 2004 etc are increasingly employed to analyse the quality of edible oils during storage and use in cooking since these methods are highly precise, sensitive, give results rapidly and require a small sample mass. The present investigation involves the study of thermal and thermoxidative stabilities and decomposition enthalphies of refined and virgin grades of ground nut (Peanut), gingley (seasome), sunflower and coconut oils by simultaneous TG and DTAs in pure nitrogen and air . These methods rapidly furnish stability data which are important for commercial production, storage, use and quality control of these edible oils..

EXPERIMENTAL

Materials and Methods

Materials

Commercially available refined and virgin edible oils viz sunflower(SNFL), coconut(COCNT), gingely(GNGLY) and ground nut(GRDNT) were purchased from the local market. All these vegetable oils are reported to have glyceryl esters of saturated fatty acids (SAF) like lauric, stearic, palmitic acid etc , monounsaturated fatty acid (MUF) such as oleic , palmit oleic etc and poly unsaturated fatty acid(PUFA) like linoleic, linolenic etc (Gouveia et al. 2004; Dweck and Sampaio 2004; Muneeshwari et al. 2017; Mathias 2003; Edward and Sahidi 2006; David 2006).

Methods

Simultaneous thermogravimetry(TG)-differential thermal analysis(DTA)

Simultaneous TG/derivative thermogravimetry (DTG) and DTA were performed in air and nitrogen in the temperature range 27-650^{0}C at a heating rate of 10^{o}C per min on NETZSCH STA 2500 Regulus (GERMAN) for a sample size 3-9 mg using alumina crucibles as sample holder and reference. The thermograms were analysed and overlayed using the proteous soft ware supplied with the equipment. The flow rate for nitrogen(99.999%) and air (N_2/O_2, 80/20) as purge gases was 60 ml/min. The flow rate of protective nitrogen gas was 40 ml/min. Temperature calibration was done using the pure metals In, Ag, Au, Cu, Al etc as standards. Enthalpy calibration was done using indium and calcium oxalate monohydrate as standard. The enthalpies in Jg^{-1} was calculated by measuring the endo or exothermic peak areas in DTA traces using the proteous software. The region enclosed between the peak and the interpolated baseline is taken as the area of the peak. The thermal stabilities of these oils were determined by the measuring their tempearures for onset degradation from the TG/DTG traces

RESULTS AND DISCUSSION

The TG, DTG and DTA traces for the refined SUNFL, COCNT, GRDNT and GNGLY oils in nitrogen and air are displayed in **Fig.1 (1A, 1B and 1C)** and **Fig.2(2A, 2B and 2C)** respectively. **Fig. 3(3A, 3B and 3C)** and **Fig.4(4A, 4B and 4C)** represent the TG, DTG and DTA thermograms of virgin oils in N_2 and air respectively.

TG/DTA analysis in nitrogen

Refined and virgin oils

Analysis of TG and DTG traces for refined (**Fig.1 (1A** and **1B)** in N_2 and those of virgin (**Fig.3 (3A** and **3B)**) oils indicated that the pure thermal stability of these oils decreased in the orders GRDNT>GNGLY> SUNF>COCNT and SNFL>GRDNT> GNGLY>COCNT for refined and virgin oils in N_2 respectively and degradations were endothermic(**Figs.1C** and **3C)** predominantly followed a single step . Even though COCNT oil started losing weight around 220^{o}C in N_2 , this temperature was around 300^{o}C in the other three oils(**Figs.1A** and **3A**). This indicated that COCNT oil may be more susceptible to thermal degradation near frying temperature than the other three oils. Since COCNT oil is reported to have >75% medium chain SAF (lauric(C_{12}), caprilic(C_6) , capric (C_{10}) etc) the above observation tend to imply that glyceryl esters of medium chain SAF may thermally decompose faster than long chain unsaturated fats (MUF & PUF). It is also reported that medium chain fatty acids in coconut oil are similar to human milk with nutraceutical benefits. Human body finds it easier to metabolize them and do not require energy for absorption and utilization (Srivastava et al., (2017); Raghavendra and Raghavarao, (2010); Hiroyuki et al.,(2008). Upto 200^{0}C the TG curves are overlapping and plateau for all the four oils implying no degradation. At 300^{O}C the percentage residues for these oils followed the order SNFL > GRDNT>GNGLY>COCNT for both refined and virgin (**Figs. 1A** and **3A**) grades. But the residual weight at 650^{o}C were found to be zero or negligible in all the oils and the percentage weight losses observed at 300^{0}C for virgin oils in nitrogen were found to be slightly higher than that observed for refined oils(**Figs. 1A** and **3A**) . These may be due to the degradative loss of nutrients, phytochemicals etc which

