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Formulation Optimization and Thermal Behavior of a Low-Trans Ternary Lipid Matrix from Fully Hydrogenated Rapeseed, Sea Buckthorn, and Rapeseed Oils for Potential Specialty Confectionery Fat Applications

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Abstract

The demand for sustainable, low-trans specialty confectionery fats drives the search for novel lipid sources. This study optimizes a ternary lipid matrix utilizing fully hydrogenated rapeseed oil (FHRSO), sea buckthorn oil (SBO), and native rapeseed oil (RSO) to overcome the structural limitations of previous binary systems. Formulations were modified via chemical interesterification, and optimization was conducted using a simplex-lattice mixture design targeting iodine value, slip melting point (SMP), and unsaturated-to-saturated fatty acid ratios. The optimal blend (57% FHRSO, 9.5% SBO, 33.5% RSO) achieved an experimental SMP of 36.3 °C and a trans fatty acid content of 0.9%, strictly complying with TR CU 024/2011 regulations. The resulting triacylglycerol profile, characterized by mixed types (SOO, SOS, LOS), demonstrated compositional compatibility with the structural requirements of confectionery fats, supported by a solid fat content profile indicating appropriate melting dynamics. The strategic incorporation of RSO effectively improved the plasticity of the lipid network. In conclusion, this study presents the development of a locally sourced, low-trans ternary fat system that presents suitable physicochemical properties for future applications in specialty confectionery fat formulations.

Keywords: chemical interesterification; ternary lipid matrix; fully hydrogenated rapeseed oil; sea buckthorn oil; low-trans fat; specialty fat; simplex-lattice design; thermal behavior

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1. Introduction

Cocoa butter (CB) is a key component in chocolate and various confectionery products, significantly influencing their organoleptic and physical characteristics [1]. As an indispensable raw material, CB imparts chocolate with its characteristic snap, gloss, bloom resistance, and smooth mouthfeel [2]. Furthermore, due to its specific fat composition and crystallinity, chocolate is notably distinguished by β″ crystals [3]. However, the increasing demand for chocolate and confectionery, coupled with supply chain instability, drives up cocoa bean prices, potentially threatening food security. Consequently, the food industry is actively seeking CB alternatives by utilizing oils extracted from various botanical sources [4].

The price of cocoa butter remains among the highest for commercial fats and oils due to limited availability and high demand. Therefore, food companies are making intensive

efforts to develop cocoa butter replacer (CBR) production methods for emerging food markets [5]. The Republic of Kazakhstan, being a landlocked country, faces challenges in sourcing tropical oils, including palm oil, leading to complete dependence on imported raw materials. This reliance poses additional risks to food security and increases production costs in the food industry. Given the lack of domestic tropical oil production, developing new lipid matrices based on accessible vegetable oils with specific targeted physicochemical characteristics is a primary objective.

According to the Technical Regulation of the Customs Union [6], since 2018, the content of trans fatty acid isomers in fat-and-oil products must not exceed 2%, with the exception of non-tempered non-lauric CBRs. Restrictions on trans isomers for this fat category came into full effect at the beginning of 2026, highlighting the immediate urgency of developing fat blends compliant with these active regulatory standards. Such restrictions stimulate the search for innovative solutions that ensure both technological functionality and food safety compliance. A highly promising approach is the interesterification of oil blends to produce specialty fats with tailored technological properties. However, the vast majority of studies utilize refined and traditional raw materials, focusing primarily on soybean oil and palm oil.

Among domestically produced oils in Kazakhstan, sea buckthorn oil (SBO), fully hydrogenated rapeseed oil (FHRSO), and rapeseed oil (RSO) attract particular attention. Sea buckthorn *(Hippophae rhamnoides)* holds a special place in the agro-industrial sector due to its versatility, high yield, and unique fruit properties. SBO exhibits potential health benefits, including cardiovascular protection and antioxidant properties, largely attributed to bioactive components like palmitoleic acid and β-carotene [7,8]. FHRSO is produced by the complete hydrogenation of rapeseed oil, yielding a fat composed entirely of saturated fatty acids and devoid of trans isomers [9,10]. The utilization of FHRSO in CBR formulations ensures regulatory compliance, lowers costs associated with imported palm oil, and provides a stable crystalline structure suitable for confectionery, while native rapeseed oil improves plasticity and functional properties.

Modifying lipid profiles through chemical interesterification is essential to meet the complex requirements of specific food applications [11]. In our previous research, we successfully demonstrated the feasibility of creating specialty fats for confectionery glazes based on a binary system comprising fully hydrogenated rapeseed oil (FHRSO) and refined sea buckthorn oil (RSBO) [12]. It was established that the chemical interesterification of this two-component blend (specifically at a 20/80 ratio) yields a trans-free fat with an acceptable melting profile and optimal rheological characteristics for coating applications. While our previous binary system (FHRSO/RSBO) successfully yielded trans-free fats suitable for basic coating applications, it exhibited inherent limitations in phase behavior, lacking the structural flexibility and precise solid fat content (SFC) modulation required for high-quality non-lauric cocoa butter replacers (CBRs). To overcome these constraints, the current study conceptualizes a more sophisticated ternary lipid matrix by introducing native rapeseed oil (RSO) as a crucial structural modifier. The integration of RSO provides a rich source of specific unsaturated acyl groups for complex randomization during chemical interesterification. This strategic addition aims to mitigate eutectic limitations and improve structural flexibility, transitioning the product from a simple binary fat to a more versatile ternary matrix. While binary systems offer basic functionality, the existing scientific gap lies in achieving the precise balance of plasticity, melting dynamics, and solid fat content required for high-quality confectionery fats without relying on tropical imports or partial hydrogenation.

