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Physicochemical properties of cocoa butter replacers from supercritical carbon dioxide extracted mango seed fat and palm oil mid-fraction blends

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Abstract

Supercritical carbon dioxide ($scCO_2$) extracted mango seed fat (MSF) was blended with palm oil mid-fraction (POMF) to obtain cocoa butter replacers (CBRs). The fatty acid constituents and physicochemical properties of the formulated blends were analysed by gas chromatography (GC). In this study, the fatty acid constituents and other physicochemical properties such as iodine value (43.2 to 43.4 g I_2/100 g fat), saponification value (195.7 to 195.9 mg KOH/g fat), acid value (2.1 to 2.7%), and slip melting point (33.8 to 34.9°C) of blends MSF/POMF at ratios 85/15, 80/20, 75/25, and 70/30 were found similar to the physicochemical properties of commercial cocoa butter. Thus, it could be concluded that the MSF/POMF blends that are blends 85/15, 80/20, 75/25, and 70/30 (3 to 6) could be suggested as CBRs in terms of the physicochemical properties like fatty acid constituents, iodine, saponification and acid values and slip melting point.

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Introduction

Mango grows in tropical and subtropical regions of the world and about 27 million tons of this fruit are produced annually (Iqbal *et al*., 2009). The production of mango fruit is increasing by the day and commercially cultivated over 103 countries (Vasanthaiah *et al*., 2007). Mango fruit and its processed form become an essential part of our diet (Tharanathan *et al*., 2007). The use of mango seed fat for the preparation of cocoa butter alternatives is very interesting since mango is an popular tropical fruits consumed all over the world. Meanwhile, mango seed fat has been frequently investigated (Lipp and Anklam, 1998; Jahurul *et al*., 2014a, 2014b, 2014c, 2015), given the interesting physicochemical properties similar to that of commercial cocoa butter.

Fat from mango seed, like other vegetable fats, are mainly constituted of palmitic, stearic and oleic acids (Jahurul *et al*., 2013). As demonstrated elsewhere (Jahurul *et al*., 2014b, 2014c), the properties of mango seed fat can be modified by blending, producing cocoa butter replacers. Recently, food engineering has made it possible to produce new lines of cocoa butter substitute by blending and fractionation of fats from various sources (Md Ali, 1996; Undurraga *et al*., 2001; Calliauw *et al*., 2005; Wang *et al*., 2006; Tchobo *et al*., 2009; Ciftci *et al*., 2009). Amongst such lines, mango seed fat is of special interest due to its high palmitic (3 to 18%), stearic and levels of oleic acids (34 to 56%) content (Solís-Fuentes and Durán-de-Bazúa, 2011; Jahurul *et al*., 2013). These fatty acid compositions make it possible to use as a source of confectionary formulations, particularly

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in chocolate industry. However, MSF alone is not enough for these applications, it is necessary to blends with other fats like POMF.

This research is to understand the effect of blending on the physicochemical properties of CBR. The MSF and POMF blends may be referred natural products as MSF extracted by food grade supercritical carbon dioxide. The aim of the present study is to investigate the fatty acid compositions, iodine value, slip melting point (SMP), saponification value, and acid value of the developed CBRs. We also present correlations between the fatty acid constituents and their properties.

Materials and Methods

Materials

Mango seed fat (waterlily) was extracted using $scCO_2$. The fatty acid constituents of MSF were described in our previous report (Jahurul *et al*., 2014a). In this study, the $scCO_2$ extracted MSF and POMF were used as a blending components. POMF was obtained from Sime Darby Research Sdn Bhd, Malaysia. All other chemicals and standards were obtained from Malaysia.

Supercritical fluid extraction

The detail procedure of $scCO_2$ extraction process was designated previously (Jahurul *et al*., 2014a).

Blending of MSF and POMF

In this study, total ten blends at various ratios of MSF/POMF, 95/5 (blend 1), 90/10 (blend 2), 85/15 (blend 3), 80/20 (blend 4), 75/25 (blend 5), 70/30 (blend 6), 65/35 (blend 7), 60/40 (blend 8), 55/45 (blend 9), 50/50 (blend 10) were introduced. All the blends were melted at 90°C using thermostatic water bath.