may be absent in the refined oils. In fact this also was reflected in the shape of the DTA curves of virgin SNFL and GRDNT (**Fig. 3C**) in N_2 which appeared to have a slight exothermic nature of degradation in nitrogen compared to those of refined oils (**Fig.1C**) which displayed an endothermic peak.

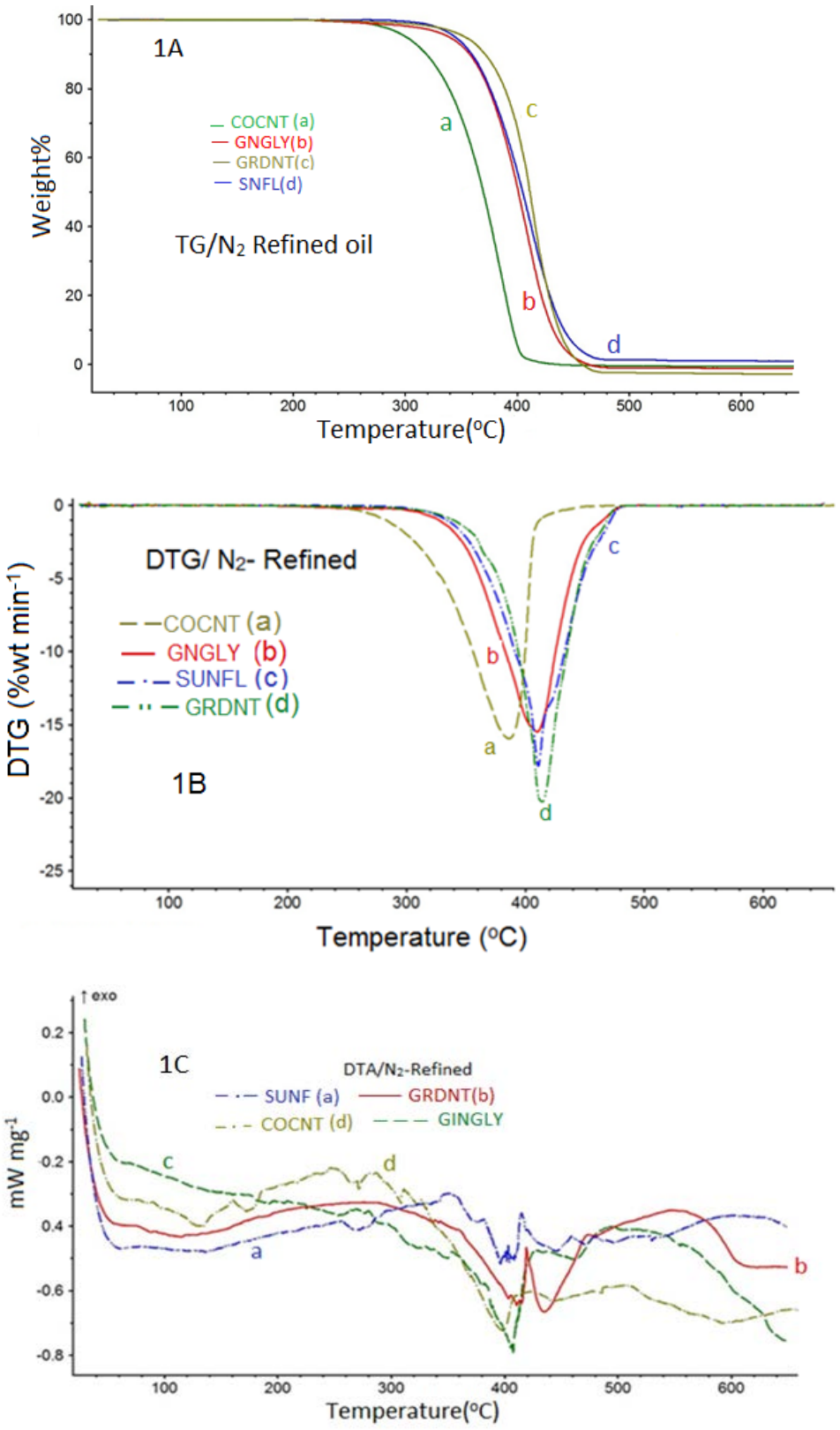

Fig.1 TG(1A), DTG(1B) and DTA(1C) thermograms of refined COCNT, GNGLY, GRDNT and SUNFL oils in N_2