Therefore, the objective of this study is to develop low-trans specialty fat blends with specific target characteristics using accessible local vegetable oils of Kazakhstan (FHRSO, SBO, and RSO), modified through the chemical interesterification process.

2. Materials and Methods

2.1. Materials and Sample Preparation

Fully hydrogenated rapeseed oil (FHRSO) and refined rapeseed oil (RSO) were purchased from Maslo-Del LLP (Almaty, Kazakhstan). Sea buckthorn fruits (*Hippophae rhamnoides* L.) were procured from a local market in Astana, Kazakhstan, and subsequently dried in a laboratory drying oven. The dried berries were pulverized using a Hotmix PRO Master thermomixer. The resulting dried and milled sea buckthorn powder was placed into a cellulose thimble and extracted with n-hexane at 60 °C for 5 h using a Soxhlet apparatus [13]. Upon completion of the extraction, the solvent was evaporated using an IKA RV 10 rotary evaporator [14]. The obtained crude sea buckthorn pulp oil was then subjected to refining in a laboratory-scale neutralizing and bleaching reactor (Scientific Research Instrument, Vadodara, India) to remove undesirable impurities.

Analytical standards, including a fatty acid methyl ester mixture (Supelco 37 Component FAME Mix), trielaidin, triolein, n-hexane, and powdered sodium methoxide (used as a catalyst), were purchased from Sigma-Aldrich (St. Louis, MO, USA). All other chemical reagents and solvents used in the experiments were of analytical grade and used without further purification.

2.2. Preparation of Fat Blends

The samples were formulated as ternary physical mixtures comprising FHRSO, SBO, and RSO at various mass ratios (w/w/w). Prior to blending, the individual lipid components were completely melted at 70 ± 1 °C to eliminate thermal history and ensure complete liquefaction. The components were then combined and thoroughly homogenized under continuous stirring for 10 min at the same temperature. The resulting physical fat blends were subsequently stored at 5 ± 1 °C until further processing and chemical interesterification.

2.3. Chemical Interesterification

Chemical interesterification of the ternary fat blends was performed in a 500 mL borosilicate glass laboratory reactor equipped with an overhead mechanical stirrer. Prior to the reaction, 200 g of the physical fat mixture was dried under reduced pressure at 130 °C and a residual pressure of 4 kPa for 30 min to remove moisture and volatile impurities. After the initial drying step, the fat blend was cooled to 80 °C, and 2.4 g of powdered sodium methoxide was added as a catalyst. The interesterification reaction was carried out at 110 °C under continuous stirring at 500 rpm for 60 min. Upon completion, the reaction was terminated by the addition of 10 mL of distilled water preheated to 60 °C to deactivate the catalyst. Finally, the reaction mixture was subjected to vacuum filtration using filter paper to remove solid residues, and the filtered fat was dried again under the same initial conditions (130 °C, 4 kPa) to remove residual moisture.

2.4. Determination of Iodine Value

The iodine value (IV) of the fat samples was determined according to the AOCS Official Method Cd 1c-85.

2.5. Determination of Slip Melting Point

The slip melting point (SMP) of the fat blends was determined using the open capillary tube method in accordance with the AOCS Official Method Cc 1-25.

2.6. Fatty Acid Composition

The fatty acid composition was determined by converting the fatty acids into their corresponding methyl esters (FAME) according to the method proposed by Rodrigues et al. [15]. FAME analysis was performed using an Agilent 7890B gas chromatograph (Agilent Technologies, Santa Clara, CA, USA) equipped with a flame ionization detector (FID) and a CP-Sil 88 capillary column (100 m length, 0.25 mm internal diameter, 0.20 μm film thickness). The operating conditions were established as follows: helium was used as the carrier gas, the FID temperature was set at 200 °C, the injector temperature was maintained at 250 °C, and the injection volume was 1 μL. Individual fatty acid peaks were identified by comparing their retention times with those of a standard fatty acid mixture analyzed under identical operating conditions. The quantitative results were expressed as the relative percentage of each individual fatty acid with respect to the total fatty acid content.

2.7. Determination of Trans Fatty Acid Isomers

The content of isolated trans fatty acid isomers in the oils and fat blends was determined using Fourier transform infrared spectroscopy with attenuated total reflectance (FTIR-ATR). This method, performed strictly in accordance with the AOCS Official Method Cd 14d-99, was selected as a rapid, highly reproducible quantitative screening tool widely recognized for evaluating modified fat blends to ensure general regulatory compliance. Infrared absorption spectra of calibration standards and fat samples were recorded using an IRSpirit-TX spectrometer (Shimadzu, Kyoto, Japan) equipped with a Quest ATR Accessory GS10800 Series (Specac, Orpington, UK) featuring a zinc selenide (ZnSe) ATR crystal. The samples were placed directly onto the ATR crystal, which was maintained at 65 °C to ensure that the crystal was completely covered by the melted sample, and each measurement was performed in triplicate. Spectra were collected continuously in the wavenumber range of 700–4000 cm^{-1} with 64 scans at a spectral resolution of 4 cm^{-1}, utilizing air as the background reference. The ATR crystal was thoroughly cleaned after each scan. The integrated absorbance area in the spectral region between 990 and 945 cm^{-1} was calculated using LabSolutions IR software and compared with calibration standards using linear regression to determine the quantity of trans fatty acid isomers.

2.8. Determination of Triacylglycerol Composition

The triacylglycerol (TAG) composition was analyzed using a high-performance liquid chromatograph (HPLC, Agilent 1200, Agilent Technologies, Santa Clara, CA, USA) equipped with a diode-array detector (DAD) set at 210 nm. Approximately 50 mg of the melted sample was dissolved in 1 mL of acetone, filtered through a 0.45 μm PTFE membrane, and injected (10 μL) into a C18 reversed-phase column (LiChrospher RP-18, 250 × 4.6 mm, 5 μm; Merck, Darmstadt, Germany). The column temperature was maintained at 35 °C. The mobile phase consisted of acetone-acetonitrile (60:40, v/v) at a flow rate of 1 mL min^{-1}. Major TAGs were identified using authentic standards (PPP, POP, SOS, SOO, OOO), while remaining TAGs were tentatively identified by comparing equivalent carbon numbers (ECN) and literature retention times.

