

## DEVELOPMENT OF FATS BASED ON OLIVE OIL BY CHEMICAL INTERESTERIFICATION AND ITS UTILIZATION IN COOKIE PRODUCTION

D. KAÇAR<sup>a</sup>, H. ERİNÇ<sup>b</sup> and D. SIVRİ ÖZAY<sup>c\*</sup>

<sup>a</sup> Food Processing Department, Çaycuma Vocational School of Food and Agriculture, Bülent Ecevit University, Zonguldak, Turkey

<sup>b</sup> Department of Food Engineering, Niğde Ömer Halisdemir University, Niğde, Turkey

<sup>c</sup> Department of Food Engineering, Hacettepe University, Beytepe Campus 06800 Cankara/Ankara, Turkey

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Refined olive oil (ROO) was blended with tristearin (TS) and palm oil (PO) at two different ratios (ROO:TS:PO; 75:15:10 and 85:10:5) and chemical interesterification was performed in order to produce two interesterified fats (IF1 and IF2) with high unsaturated fatty acids and low *trans* fatty acids contents.

Solid fat content (SFC), slip melting point (SMP), and fatty acid composition were determined in IF1 and IF2; the values were compared to that of partially hydrogenated industrial cookie shortening (ICS). Higher SMP was measured in interesterified fats containing 75% ROO (IF1) and lower SMP was obtained in interesterified fats containing 85% ROO (IF2). SFC values of interesterified ROO blended oils were lower than ICS. Interesterified fats were used for cookie production, and cookie quality parameters, including oxidation stability, were determined.

Results showed that hardness values of cookies were not affected by fat types and higher spread ratios and lighter colors were obtained. IF1 and IF2 had higher total oxidation values as compared to ICS.

**Keywords:** chemical interesterification, olive oil, cookie, quality

Semi-solid plastic fats called shortenings are the most important ingredients succeeding flour and sugar in cookie production. Textural characteristics of cookies are provided by their high fat content. Generally, cookies contain 10–30% shortening depending on the formulations. Shortening is a solid fat product that is commonly used in baking industry because of its plasticity. Shortening is normally produced by partial hydrogenation process of vegetable oils such as cottonseed, palm, palm seed, sunflower, safflower, peanut, soybean, and canola oils (O'BRIEN, 1998; ZOULIAS et al., 2002). Hydrogenation is the saturation of double bonds in fatty acids with hydrogen by using a catalyst. Hydrogenation increases the slip melting point (SMP) of the fat, decreases the iodine value, and forms a fat more solid and saturated. However, hydrogenation causes return of the remaining unsaturated fatty acids to *trans* forms, decreasing the amount of essential fatty acids. *Trans* fatty acids are responsible for high low density lipoprotein (LDL) cholesterol levels and bad total cholesterol to high-density lipoprotein (HDL) cholesterol ratios causing arteriosclerosis. In addition, they are also associated with increased risk of breast cancer, disorders of the nervous system, colon cancer, diabetes, and allergy (DINC et al., 2014). Several studies showed that the major source of *trans* fatty acids in human diet is bakery products such as cookies, cakes, and breads (NORHAYATI et al., 2011). Fats for human consumption should contain less than 1% of the total

\* To whom correspondence should be addressed.

Phone: +90 312 297 62 11; fax: +90 312 299 21 23; e-mail: sivri@hacettepe.edu.tr

fat as *trans* fatty acids according to the World Health Organization (WHO) and Food Agriculture Organization (FAO).

NORHAYATI and co-workers (2011) determined total *trans* fatty acid content of local (packed) and imported biscuits as 0.35–4.83% and 0.45–2.90%, respectively. They also reported high level (51.6%) of *trans* fatty acid content in local unpacked biscuits. *Trans* fatty acids of cookies and cakes that are produced in Turkey were determined as 1–30% and 4.6%, respectively (DAGLIOGLU et al., 2002). KARABULUT (2007) found *trans* fatty acid content of cookies in the range of 2.25–3.54%.