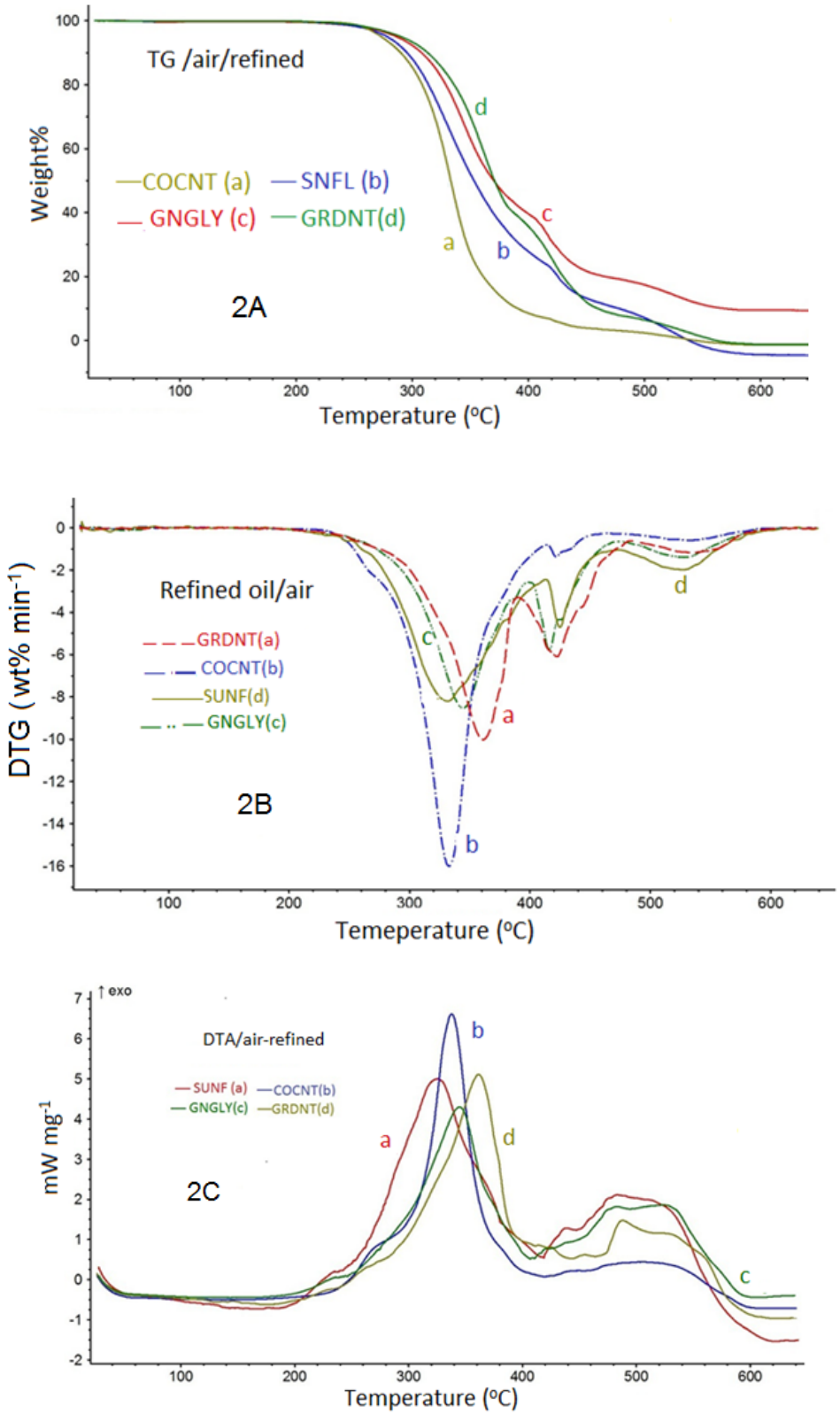

Fig. 2 TG(2A), DTG(2B) and DTA(2C) thermograms of refined COCNT, SUNFL, GNGLY and GRDNT oils in air