Determination of physicochemical properties

According to method describes by American Oil Chemists' Society (AOCS, 2003) and Jahurul *et al*. (2014a) were used to determine the fatty acids, slip melting point (SMP), saponification value, iodine value, and acid value.

Statistical analysis

In this study, the analyses were carried out in triplicates. ANOVA was used to test the differences between different fatty acids, acid value, iodine value and saponification value in different blends.

Results and Discussion

Fatty acid compositions

Table 1 shows the fatty acids of the blends, and commercial cocoa butter. Figure 1 shows some of the GC chromatograms of certain blends. For all blends, the fatty acid constituents such as $C_{12:0,}$ $C_{14:0}$, $C_{20:0}$, and $C_{22:0}$ were comparable to that of commercial cocoa

Table 1. Fatty acid compositions (area %) of scCO2 extracted MSF and POMF blends at various ratios 5 to 50% of POMF, and commercial cocoa butter

Blends number	Fatty acid composition								
	C_{12}	C_{14}	C_{16}	$C_{18:0}$	$C_{18:1}$	$C_{18:2}$	$C_{18:3}$	C_{20}	C_{22}
1	Tr^{ace}	0.15^{a}	11.35± 0.05^{a}	39.61± 0.07^{a}	41.02± 0.1^{a}	5.09± 0.01^{a}	0.53^{a}	1.63± 0.01^{a}	0.34^{a}
2	0.04^{a}	0.17^{a}	13.68± 0.08^{b}	37.22± 0.09^{b}	41.02± 0.1^{a}	5.25± 0.01^{b}	0.45^{a}	1.59± 0.01^{a}	0.31^{a}
3	0.05^{a}	0.22^{a}	16.35± 0.06^{c}	34.89± 0.11^{c}	40.68± 0.01^{b}	5.41± 0.01^{c}	0.38^{b}	1.47± 0.01^{b}	0.31^{a}
4	0.05^{a}	0.28^{a}	17.97± 0.09^{d}	33.30± 0.16^{d}	40.55± 0.07^{b}	5.45± 0.01^{c}	0.43^{b}	1.44± 0.02^{b}	0.29^{a}
5	0.06^{b}	0.34^{b}	21.54± 0.12^{e}	30.31± 0.08^{e}	39.95± 0.09^{c}	5.57± 0.01^{c}	0.40^{b}	1.35± 0.01^{b}	0.26^{a}
6	0.07^{b}	0.41^{b}	23.32± 0.1^{f}	28.58± 0.06^{f}	39.85± 0.07^{c}	5.68± 0.01^{c}	0.38^{b}	1.26± 0.03^{c}	0.24^{b}
7	0.07^{b}	0.43^{b}	25.18± 0.09^{g}	26.67± 0.1^{g}	39.89± 0.09^{c}	5.85± 0.01^{c}	0.32^{b}	1.16± 0.01^{c}	0.24^{b}
8	0.08^{c}	0.48^{b}	27.39± 0.15^{h}	24.67± 0.14^{h}	39.67± 0.1^{c}	5.96^{d}	0.29^{c}	1.06± 0.01^{d}	0.22^{b}
9	0.10^{d}	0.54^{c}	28.97± 0.13^{i}	23.38± 0.09^{i}	39.41± 0.08^{c}	5.97^{d}	0.26^{c}	1.00^{d}	0.21^{b}
10	0.11^{d}	0.59^{c}	29.66± 0.11^{j}	23.22± 0.06^{i}	38.98± 0.11^{d}	5.96± 0.01^{d}	0.23^{c}	0.97^{d}	0.16^{b}
MSF	0.01	0.05	7.71	42.27	41.41	5.09	0.62	1.78	0.38
POMF	0.20	1.01	50.66	4.42	36.43	6.86	0.31	0.05	0.06
Cocoa butter[a,b,c]	Tr^{ace}	0.7	25.2	35.2	35.2	3.2	0.2	1.2	0.2

Mean value ± standard deviation of three replications with different superscript letter within the same column indicate significant differences ($p<0.05$).