Due to the adverse effect of *trans* fatty acids on health, interesterification process is widely used as an alternative of hydrogenation for the modification of oils. Interesterification is an acyl-rearrangement reaction on the glycerol molecule in a random manner without any change in chemical composition, and modifies the physico-chemical characteristics of oils and fats (AMIR et al., 2012). No transformation and isomeration occur in fatty acids of interesterified fats, and the nutritional properties of the triglycerides of interesterified oils are superior compared to the fats produced by the hydrogenation (SENANAYAKE & SHAHIDI, 2005). Interesterification improves the desirable characteristics of the mixture of lipids and enables to produce fats with suitable plasticity, melting point, and fatty acid composition (DA SILVA et al., 2010). Although enzymatic interesterification is available, chemical interesterification is mostly used due to being a simple, cheap, and easy modification method. In chemical interesterification sodium methoxide is used as catalyst. It is relatively inexpensive, readily available, and is allowed to be used as an adjuvant in foods. Sodium methoxide is a toxic chemical and is involved in the “Inventory of Substances used as Processing Aids (IPA) – Main List” in CODEX ALIMENTARIUS COMMISSION (2012). It is stated that the amount of residue should be <1 ppm. Although there have been many studies in the literature about fat production by chemical interesterification method, no studies have been found on residue detection in fats and in foods produced with these fats. In related works, sodium methoxide was assumed to be removed by consecutive water washings.

Olive oil with its fatty acid content of 55–83% monounsaturated fatty acid (oleic acid) and 3.5–21% polyunsaturated fatty acid (linoleic acid) is the member of the oleic-linoleic group oils, and has been reported to have beneficial effects on human health, such as lowering serum cholesterol levels, suppressing certain types of cancer, enhancing liver function, and reducing the effects of aging and heart disease. Many nutritionists have attributed that benefit to the high proportion of monounsaturated fatty acids (MUFA) and minor components such as polyphenols, antioxidants, sterols, etc. (BOSKOU, 2006; DA SILVA et al., 2010). DINC and co-workers (2014) chemically interesterified olive oil and its blends (25, 50, and 75%) with palm oil and produced zero-*trans* interesterified fats for use in cookie production. Oleic acid content increased with increasing olive oil content. Higher spread ratios were obtained in cookies produced with interesterified fats containing 50% and 100% olive oil. Lightness (L) and redness (a) values of the cookies decreased with olive oil addition, but yellowness (b) values were not affected.

In this study, the aim was to produce interesterified fats with high content of unsaturated fatty acids based on olive oil, with low *trans* fat contents for use in cookie production. Refined olive oil (ROO) was blended with tristearin (TS) and palm oil (PO) at two different ratios (ROO:TS:PO; 75:15:10 and 85:10:5), chemical interesterification was performed, and two interesterified fats (IF1 and IF2) were produced. Some physical and chemical parameters of interesterified fats and quality parameters of cookie samples were determined.

## 1. Materials and methods

### 1.1. Materials

The commercially refined, bleached, and deodorized olive oil was purchased from a local market. Palm oil and tristearin were obtained from Ülker Biscuits A.Ş. (Ankara, Turkey). Industrial cookie shortening (ICS) used as standard was supplied by ETİ Gıda Sanayi ve Tic. A.Ş. (Eskişehir, Turkey). A soft white wheat (cv. Eser) flour was used in cookie production.

### 1.2. Interesterification procedure

Interesterification reactions were carried out in a jacketed stirred-batch reactor (1 l) using a hot plate stirrer according to the method of ROUSSEAU and MARANGONI (1999) with some modifications. Total 300 g of ROO, TS, and PO were mixed in the ratios of 75:15:10 (IF1) and 85:10:5 (IF2), then the mixes were chemically interesterified for 50 min at 100 °C in the presence of the catalyst sodium methoxide (0.5%) under vacuum, and the reaction was completed by adding citric acid. The reaction mixture was washed with 55 °C (3 times × 150 ml) water and transferred to a beaker. For removing residual water, sodium sulphate was added to the mixture and vacuum filtration was applied. After filtration, the interesterified fats were stored at 4 °C.

### 1.3. Fat analysis

SMP of the samples were measured according to AOCS (2003) Official Method Cc 3-25. The solid fat content (SFC) was measured by low resolution pulsed NMR using Maran SFC (Resonance Instrument Ltd., Witney, UK) according to AOCS (2003) Official Method Cd 16b-93. Fatty acid methyl esters (FAMES) were prepared according to IUPAC (1987) method. The fatty acid compositions including *trans* forms were determined by Shimadzu GC-2010 gas chromatograph equipped with DB23 (60 m×0.250 mm× 0.25 µm) capillary column and flame ionization detector (FID). The carrier gas was helium (0.3 ml min<sup>-1</sup>). The split ratio was 1:80. The working temperatures of the injector, column, and detector were 230, 195, and 240 °C, respectively. FAMES were identified by standard FAMES.