2.9. Determination of Solid Fat Content

The solid fat content (SFC) was determined using a Minispec mq one nuclear magnetic resonance (NMR) spectrometer (Bruker Corporation, Bremen, Germany). Measurements were performed using the direct method, and readings were recorded sequentially at temperatures of 10, 20, 25, 30, 35, and 40 °C in accordance with the AOCS Official Method Cd 16b-93.

2.10. Experimental Design and Statistical Analysis

To optimize the ternary fat composition comprising FHRSO, SBO, and RSO, a three-component simplex-lattice mixture design was applied. This approach generated 10 experimental points under the constraint that the sum of the component proportions equaled 100%, with an additional constraint limiting the SBO fraction (≤40%). The response variables evaluated were the iodine value (IV), the unsaturated to saturated fatty acid ratio (USFA/SFA), and the slip melting point (SMP). The experimental data for IV and USFA/SFA were analyzed using special cubic regression models, whereas the SMP data were fitted to a quadratic regression model. The significance of the models was evaluated by analysis of variance (ANOVA) at a confidence level of 95% ($p < 0.05$). The validity of the regression models was confirmed by calculating the coefficients of determination (R^2). Formulation optimization was conducted analytically via response surface methodology (RSM) by overlaying the contour plots. The target parameters were defined based on the optimal physical requirements for specialty confectionery fats: IV = 47.0–48.0 g I_2/100 g, USFA/SFA = 1.0, and SMP = 36.0–37.0 °C. All statistical analyses for this study were performed using Statgraphics 19 software (version 19, Statgraphics Technologies, Inc., The Plains, VA, USA).

3. Results and Discussion

3.1. Characterization of the Initial Vegetable Oils

The fatty acid and triacylglycerol (TAG) compositions of the oils used as raw materials are presented in Table 1. The results revealed marked differences among FHRSO, SBO and RSO, which define their thermal and structural behavior in subsequent interesterification.

The FHRSO showed an extremely high proportion of saturated fatty acids (SFA = 95.5%, IV = 4.6 ± 0.3 g I_2/100 g), dominated by stearic (C18:0, 89.8%) and palmitic (C16:0, 4.4%) acids. Its TAG composition consisted mainly of tristearin (SSS, 91.4%) and distearoyl-palmitoyl-glycerol (SSP, 5.6%), corresponding to the structure of a completely hydrogenated fat. Similar data were reported for fully hydrogenated canola and soybean oils by Ribeiro et al. [16] and Ehsan Jenab et al. [17], who observed predominant SSS and SOS fractions responsible for high melting points (60–65 °C) and a strong tendency toward β-type crystallization. Such structural organization provides rigidity and oxidative stability, which is essential for the hard-fat component in low-trans fat systems [9,18].

The SBO exhibited a remarkably different composition, characterized by palmitoleic acid (C16:1, 45.8%) and palmitic acid (C16:0, 26.1%) as the main components, with an iodine value of 78.1 ± 1.1 g I_2/100 g. This is in agreement with Arnau Vilas-Franquesa et al. [13], Dolzhenko et al. [19], and Li et al. [20], who described sea buckthorn oil as rich in C16:1 and minor C18:1-C18:2 fractions.

Its TAG profile was dominated by POO (28.6%), PPP (32.4%), LOO (18.8%), and OOO (13.2%), consistent with previous reports [21,22], indicating the coexistence of both symmetric (PPP) and asymmetric (POO, LOO) TAGs. This TAG distribution suggests a strong tendency toward β′-type crystallization and contributes to a low slip melting point (25.7 ± 0.5 °C), which is beneficial for soft, smooth-textured fats [23]. In addition, the high

content of palmitoleic acid contributes specifically to the compositional diversity of the lipid matrix [7,8].

Table 1. Chemical composition and properties of FHRSO, SBO and RSO.

Items	FHRSO	SBO	RSO
Iodine value (I2/100 g)	4.6 ± 0.3	78.1 ± 1.1	111.5 ± 1.4
Slip melting point (°C)	63.4 ± 0.3	25.7 ± 0.5	-
Main fatty acids (%)			
C14:0	ND	0.3 ± 0.2	ND
C16:0	4.4 ± 0.1	26.1 ± 0.3	4.2 ± 0.1
C16:1	ND	45.8 ± 0.5	ND
C18:0	89.8 ± 0.5	2.5 ± 0.7	1.7 ± 0.5
C18:1	3.6 ± 0.6	10.8 ± 0.7	60.9 ± 0.5
C18:2	0.1 ± 0.7	13.5 ± 0.6	19.8 ± 0.6
C18:3	0.1 ± 0.5	0.6 ± 0.5	9.5 ± 0.7
C20:0	1.3 ± 0.7	ND	ND
C22:0	ND	ND	2.8 ± 0.5
TFA	0.7 ± 0.2	0.8 ± 0.3	0.5 ± 0.3
SFA	95.5	28.9	8.7
USFA	3.7	70.7	90.2
TAG composition (%)			
LnLO	ND	ND	2.8
LLO	ND	ND	4.5
LnOO	ND	ND	7.9
LOO	ND	18.81	28.7
LOP	ND	ND	2.1
OOO	ND	13.2	47.9
POO	ND	28.6	2.8
PPP	ND	32.4	0.4
SOO	ND	ND	0.8
SSP	5.6	ND	ND
SSS	91.4	ND	ND

ND: Not detected.