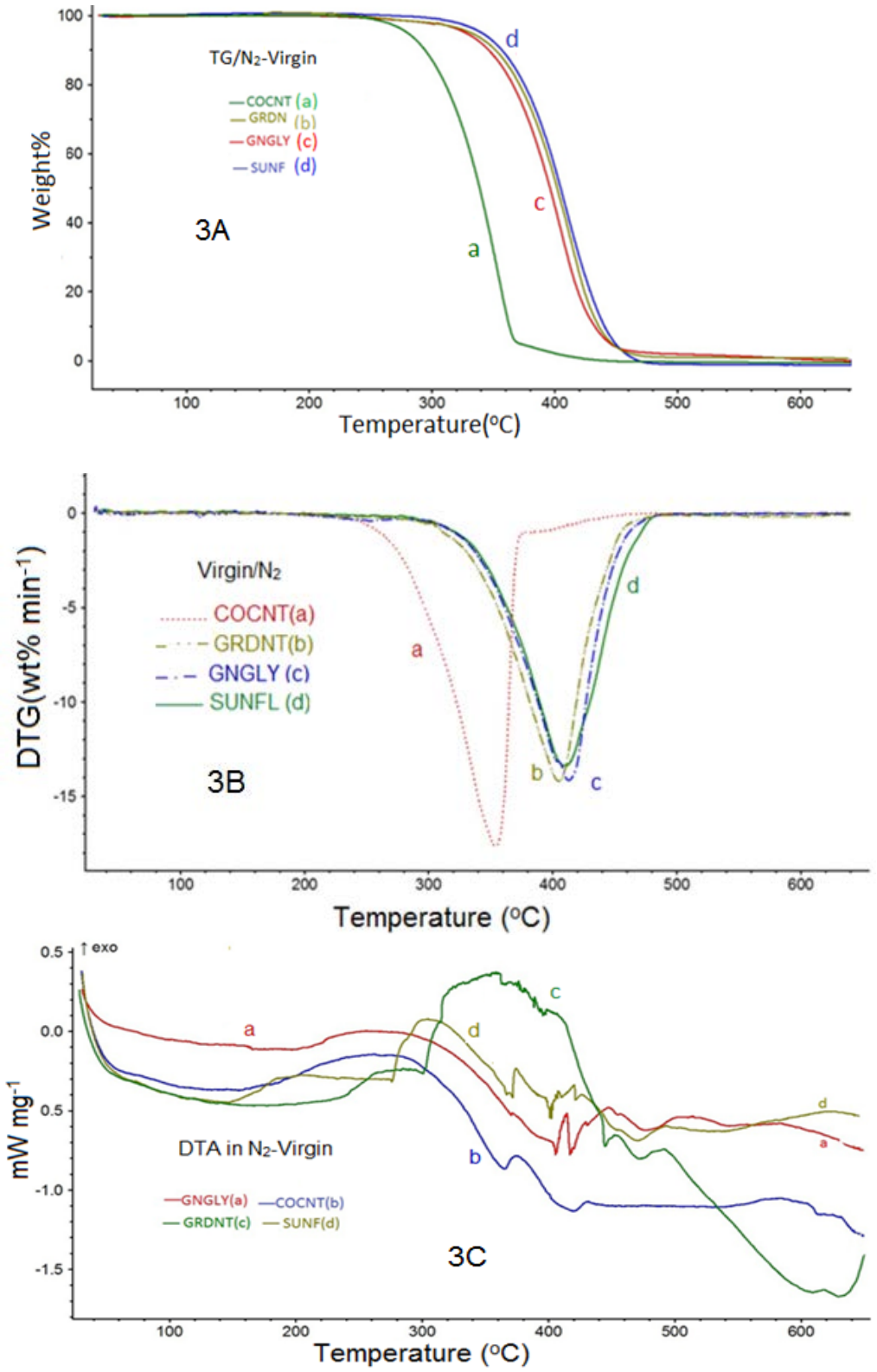

Fig. 3 TG(3A), DTG(3B) and DTA(3C) thermograms of virgin COCNT , GNGLY, GRDNT and SUNFL oils in N_2