[a]Kheiri (1982); [b]Pease (1985); [c]Lipp and Anklam (1998)

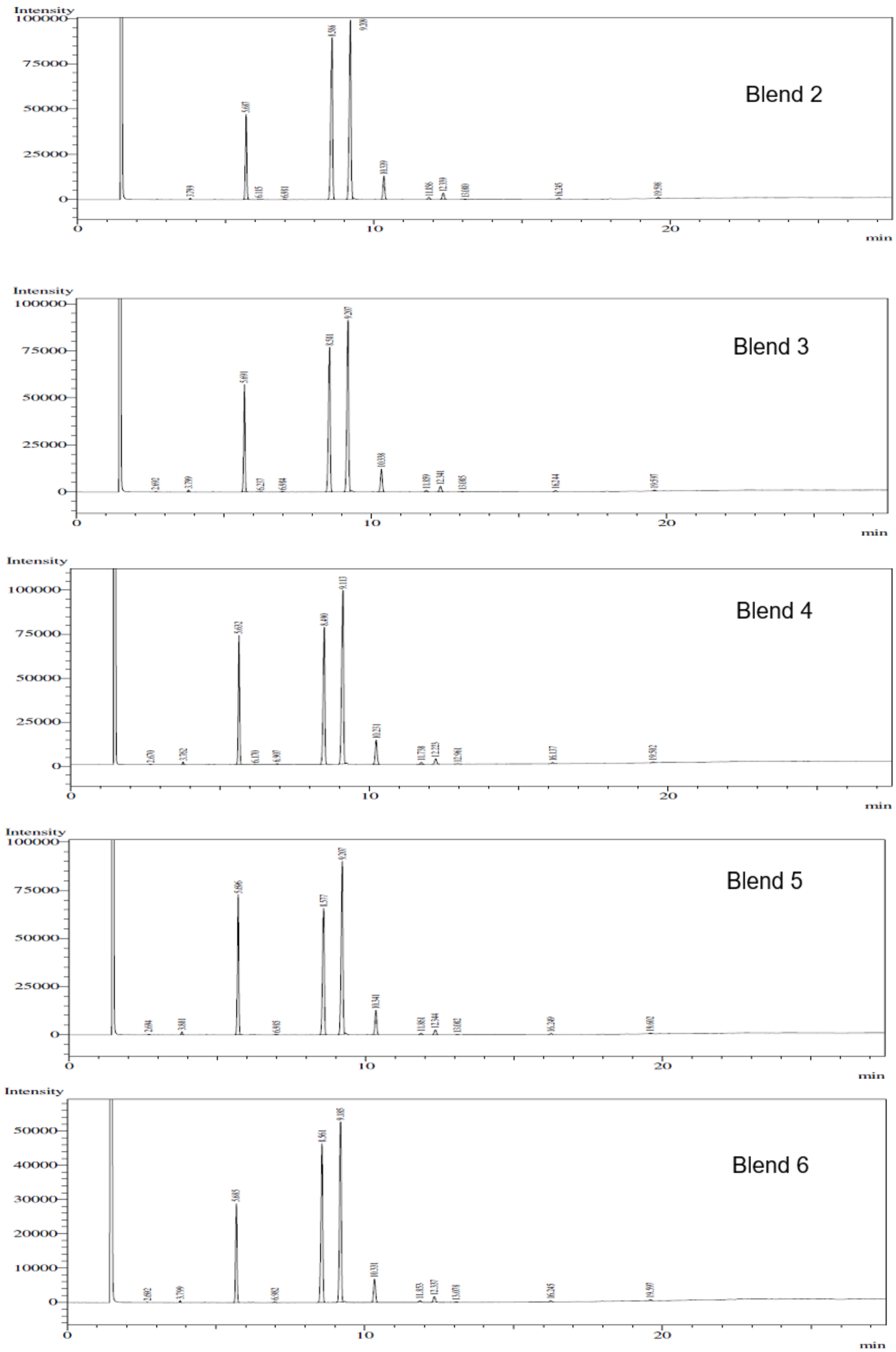

Figure 1. GC chromatogram of fatty acid methyl esters of MSF and POMF blends at various ratios 10 to 30% of POMF.

butter. In particular, the amounts of these fatty acid constituents in blends 3 to 6 were almost similar to that of commercial cocoa butter. The major changes in the fatty acid constituents observed in the blends. These changes of fatty acids could be due to the blending of MSF with POMF. For example, the $C_{16:0}$ content increased from 11.3 to 21.5% in the blends 1 and 5, whereas the $C_{18:0}$ was decreased from 39.6 to 30.3%.