The RSO contained predominantly unsaturated fatty acids (USFA = 90.2%, IV = 111.5 ± 1.4 g I_2/100 g), with oleic (C18:1, 60.9%), linoleic (C18:2, 19.8%), and linolenic (C18:3, 9.5%) as the major components. The TAG profile—OOO (47.9%), LOO (28.7%), and LnOO (7.9%)—is typical for refined rapeseed oils, as also reported by Jenab & Temelli [10] and Lazdovica & Kampars [24]. This composition ensures high fluidity at ambient conditions and provides a rich source of unsaturated acyl groups for redistribution during interesterification, which can yield mixed TAGs such as SOS, SOO, and POS [25,26].

Overall, the comparison indicates that FHRSO provides a rigid, high-melting matrix (SSS/SOS), whereas SBO and RSO supply low- and medium-melting components (POO, LOO, OOO) that contribute to the desired plasticity and structural flexibility. This complementary combination aligns with earlier reports on the design of low-trans specialty confectionery fats using mixtures of fully hydrogenated and unsaturated vegetable oils [4,11,27].

The resulting mixture, after interesterification, is expected to exhibit a narrow melting range (35–42 °C) and a clean melt comparable to the established physical profiles of natural cocoa butter [1,2].

3.2. Fatty Acid Composition of Fat Blends

The fatty acid composition of the interesterified ternary fat blends, as detailed in Table 2, demonstrates significant variations depending on the ratio of the starting oils, reflecting the redistribution of acyl groups among triacylglycerols (TAGs). Pure FHRSO retained its high SFA content of 95.5%, with a minimal USFA content of 3.7% and TFA levels of 0.8 ± 0.1%, serving as the baseline for the hard fat. Blends incorporating SBO, such as FHRSO:SBO:RSO = 66.6:33.3:0, exhibited a reduced USFA content of 26.0% and an elevated SFA content of 73.3%, with a notable increase in palmitoleic acid (15.8 ± 0.8%) and linoleic acid (4.6 ± 0.1%) derived from the SBO. This shift is consistent with the interesterification process randomizing fatty acids, as described by Norizzah et al. [28], who observed similar changes in palm oil-based blends leading to improved functionality.

Table 2. Fatty acid composition of the interesterified fat blends.

Fat Blends (w%/w%/w%)			Fatty Acid (%)									TFA
FHRSO	**SBO**	**RSO**	**C14:0**	**C16:0**	**C16:1**	**C18:0**	**C18:1**	**C18:2**	**C18:3**	**C20:0**	**C22:0**	
100	0	0	ND	4.4 ± 0.5	ND	89.8 ± 0.5	3.6 ± 0.2	0.1 ± 0.3	ND	1.3 ± 0.2	ND	0.8 ± 0.1
66.6	33.3	0	0.1 ± 0.4	11.3 ± 0.5	15.8 ± 0.8	60.6 ± 1.6	5.9 ± 0.2	4.6 ± 0.1	0.2 ± 0.1	0.9 ± 0.3	0.9 ± 0.3	1.1 ± 0.2
66.6	0	33.3	ND	4.3 ± 0.1	ND	58.4 ± 1.5	22.7 ± 1.1	6.6 ± 0.2	3.2 ± 0.3	0.8 ± 0.4	ND	1.0 ± 0.3
33.3	66.6	0	0.2 ± 0.5	18.7 ± 1.7	28.9 ± 1.0	31.8 ± 1.0	8.3 ± 0.3	8.7 ± 0.6	0.4 ± 0.1	0.4 ± 0.2	0.9 ± 0.5	1.5 ± 0.2
33.3	33.3	33.3	0.1 ± 0.6	11.4 ± 1.1	15.0 ± 0.6	30.9 ± 0.2	25.3 ± 0.4	11.4 ± 0.5	3.4 ± 0.2	0.3 ± 0.5	1.8 ± 0.4	0.9 ± 0.1
33.3	0	66.6	ND	4.4 ± 0.4	ND	32.1 ± 0.6	42.9 ± 1.2	12.9 ± 0.7	6.3 ± 0.1	0.4 ± 0.3	ND	1.1 ± 0.3
0	100	0	0.3 ± 0.7	26.0 ± 1.2	45.4 ± 0.5	2.5 ± 0.1	11.2 ± 0.6	13.5 ± 0.5	0.6 ± 0.2	ND	ND	0.7 ± 0.3
0	66.6	33.3	0.2 ± 0.5	18.6 ± 0.4	29.9 ± 0.4	2.2 ± 0.1	27.3 ± 0.9	15.8 ± 0.6	3.6 ± 0.3	ND	0.9 ± 0.2	1.4 ± 0.1
0	33.3	66.6	0.1 ± 0.7	11.1 ± 0.6	15.6 ± 0.4	2.1 ± 0.2	44.7 ± 1.1	17.9 ± 0.7	6.5 ± 0.5	ND	1.7 ± 0.4	1.2 ± 0.2
0	0	100	ND	4.3 ± 0.2	ND	1.8 ± 0.1	60.9 ± 1.4	19.8 ± 0.8	9.5 ± 0.6	ND	2.9 ± 0.3	0.7 ± 0.1

ND: Not detected.

The FHRSO:SBO:RSO = 33.3:33.3:33.3 blend achieved a balanced USFA/SFA ratio of approximately 1.2, with oleic acid (25.3 ± 0.4%) and linoleic acid (11.4 ± 0.5%) contributing to fluidity, while stearic acid (30.9 ± 0.2%) provided structural integrity. TFA levels remained consistently low across all blends (0.7–1.5%), confirming the efficacy of utilizing complete hydrogenation to prevent the formation of trans isomers, as noted by Farmani et al. [18] in rapeseed oil blends. Blends with a higher RSO proportion, such as FHRSO:SBO:RSO = 0:0:100, showed the highest USFA content at 90.2%, reflecting the native RSO profile with minor adjustments post-interesterification.