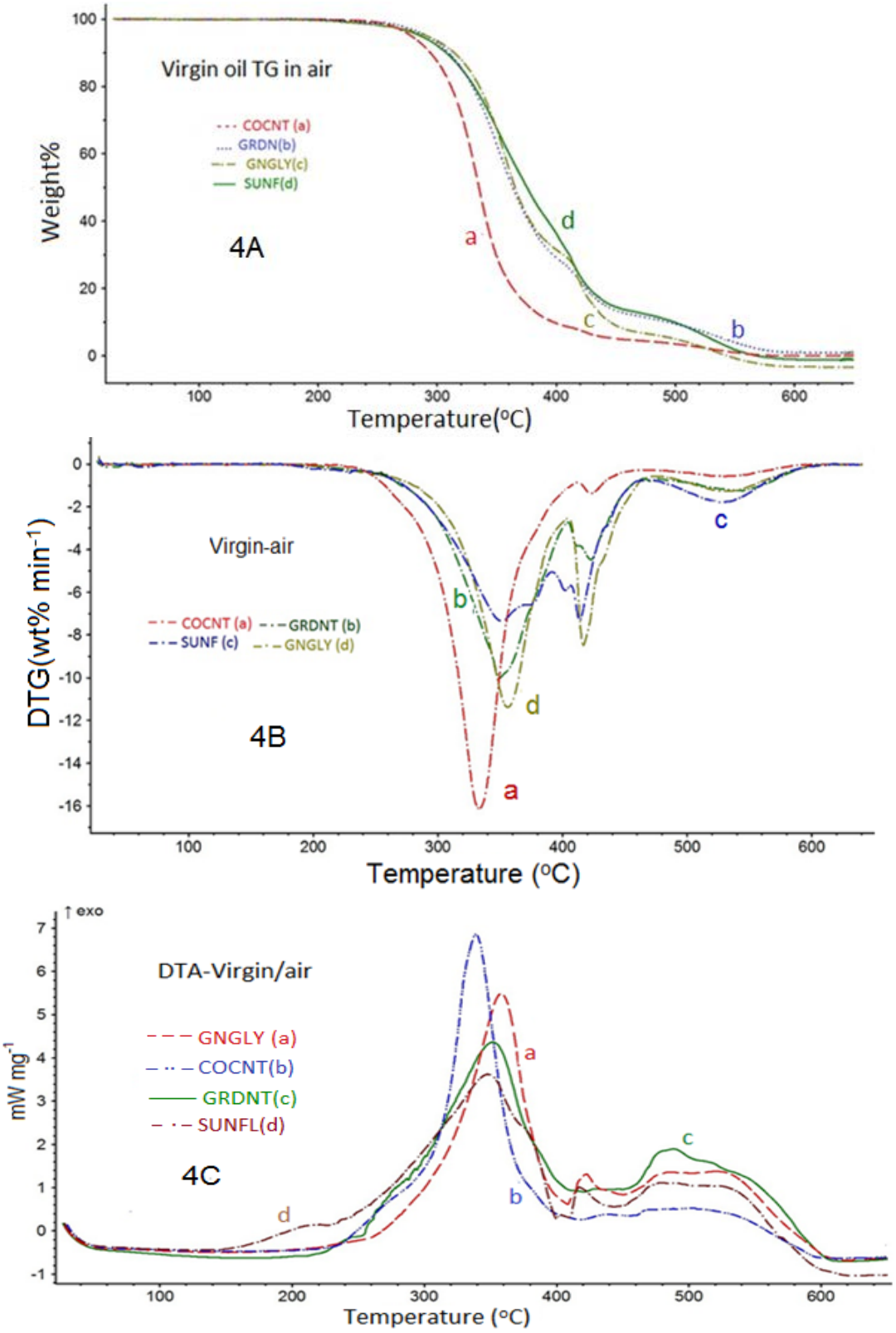

Fig. 4 TG(4A), DTG(4B) and DTA(4C) thermograms of virgin GRDNT, COCNT, GNGLY and SUNFL oils in air