The $C_{18:1}$ constitute diluted from 41.0 to 39.9%, while $C_{18:2}$ increased from 5.1 to 5.6%. Results showed a gradual increase in the content of $C_{16:0}$ as a function of increase in the ratio of POMF. The constituents of $C_{16:0}$ of each blend increased up to 2.5% or more due to every 5% addition of POMF. Contrary trends were observed where the level of $C_{18:0}$ and $C_{18:1}$ decreased within a narrow range. As the longer chain fatty acids increased and shorter chain fatty acids decreased, it was not necessary to further blend the MSF more than 85%. Our results are in line with the results of Jahurul *et al*. (2014c), Kang *et al*. (2013), Ramadan and Wahdan (2012), Salas *et al*. (2011), Anwar *et al*. (2007), Mariod *et al*. (2005), Shukla (2005), who reported the modification of fatty acid after blending of fats from various sources.

Physicochemical properties

The physicochemical properties of commercial cocoa butter and blend of MSF and POMF and are shown in Table 2.

Iodine value

The iodine values determined for all blends varied from 43.0 to 43.7 g iodine/100 g fat when mango seed fat and POMF were used as blending components. Table 2 shows that the iodine value of blend 1 was lower, while it was higher with a small extent in the blend 10. The lower iodine value found in blend 1 could be due to the higher amounts of saturated fatty acids in particular, C18:0 constituent exists in blend 1. On the other hand, the higher iodine value found in blend 10 presumably was due to the higher amounts of unsaturated fatty acids present in that blend. Although, higher and lower values were obtained in blends 1 and 10, it was acceptable to that of commercial cocoa butter.

Zaidul *et al*. (2007) developed CBRs by blending fractionated palm kernel oil and palm oil and observed that the iodine values ranged from 32.8 to 42.1 (g I_2/100 g fat). The authors also claimed that the iodine values of all blends were close to that of commercial cocoa butter. In another study, Jahurul *et al*. (2014c) formulated CBRs by blending of mango seed fat and palm stearin. The authors reported that the iodine values range from 40.3 to 42.7(g I_2/100 g fat). Chaiseri and Dimick (1989) found wide ranges of iodine value (34.4 to 38.65 g I_2/100 g fat) in various cocoa butter. Our results were in good agreement with the results reported by Jahurul *et al*. (2013; 2014b, c), Zaidul *et al*. (2007), Shukla (2005), and Chaiseri and Dimick (1989).

Saponification value

The saponification values ranged from 195.5 to 196.2 (mg KOH/g fat) (Table 2). Variation of results within a narrow range was observed among all blends.

Table 2. Iodine value, slip melting point, saponification value, and acid value determined (referred to sc-co_2 extracted MSF and POMF as a blending components) for all blends and commercial cocoa butter

Blend number	Iodine value (g I_2/100 g fat)	Slip melting point (°C)	Saponification value (mg KOH/g fat)	Acid value (%)
1	43.0±1.0^{a}	35.8±0.21^{a}	195.5±0.05^{a}	3.1±0.11^{a}
2	43.1±0.56^{a}	35.4±0.17^{b}	195.6±0.10^{a}	2.9±0.08^{a}
3	43.2±0.71^{a}	34.9±0.30^{c}	195.7±0.09^{a}	2.7±0.17^{b}
4	43.2±0.44^{a}	34.6±0.41^{c}	195.7±0.28^{a}	2.5±0.21^{b}
5	43.3±0.81^{b}	34.2±0.08^{d}	195.8±0.12^{b}	2.4±0.09^{b}
6	43.4±0.62^{b}	33.8±0.11^{d}	195.9±0.07^{b}	2.1±0.13^{c}
7	43.5±0.90^{b}	33.4±0.36^{e}	195.9±0.09^{b}	1.8±0.10^{c}
8	43.5±0.78^{b}	32.9±0.28^{e}	196.0±0.15^{b}	1.6±0.07^{d}
9	43.6±1.0^{c}	32.5±0.09^{f}	196.0±0.19^{b}	1.4±0.12^{d}
10	43.7±0.55^{c}	32.2±0.41^{f}	196.1±0.06^{b}	1.1±0.20^{e}
MSF	42.9±0.82	36.2±0.32	195.5±0.08	3.23±0.23
POMF	44.71±0.75	28.1±0.48	196.7±0.22	0.05
Cocoa butter	37.91	35.02	193.5-196.71	0.4-3.11

Mean value ± standard deviation of three replications with different superscript letter within the same column indicate significant differences ($p<0.05$).