Compared to the literature, these profiles resemble those structured confectionery fats derived from mahua oil and palm stearin [3], where interesterification enhanced lipid compatibility. The incorporation of SBO introduced unique monounsaturated fatty acids, contributing to the diversity of the lipid matrix, as discussed by Oliveira et al. [29] regarding fatty acid distribution. This randomization via sodium methoxide catalysis [24] effectively tailored the blends for specialty fat applications, reducing the reliance on palm oils and aligning with the findings of Jahurul et al. [5].

3.3. Solid Fat Content

Figure 1 illustrates the solid fat content (SFC) profiles of the interesterified blends at temperatures ranging from 10 to 40 °C, demonstrating the influence of blend composition on melting characteristics, which are critical for chocolate-like textures. Pure FHRSO exhibited the highest SFC, ranging from 95.8% at 10 °C to 79.8% at 40 °C, indicating its fully saturated nature and suggesting the presence of stable β-crystals, analogous to fully hydrogenated oils in shortening formulations [30]. The incorporation of SBO and RSO progressively decreased the SFC; for instance, the FHRSO:SBO:RSO = 66.6:33.3:0 blend showed an SFC of 71.8% at 10 °C, which decreased to 27.0% at 40 °C, reflecting the softening effect of the USFAs from SBO that disrupt crystal packing [31].

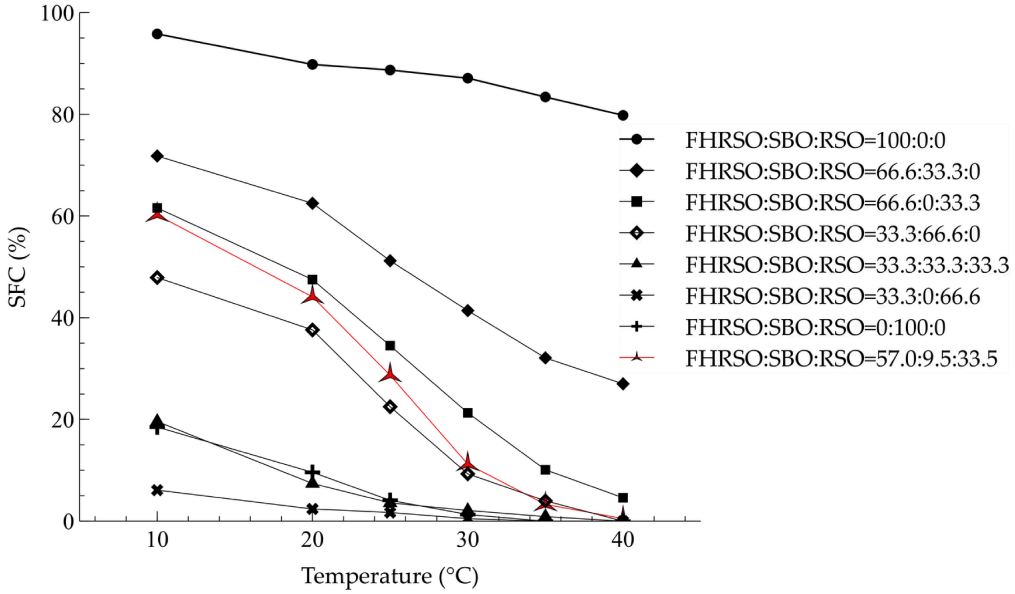


Figure 1. Solid fat content (SFC) profiles of the interesterified ternary fat blends at temperatures ranging from 10 to 40 °C.

Blends with a higher RSO content, such as FHRSO:SBO:RSO = 33.3:0:66.6, exhibited a low SFC of 6.1% at 10 °C and 0% at 35 °C, behaving more like liquid oils, which aligns with the findings of Ebrahimi et al. [25], who modeled sigmoidal melting curves in hydrogenated soybean oil blends. Notably, the FHRSO:SBO:RSO = 33.3:33.3:33.3 blend had an SFC of 19.6% at 10 °C, dropping sharply to 0% at 40 °C. This suggests a steep melting profile, which is considered a critical thermal prerequisite for achieving the desired textural properties in specialty confectionery fats, similar to the rapid melting behavior of cocoa butter as widely reported in the literature [2]. This is attributed to the interesterification process randomizing the TAGs, reducing eutectic interactions, and broadening the melting ranges, as observed in crambe oil blends [26].

Blends containing ≥66.6% SBO or RSO were completely liquid (SFC = 0%) at temperatures above 20 °C, limiting their application but underscoring the role of SBO in fluidity [32]. Compared to enzyme-catalyzed blends [17], the chemical interesterification applied in this study achieved a comparable SFC modulation at lower costs [33], while the low TFA content ensured compliance with regulatory requirements [34]. These characteristics position the developed blends as viable low-trans alternatives to palm oil-based specialty confectionery fats [35].

3.4. Experimental Design and Optimization of the Three-Component Fat Blends

The experimental design and statistical analysis were conducted using a three-component (FHRSO, SBO, and RSO) simplex-lattice mixture design of degree 2, which yielded 10 experimental points (Table 3), under the constraint that the sum of the component proportions equaled 100%, with an additional constraint limiting the SBO fraction to ≤40%. The evaluated response variables were iodine value (IV), slip melting point (SMP), and the ratio of unsaturated to saturated fatty acids (USFA/SFA). Since native RSO is completely liquid at ambient temperatures, its slip melting point (SMP) cannot be physically determined using the standard capillary method. Due to this physically justified absence of data at the liquid vertex, the SMP response was adequately fitted to a quadratic regression model, whereas IV and USFA/SFA data, being chemically determinable across all physical states, were fitted to special cubic regression models.