Table 1 Proportion of SAF, MUF and PUF in SNFL,COCNT,GNGLY and GRDNT oils

Type of fat	SNFL			COCNT			GNGLY			GRDNT		
	REF	REF#	VIR	REF	REF#	VIR	REF	REF#	VIR	REF	REF#	VIR
SAF	11	11	17.6	93.17	92	75.27	21	14	14.9	10.63	13	15.42
MUF	22	20	14.4	3.83	6	18.56	34.28	43	26.1	31.57	25	24.45
PUF	67	69	68	2.82	2	6.17	43.69	43	58.39	57.8	62	60.13

REF# - Values of refined oil taken from references: Orsavoval et al. 2015 ; Benjamin 2003

Table 2 Enthalpy values from DTA traces for the degradation of GNGLY,COCNT, SNFL and GRDNT oils in air

Vegetable oil	Enthalpy(Jg^{-1}) from DTA for maximum weight loss temperature (^oC)			
	Refined oil		Virgin oils	
	Enthalpy	Weight loss temperature	Enthalpy	Weight loss temperature
	Nitrogen atmosphere			
GNGLY	-89.33	407	-147.6	406-417
COCNT	-51.68	397.4	-20.97	366
SUNF	-54.65	416.4	+108.3	334.5
GRDNT	-134.9	416	+433.1	358.5
	Pure air atmosphere			
GNGLY	1636 1023	348 524	1682 627	358 519
COCNT	2052 432	338 508	2026 463.4	338 506.7
SUNFL	2539 1510	325 483	1839 1002	347 479
GRDNT	1661 765.7	361.5 480-520	1820 766	351 488.5

Note: -ve sign: endothermic & +ve sign exothermic

TGA-DTA Analysis in air

Comparison of the TG/DTG thermogrames of the chosen oils in nitrogen (**Figs 1A, 1B, 3A** and **3B)** and air (Figs. 2A,2B,4A and 4B)indicated that their thermal stabilities based on onset degradation temperature for the refined and virgin oils decreased in air due to thermo oxidative degradation . COCNT oil which contains predominantly medium chain fatty acids displayed less thermal stability in air compared to other oils(**Figs. 2A, 2B, 4A** and **4B**). These thermograms in air also revealed negligible weight loss upto 200^{o}C for refined oils(**Figs.2A** and **2B**) and 190-195^{0}C for virgin oils(**Figs.4A** and **4B**). The vegetable oils under study were nearly completely decomposed in air at 600^{0}C except refined GNGLY oil which displayed a residue of 9.5%. This may be due to the additives (antioxidant, jaggery powder, seasome powder etc) added by the manufacturer. The thermal stabilities of these oils in air increased in the orders SUNF<COCNT<GRDNT<GNGLY and SUNF<COCNT< GNGLY<GRDNT for virgin and refined respectively . But based on the mass loss at 250^{o}C the thermal stabilities were increased in the order COCNT<SUNF < GNGLY<GRDNT and COCNT<SUNF < GRDNT<GNGLY for refined and virgin grade oils respectively.

The TG, DTG and DTA traces for the refined(**Fig.2**) and virgin (**Fig.4**) oils in air showed a three step exothermic oxidative degradation. The behavior of DTG plot(Figs 2B and 4B) with respect to TG plot (**Figs.2A and 4A**) delineates different stages of thermal degradation These were attributed to the different components of the oil namely, saturated(SAF), monounsaturated (MUF) and poly unsaturated (PUF) fats (Manchand and Jain 2016; Leong et al. 2015; Santos and Souza 2007; Dweck and Sampaio 2004; Srivastava 2017). For long chain fats the three tandems steps, were reported to occur in the temperature ranges 200–380^{o}C, 380–480^o C and 480–600 ^{0}C respectively. The first two steps of thermal decomposition are marked by decomposition of unsaturated fatty acids (UFA).