Oxidative stability parameters (peroxide, conjugated dienes and trienes, *p*-anisidine values) were determined according to AOCS (2003) Official Method Cd 8-53, Ti-1a-64, Cd 18-90. Total oxidation (TOTOX) value was calculated according to SHAHIDI and WANASUNDARA (2002).

### 1.4. Flour analysis

Wheat flour was analysed for ash (08-01) according to AACC (1990) Standard Methods. Protein content of the flour was determined by NIR Systems Model 6500 Scanning spectrophotometer (A Perstorp Analytical Company, USA). Wet gluten was determined according to AACC (1990) Method No:38-11. Water absorption and physical dough properties were determined using the Brabender Farinograph (Duisburg, Germany) according to AACC (1990) Method No: 54-21.

### 1.5. Preparation of cookies and cookie analysis

IF1, IF2, and ICS were used in the cookie production according to AACC (1990) Method No. 10–54 in duplicate. Dough was rolled out (sheeted with the help of a rolling pin) and cut

using a circular die of 6.0 cm diameter. Cookies were baked at  $205 \pm 2$  °C for 11 min in a rotating electric oven (Şimşek Laborteknik Ltd. Şti., Ankara). After cooling of the cookies for 30 min, diameter (D) and thickness (T) measurements were taken using a caliper. Spread ratio was calculated from the ratio of diameter and thickness (D/T). Colour values ( $L^*$ ,  $a^*$ , and  $b^*$ ) were measured by Minolta Spectrophotometer CM-3600d (Japan). Breaking strength of the cookies was measured in a Texture Analyzer (TA-XTplus, UK) with 3-point bending rig (HDP/3PB). A crosshead speed of  $10 \text{ mm min}^{-1}$  with a load cell of 50 kg was used in the study. The span between the two platforms was 10 mm. The force required to break cookies individually were recorded and average values were reported as fracture force.

### 1.6. Statistical analysis

The spread ratio, color, and texture values of cookies and sensory analysis results were statistically evaluated by one-way analysis of variance procedure using ANOVA at 5% level of significance. The least significant difference (LSD) test was used to determine the differences among means. SPSS 11.5 programme was used for statistical analysis.

## 2. Results and discussion

### 2.1. Fat analysis

SMP value of ICS was determined as 41 °C. Higher SMP (45 °C) was measured in IF1 than in IF2 (25 °C). SMP of palm stearin changes between 46.6–53.8 °C. Thus, it could be explained with the higher percentage of palm stearin and lower percentage of ROO in IF1. DA SILVA and co-workers (2010) showed that chemical interesterification decreased the SMP of all virgine olive oil and palm stearin blends. This was because of diminishing of  $S_3+S_2U$  triacylglycerol with the contribution of unsaturated fatty acids in olive oil after interesterification. SFC values at different temperatures determine important characteristics of fats like physical appearance, organoleptic properties, consistency, and stability (CRIADO et al., 2008; RIBERIO et al., 2009). SFC curves of IF1, IF2, and ICS are presented in Fig. 1, measured at different temperatures (0, 10, 21.1, 33.3, and 40.6 °C). The SFC values of ICS, IF1, and IF2 were determined as 34.7%, 15.7%, and 5.1% at 21.1 °C, respectively. SFC values of IF1 and IF2 were lower than ICS due to low saturated fatty acid content. Addition of olive oil increased monounsaturated fatty acid percent and decreased SFC values. CRIADO and co-workers (2008) reported increase in SFC values with the increase of the ratio of the fully hydrogenated palm oil (HPO) in the blends of HPO and virgin olive oil. ALPASLAN and KARAALI (1998) suggested that enzymatically and chemically interesterified blends of olive oil and partially hydrogenated palm oil (30:70 and 40:60) could be used for commercial margarine production.