Table 3. Experimental data of the simplex-lattice mixture design for the optimization of the ternary fat blends.

Fat Blends (w%/w%/w%)			IV (g/100 g)	SMP (°C)	USFA/SFA
FHRSO	SBO	RSO			
100	0	0	4.6	63.4	0
66.6	33.3	0	29.6	50.9	0.4
66.6	0	33.3	39.9	41.8	0.5
33.3	66.6	0	54.3	34	0.9
33.3	33.3	33.3	65	24.5	1.2
33.3	0	66.6	77.6	13.3	1.6
0	100	0	78.6	25.1	2.4
0	66.6	33.3	90.9	-	3.5
0	33.3	66.6	99.7	-	5.4
0	0	100	112	-	10.4

For IV, ANOVA indicated a highly significant model (F-value = 1170.53, $p = 0.0000$) with $R^2 = 99.96\%$ (adjusted $R^2 = 99.87\%$), confirming an excellent fit. The quadratic model for SMP was also highly reliable ($p = 0.1099$) with $R^2 = 99.57\%$ (adjusted $R^2 = 97.40\%$). For USFA/SFA, the model showed a significant correlation (F-value = 16.23, $p = 0.0218$) with $R^2 = 97.01\%$ (adjusted $R^2 = 91.03\%$), yielding the following regression equations:

$$\begin{aligned} IV = {} & 4.32383 \times FHRSO + 78.9381 \times SBO + 111.938 \times RSO + 1.43568 \times FHRSO \times \\ & SBO + 2.78571 \times FHRSO \times RSO - 0.621391 \times SBO \times RSO - 12.5983 \times FHRSO \times SBO \\ & \times RSO \end{aligned} \quad (1)$$

$$\begin{aligned} SMP = {} & 64.02 \times FHRSO + 24.48 \times SBO - 21.4798 \times RSO - 8.1 \times FHRSO \times SBO + \\ & 28.25.96 \times FHRSO \times RSO - 0.719903 \times SBO \times RSO \end{aligned} \quad (2)$$

$$\begin{aligned} USFA/SFA = {} & 0.380954 \times FHRSO + 2.46667 \times SBO + 9.95238 \times RSO - 3.48215 \times \\ & FHRSO \times SBO - 18.525 \times FHRSO \times RSO - 7.91786 \times SBO \times RSO + 6.975 \times FHRSO \times \\ & SBO \times RSO \end{aligned} \quad (3)$$

It should be noted that the noticeable difference between R^2 and adjusted R^2 (e.g., 97.01% vs. 91.03% for the USFA/SFA ratio) is mathematically expected due to the limited degrees of freedom inherent in a 10-point special cubic mixture design. However, all evaluated models remained statistically highly significant ($p < 0.05$). These diagnostics confirm that the selected equations adequately predict the experimental responses without substantial overfitting. The contour plots (Figure 2 for IV, Figure 3 for SMP, and Figure 4 for USFA/SFA) illustrate the response surfaces, demonstrating how an increase in the proportions of SBO and RSO elevates IV and USFA/SFA while decreasing SMP, with optimal regions centered around 50–60% FHRSO, 5–15% SBO, and 25–40% RSO. Formulation optimization was conducted analytically via response surface methodology (RSM) by overlaying the contour plots. The target formulation criteria were defined based on the physical requirements for specialty confectionery fats: an SMP of 36.0–37.0 °C (to ensure complete oral melting at human body temperature) and an IV of 47.0–48.0 g I_2/100 g for adequate plasticity. This graphical optimization identified an optimal blend of 57.0% FHRSO, 9.5% SBO, and 33.5% RSO. At these coordinates, the updated models accurately predicted values of IV ≈ 47.8 g I_2/100 g, USFA/SFA ≈ 1.0, and SMP ≈ 36.6 °C.

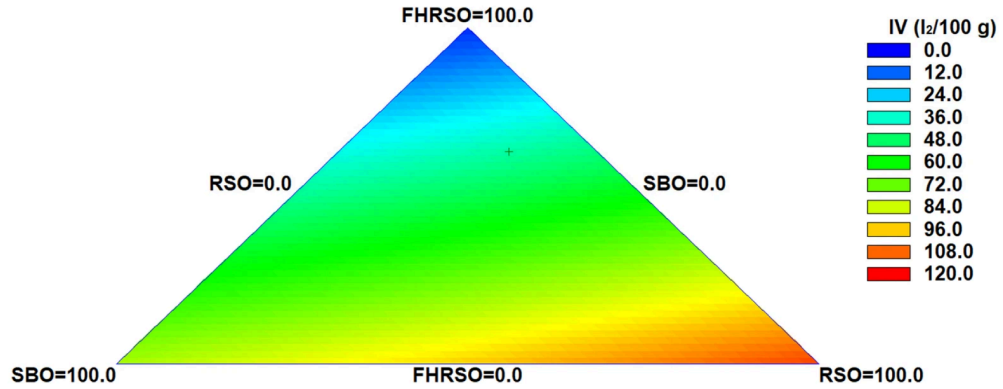


Figure 2. Contour plot showing the effect of the ternary blend composition on the iodine value (IV, g I_2/100 g).