The oxidative decomposition of coconut oil which contains >75% medium chain saturated fats showed a weight loss before 340°C and its TG trace are steep (Figs 2A and 4A) associated with a high exothermic peak in DTA(Figs. 2C and 4C). These observations indicate that COCNT oil has better oxidative properties because of the saturated nature of its medium chain fatty acid constituents. The TG trace in the oxidative decomposition of SNFL, GNGLY and GRDNT oils which are rich in unsaturated fats are flatter and this indicated lower rate of weight loss (dWt%/dt) (**Figs. 2B and 4B**) compared to SAF rich coconut oil . This was due to the fact that the thermo oxidative process involves oxygen insertion across the double bond (peroxidation)followed by breaking down of double bond to form primary and secondary oxidation products leading to some weight gain in the sample before being decomposed to volatile short chain fatty acids and carbonyl compounds. The relative

differences in the dWt% /dt (%/min) of the SAF rich COCNT oil, unsaturated fat (MUF & PUF) rich GNGLY, GNDNT and SNFL oils give different decomposition steps in TG (Saad et al. 2008).

The percentage weight losses observed in the three steps of oxidative degradation calculated from the TG traces (**Figs. 2A and 4A**) for the four oils in air were correlated to SAF, MUF and PUF contents and presented in Table 1. The 93.2% weight loss in the major weight loss step for COCNT oil was attributed to the decomposition of medium chain fatty acids (lauric, caprilic & capric acids). The 3.83 and 2.82% weight loses for refined COCNT oil **(Fig. 2A)** noted around 420 and 500^{o}C respectively may be attributed to the PUF and MUF respectively. Moreover the marginal differences in the weight loss observed for refined and virgin COCNT oils in air(**Figs. 2A** and **4A)** and nitrogen(**Figs.1A** and **3A**) at 300^{0} C more likely indicates that weight loss below 340^{o}C in COCNT may be attributed to its SAF content. The percentage weight losses observed at 300^{o}C in air for the virgin oils (**Fig.4A)** are slightly less than those observed for refined oils in air(**Fig. 2A)** which is the reverse of that observed in nitrogen. This may be attributed to the effect of antioxidant nutrient present in virgin oils.

The reported (Muneeshwari et al. 2017) iodine numbers which is a measure of unsaturation of these oils decreased in the order SNFL>GNGLY>GRDNT>COCNT for both the virgin and refined grades oils. The measured enthalpies (Jg^{-1}) (**Table 2**) for the exotherms of these oils from DTA trace in air(**Figs.2C** and **4C)** decreased in the order SUNF>GNGLY> GRNDNT indicating that their unsaturation may also decrease in this order because greater the unsaturation greater will be the thermoxidative degradation and hence greater the enthalpy change.

CONCLUSIONS

Thermal and thermooxidative stabilities of four commonly and commercially used refined and virgin grades of edible vegetable oils viz., GNGLY, COCNT, GRDNT and SUNFL oils were evaluated by simultaneous TGA-DTA analysis in pure air (80% N_2 & 20% O_2) and nitrogen. The thermal degradation features depended on the proportions of saturated and unsaturated fats (SAF, MUF, PUF) and the environment. The pure thermal degradation of these oils in nitrogen was endothermic and their oxidative thermal degradation in air was exothermic. TG data in N_2 and air for refined and virgin oils revealed that their thermal stabilities decreased in the order groundnut>gingley> sunflower>coconut and sunflower>groundnut> gingley>coconut in N_2 and groundnut> gingley> coconut>sunflower and gingley>groundnut> coconut> sunflower in air respectively. Eventhough COCNT oil is reported to have > 75 % medium chain saturated fat, it was found to be the least stable both thermally and thermooxidatively. This indicated the involvement of other factors apart from unsaturation in deciding thermal stability. In air the oils except COCNT undergone a distinctive three step oxidative degradation due to SAF, MUF and PUF contents. Based on the mass loss in the three steps the proportions of these fat components were calculated and compared with literature values. In general increased unsaturation will bring down the thermal and oxidative stability. Enthalpy data in air indicated that SNFL oil has the maxium unsaturation. The generated thermal stability data and degradation features will be useful for optimizing the conditions for processability, storage, reusability and quality control of edible oils for culinary use and other industrial applications such as biodiesel, lubricants and thermal energy storage fluids . Since thermal data are furnished rapidly with very small quantity of samples thermoanalytical methods are receiving increasing research interest.

ACKNOWLEDGEMENT

The author is very thankful to DST-FIST, Ministry of Science and Technology, New Delhi Government of India and Bannari Amman Institute of Technology, Sathyamangalam for proving the required Thermoanalytical instrument facility in the form DST-FIST project to carry out this research work.

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