ICS had 45.92% palmitic acid content (results not shown). It is possible to say that palm oil was used in high amounts in the production of the ICS. In addition, other saturated fatty acids, such as lauric and myristic acid, contents of the ICS seemed to be higher than that of IF1 and IF2. In terms of stearic acid, IF1 and IF2 had higher stearic acid content. Due to the high amount of oleic acid in olive oil, oleic acid contents were quite high in IF1 and IF2 (with the value of 61.91% and 63.37%, respectively) as expected. Monounsaturated (MUFA), polyunsaturated (PUFA), and *trans* fatty acid (TFA) contents of the fats were given in Table 1. Saturated fatty acid content was the highest (60.57%) in ICS followed by IF1 (27.43%)

and IF2 (25.91%). MUFA content was the highest (64.71%) in IF2. The increase of olive oil content of the interesterified fats resulted in higher MUFA and lower PUFA contents (linoleic and linolenic acids) in IF1 and IF2.

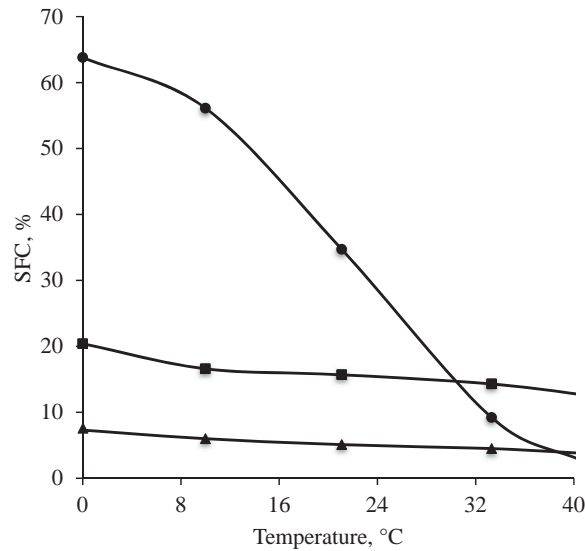


Fig. 1. Solid fat content curves of IF1, IF2, and ICS. ICS: Industrial cookie shortening; IF1: Interesterified fat 1: (ROO:TS:PO; 75:15:10); IF2: Interesterified fat 2: (ROO:TS:PO; 85:10:5)  
 ● : ICS; ■ : IF1; ▲ : IF2

Table 1. Saturated, monounsaturated, polyunsaturated, and *trans* fatty acid values of industrial cookie shortening and interesterified fats (%)

Fatty acids	Fat samples		
	ICS	IF1	IF2
Saturated	60.57	27.43	25.91
Monounsaturated	28.12	62.89	64.71
Polyunsaturated	10.96	9.02	8.86
<i>Trans</i>	0.35	0.58	0.51

ICS: Industrial cookie shortening; IF1: Interesterified fat 1: (ROO:TS:PO; 75:15:10); IF2: Interesterified fat 2: (ROO:TS:PO; 85:10:5)

*Trans* fatty acid values (0.58% and 0.51% for IF1 and IF2, respectively) of olive oil based interesterified fats were higher than that of ICS, because of thermal oxidation of fats during interesterification reaction (MOYA MORENO et al., 1999). On the other hand, these fats could be labeled as “zero *trans*” according to TURKISH FOOD CODEX (2007). DOĞAN and co-workers (2007) produced chemically interesterified “zero *trans*” fats from cottonseed and palm oil (50:50).

The oxidative stability parameters of fat samples are given in Table 2. The highest peroxide value in IF2 could be associated with higher ratio of polyunsaturated fats. Calculated

TOTOX value of IF2 was higher than IF1. It suggests that IF2 with higher amount of monounsaturated fatty acid content made it more susceptible for oxidation than IF1. Conjugated diene, *p*-anisidine and TOTOX values were the highest in ICS. It could be concluded that oxidative stability analysis in IF1 and IF2 were performed right after interesterification.

Table 2. Oxidative stability of industrial cookie shortening and interesterified fats

Oxidative stability parameters	ICS	IF1	IF2
Peroxide value (meq O <sub>2</sub> /kg fat)	3.3 ±0.04	3.1 ±0.13	3.9 ±0.34
Conjugated diene value (233 nm)	0.17 ±0.011	0.14 ±0.009	0.15 ±0.004
<i>p</i> -anisidine value (mMol/kg fat)	5.40 ±0.132	2.24 ±0.072	1.38 ±0.043
TOTOX Value	12.00	8.44	9.18

Values are the mean of duplicates.