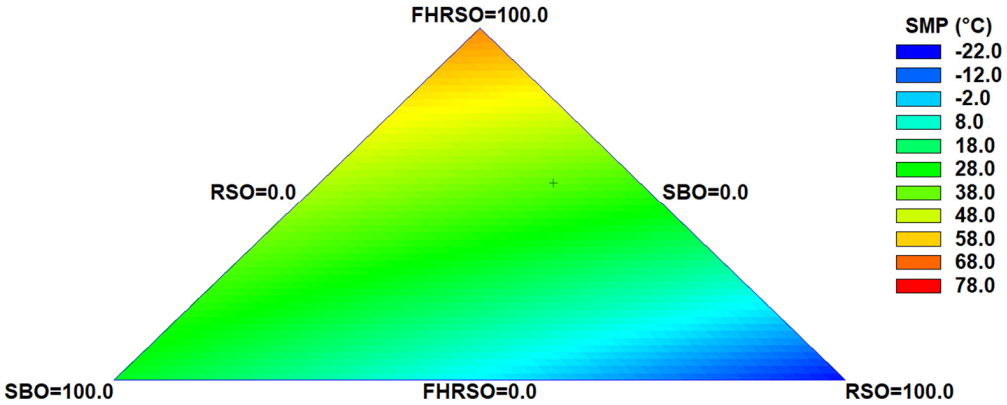


Figure 3. Contour plot showing the effect of the ternary blend composition on the slip melting point (SMP, °C).

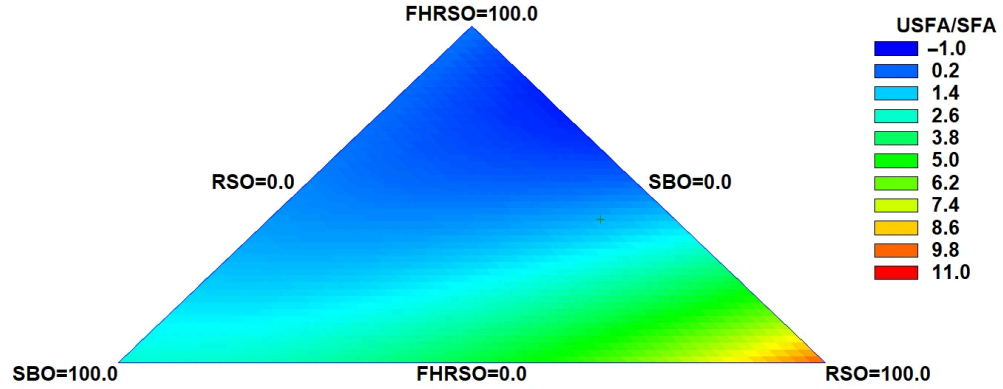


Figure 4. Contour plot showing the effect of the ternary blend composition on the unsaturated to saturated fatty acid ratio (USFA/SFA).

The simplex-lattice design effectively captured the interactions between FHRSO, SBO, and RSO. The applied regression models provided a robust fit (R^2 > 97%). While the special cubic models effectively captured the complex nonlinear interactions for the chemical parameters (IV and USFA/SFA), the quadratic model accurately described the thermal behavior (SMP) within the solid fat domain. This approach successfully avoided the mathematical overfitting and artificial ternary interactions that higher-order models tend to generate when modeling physical phase transitions [28]. Despite the constrained number of design points, the high R^2 values and low *p*-values in the ANOVA confirm the model's reliability for response prediction within the studied experimental domain, which is con-

sistent with the application of mixture modeling in fat formulations [36]. The contour plots (Figures 2–4) showed that FHRSO dominates the increase in SMP due to its high SFA content (95.5%), whereas SBO and RSO elevate IV and the USFA/SFA ratio due to their PUFA-rich profiles (70.7% and 90.2% USFA, respectively), facilitating the creation of a balanced specialty fat blend with properties mimicking non-tempered non-lauric types [31].

The optimized blend (57% FHRSO, 9.5% SBO, 33.5% RSO) achieved the targeted parameters with a low TFA content (<1%), complying with the TR CU 024/2011 requirements and outperforming traditional specialty confectionery fats based on partial hydrogenation, where TFA contents often exceed 2% [9]. The inclusion of 9.5% SBO provides specific monounsaturated fatty acids (e.g., palmitoleic acid constitutes 45.8% in SBO), contributing to the compositional profile compared to conventional palm oil-based blends [5,29]. This contrasts with specialty confectionery fats described in the literature, such as mahua oil and palm stearin blends [3] or bambangan kernel fat blends [37], which lack these specific monounsaturated fatty acids and rely on imported oils. While the utilization of local FHRSO and RSO mitigates supply chain risks in landlocked regions like Kazakhstan [4], the primary objective of this formulation was achieving a lipid composition with specific target characteristics. The applied graphical optimization ensured a multi-objective balance [11], introducing a novel approach by incorporating SBO for specialty fat blends. Further validation through sensory and shelf-life studies could substantiate the applicability of this method in the production of premium confectionery products.

3.5. Validation of the Optimized Fat Blend by Experimental Studies

The experimentally prepared optimized fat blend (FHRSO/SBO/RSO, 57.0/9.5/33.5 w/w/w) exhibited physicochemical characteristics that were statistically compared with the model predictions to validate the adequacy of the simplex-lattice design (Table 4). The experimental slip melting point (SMP) was 36.3 ± 0.3 °C demonstrating consistent alignment with the revised predicted value of 36.56 °C (a relative error of less than 1%). The experimental USFA/SFA ratio was 0.96 (based on SFA = 50.9% and USFA = 49.1%), closely matching the predicted target of 1.0. Furthermore, the experimental iodine value (IV) was 47.1 ± 0.5 g I_2/100 g, which aligned well with the theoretically predicted value of 47.8 g I_2/100 g. This verifies the high precision of the applied regression models and confirms theoretical mass-balance additivity. This thermal behavior is further corroborated by the solid fat content (SFC) profile of the optimized mixture, which was determined experimentally and is illustrated in Figure 1, demonstrating a melting dynamic consistent with the measured SMP.

The fatty acid profile of the optimized fat reflected the contributions of the source oils: the high stearic content (C18:0 = 41.4%) was inherited from FHRSO, providing essential structural rigidity, while oleic (C18:1 = 29.6%), linoleic (C18:2 = 10.2%), and linolenic (C18:3 = 4.2%) acids derived from RSO and SBO contributed to improved plasticity and melting behavior [8,20].