ICS: Industrial cookie shortening; IF1: Interesterified fat1: (ROO:TS:PO; 75:15:10);

IF2: Interesterified fat 2: (ROO:TS:PO; 85:10:5)

## 2.2. Flour analysis

Soft white winter (SWW) wheat flour (cv. Eser) used in cookie production had 0.67% ash, 11.9% protein, and 32% wet gluten contents. Farinograph water absorption of the flour was 53.0%.

## 2.3. Cookie analysis

Colour values (L\*, a\*, and b\* values) of cookies are given in Table 3. According to this data, L (lightness) values of cookies produced with ICS were significantly higher than of the cookies produced with IF1 and IF2 (P<0.05). However, the difference between IF1 and IF2 was statistically not important. No significant difference was observed in a (redness) and b values (yellowness) among the cookie samples.

Table 3. Quality characteristics of cookie samples produced by using industrial cookie shortening and interesterified fats

Quality parameters	ICS	IF1	IF2
Cookie L (lightness) value	71.09 <sup>a</sup>	67.81 <sup>b</sup>	67.88 <sup>b</sup>
Cookie a (redness) value	6.99 <sup>a</sup>	7.08 <sup>a</sup>	6.96 <sup>a</sup>
Cookie b (yellowness) value	29.69 <sup>a</sup>	29.97 <sup>a</sup>	29.95 <sup>a</sup>
Diameter (cm)	7.72 <sup>c</sup>	8.08 <sup>b</sup>	8.24 <sup>a</sup>
Thickness (cm)	0.95 <sup>a</sup>	0.92 <sup>ab</sup>	0.91 <sup>b</sup>
Spread ratio	8.09 <sup>c</sup>	8.76 <sup>b</sup>	9.11 <sup>a</sup>
Hardness value (N)	6039 <sup>a</sup>	5508 <sup>a</sup>	6345 <sup>a</sup>

Values are means of duplicate measurements. LSD: the least significant difference.

<sup>a,b,c</sup>: Means followed by different letter within each row are significantly different according to the LSD test (P<0.05)

ICS: Industrial cookie shortening, IF1: Interesterified fat 1: (ROO:TS:PO; 75:15:10); IF2: Interesterified fat 2: (ROO:TS:PO; 85:10:5)

Spread ratio (D/T) is one of the most important properties in cookie quality. Greater spread ratios are desirable and indicate a better cookie quality (SEKER et al., 2010). Diameter, thickness, and spread ratio values of cookies are given in Table 3. The highest diameter values were obtained from the cookies produced with IF2 ( $P<0.05$ ). As the olive oil ratio in interesterified fats increased, the spread ratios also increased indicating better quality.

Textural properties of the cookies are given in Table 3. Cookies produced with IF2 had higher hardness values than cookies produced with IF1. Textural properties of cookies were comparable ( $P>0.05$ ). This indicates that hardness values increased with the increasing olive oil content in formulation. It is well known that fat types (bakery fat, hydrogenated fat, margarine, and sunflower oil) have different effects on the quality of cookies, and cookies produced with oils have relatively harder texture due to poor entrapment of air during creaming (JACOB & LEELAVATHI, 2006).

### 3. Conclusions

Olive oil based interesterified fat containing 85% olive oil with high amount of monounsaturated fatty acid (64.71%) was produced by chemical interesterification. The amounts of unsaturated fat increased and saturated fat decreased with increase of olive oil content in interesterified fat formulation. It should be taken into account that a solid fat product having high amount of olive oil was produced, while in the market the maximum amount of olive oil in margarines produced with olive oil is 12%.

The resulting interesterified fats had lower amounts of SFC when compared with ICS, and SMP of IF2 was found to be lower than IF1. Cookie texture was not affected by the fat type, and higher spread ratios were obtained in cookies produced with IF1 and IF2 compared with ICS.

It could be suggested that lower amount of olive oil (50–60%) used in interesterified olive oil blends could result in harder fats with higher SFC values therefore better cookie quality could be obtained. Taken into consideration that two different interesterified “low *trans*” fat products containing olive oil at high rates were produced, it is possible to say these products have commercial potential in food industry.

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