The TAG composition of the optimized fat was dominated by SOO (30.5%), SOS (17.5%), and LOS (10.9%), followed by LOO (8.8%), OOO (9.6%), and minor amounts of SOP (1.9%) and SSS (6.0%). Such a distribution reflects the expected complex composition of the interesterified matrix, highlighting the predominant presence of mixed TAG types (SOO, SOS, LOS) alongside a restricted proportion of high-melting trisaturated TAGs (SSS, SSP). Such a TAG distribution is often reported in the literature to be compatible with a narrow melting range, which is generally considered a desirable structural characteristic for specialty confectionery fats.

These findings agree with earlier studies on non-lauric specialty confectionery fats systems developed from hydrogenated and unsaturated oils [11,33,38,39], where the optimal

functionality was achieved when SOS + SOO accounted for 40–50% of the total TAGs. The presence of 17.5% SOS and ≈30% SOO in the optimized blend provides a triacylglycerol foundation that shares some compositional similarities with natural cocoa butter (which typically contains 60–70% POP/SOS-type TAGs [2,3]), potentially contributing to compatible melting dynamics. The moderate level of SSS ensures mechanical stability while minimizing the risk of a waxy texture or delayed melting upon heating.

Table 4. Experimental characteristics of the optimized fat blend.

Items	Value
Iodine value (I_2/100 g)	47.1 ± 0.5
Slip melting point (°C)	36.3 ± 0.3
Main fatty acids (%)	
C14:0	0.1 ± 0.1
C16:0	7.6 ± 0.3
C16:1	5.0 ± 0.2
C18:0	41.4 ± 0.5
C18:1	29.6 ± 0.4
C18:2	10.2 ± 0.3
C18:3	4.2 ± 0.2
C20:0	0.6 ± 0.1
C22:0	1.2 ± 0.1
TFA	0.9 ± 0.2
SFA	50.9
USFA	49.1
TAG composition (%)	
LOO	8.8
LOP	6.1
OOO	9.6
LOS	10.9
POO	2.6
SOO	30.5
SSL	3.0
SOP	1.9
SOS	17.5
SSS	6.0

Overall, the validated composition confirms that the model-based formulation achieved the targeted physicochemical parameters. The obtained TAG distribution suggests a compositional profile that may be compatible with the structural requirements of a low-trans specialty fat blend. The obtained blend combines the structural properties of FHRSO with the plasticity provided by RSO and the specific compositional contribution of SBO, demonstrating potential as a locally sourced matrix for specialized confectionery fat formulations.

While the current study successfully demonstrates the physicochemical suitability and macro-structural properties of the optimized ternary blend, a limitation of this work is the absence of detailed microstructural characterization and direct instrumental comparisons with control samples. Advanced analyses—particularly X-ray diffraction (XRD) for evaluating specific crystalline polymorphic forms, differential scanning calorimetry (DSC) for precise thermal phase transition mapping, and direct side-by-side experimental comparisons with natural cocoa butter and the previous binary FHRSO/SBO system—fall beyond the scope of this formulation-driven optimization. These critical comparative and microstructural aspects will be investigated in the next phase of our research.

It should be noted that while basic lipid quality and process-control parameters (such as oxidative stability and free fatty acids) were routinely monitored and confirmed to be within normative industry limits for fresh refined blends, their detailed reporting falls outside the primary scope of this manuscript, which is specifically dedicated to the structural optimization and thermal behavior of the ternary matrix.

4. Conclusions

This study addressed its objective of developing a low-trans specialty fat blend by engineering a complex ternary lipid matrix based on local plant oils from Kazakhstan. By utilizing fully hydrogenated rapeseed oil (FHRSO), sea buckthorn oil (SBO), and native rapeseed oil (RSO) through a chemical interesterification process, the inherent structural limitations of simpler binary blends were effectively overcome. Using a simplex-lattice mixture design, the optimal blend formulation was determined to be 57% FHRSO, 9.5% SBO, and 33.5% RSO. The experimental values for this optimal blend demonstrated consistent agreement with the revised theoretical models, displaying high predictive accuracy for all critical physicochemical parameters (SMP = 36.3 °C, IV = 47.1 g I_2/100 g, and USFA/SFA = 0.96).

Experimental validation confirmed the targeted physicochemical characteristics of the optimized fat blend. Regarding regulatory compliance, the measured trans fatty acid (TFA) content in the optimized mixture was only 0.9 ± 0.2%. This result indicates that the blend's measured TFA content is below the 2% threshold stipulated by the Technical Regulation of the Customs Union (TR CU 024/2011), supporting its potential for compliant industrial food applications. In terms of functionality, the measured slip melting point (SMP) was 36.3 ± 0.3 °C. This value, being close to human body temperature, provides a suitable final melting temperature that aligns with the general thermal requirements for confectionery fat systems. Furthermore, the inclusion of 9.5% SBO in the formulation provided a specific compositional contribution, notably through the presence of palmitoleic acid (5.0 ± 0.2%).

In summary, the developed ternary blend based on local Kazakhstani oils provides a lipid composition with specific target characteristics, representing a potential high-quality alternative matrix for specialized confectionery fats.

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Abbreviations

The following abbreviations are used in this manuscript:

CBR	Cocoa-butter replacer
ECN	Equivalent carbon number
FAME	Fatty acid methyl esters
FHRSO	Fully hydrogenated rapeseed oil
FTIR-ATR	Fourier transform infrared spectroscopy with attenuated total reflectance
IV	Iodine value
RSO	Rapeseed oil
SBO	Sea buckthorn oil
SFA	Saturated fatty acids
SMP	Slip melting point
TAG	Triacylglycerol
TFA	Trans fatty acids
USFA	Unsaturated fatty acids

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