[1]Chaiseri and Dimick (1989); 2Kheiri (1982)

Table 3. Multiple regression coefficients of fatty acid constituents of MSF: POMF blends, E is constant, and R^2 is coefficient determination

Properties	A (C_{14})	B (C_{16})	C ($C_{18:0}$)	D ($C_{18:1}$)	E ($C_{18:2}$)	F ($C_{18:3}$)	G (C_{20})	H (C_{22})	I (constant)	R^2
Iodine value	3.8099	1.8745	1.8609	1.9512	2.0730	0.5775	3.1215	2.1087	-149.263	0.9955
Slip melting point	-5.4080	-2.6452	-2.4551	-3.1334	-0.3805	0.3536	-1.3361	-0.8694	296.6273	0.9993
Saponification value	-2.7750	-3.5312	-3.3896	-3.9288	-1.5784	-3.1258	-3.8811	-4.3295	548.9074	0.9999
Acid value	-9.74	-5.9061	-5.8179	-6.6523	-4.9075	-3.8397	-6.3426	-2.4186	613.0927	0.9999

This was due to the fact that the saponification values of pure mango seed fat and POMF were very close to each other. However, most of the results found in this study were closer to that of commercial cocoa butter. As shown in Table 2, results were getting closer when POMF was increased in the blends to that of commercial cocoa butter. It was true for all blends. Recently, Jahurul *et al*. (2014c) reported the range of saponification value (195.5 to196.2 mg KOH/g fat) for MSF and palm stearin blends. The values found in this study were similar to the values reported by Jahurul *et al*. (2014c), Manifie (1999) and Chaiseri and Dimick (1989).

Slip melting point (SMP)

The SMP ranges for blends 1 to 10 were found to be from 32.2 to 35.8°C that were closer to that of commercial cocoa butter. Moreover, there were no significant differences observed in SMP among the blends when MSF blended with POMF. The SMP of mango seed fat (36.2°C) exhibited higher than the SMP of POMF (28.1°C). Therefore, SMP decreased as the POMF were increased. As shown in Table 2, blends no. 1 to 6 were found to be the best blends for SMP compared with commercial CB. Zaidul *et al*. (2007) studied SMP of different blends of fractionated palm kernel oil and palm oil and reported that it ranged from 32.8 to 42.1°C. They also compared their results with commercial cocoa butter and claimed that the SMP for all blends were closer to that of commercial cocoa butter.

Acid value

Acid values of the commercial cocoa butter, POMF, pure MSF and blends of MSF and POMF are presented in Table 2. The acid values for all blends ranged from 1.1 to 3.1%. The acid value of the blend 10 was lowest, while it was highest in blend 1. Due to the blending ratios (MSF/POMF), various acid values were observed in the present study. Chaiseri and Dimick (1989) reported that the acid values for commercial cocoa butter cultivated in different region varied from 0.42 to 3.11%. The results obtained in this study were closed with the results of commercial cocoa butter.

Correlation between the fatty acid constituents of all blends and their properties by multiple regression equation

It was necessary to develop such a relation that allows all blends to be studied for suitability as CBRs. In order to make a correlation between the fatty acid constituents of all blends (referred to as MSF with POMF) and their properties such as iodine value, SMP, saponification value and acid value, a multiple linear regression model was applied (Zaidul *et al*. 2007), represented by the following equation.

$$Y = AX_1 + BX_2 + CX_3 + DX_4 + EX_5 + FX_6 + GX_7 + KX_8 + 1 \quad (1)$$

$$Y = \sum_{i=1}^{n} A_i X_i + I \quad (2)$$

where y is the blend properties and x_i is the fatty acid constituents of all blends of MSF with POMF, Ai is regression coefficient of x_i and I is a constant. The 10 blends were taken into account to study the relationship between fatty acid constituents and their properties such as iodine value, slip melting point, saponification value and acid value. The multiple regression coefficient results of fatty acid constituents are shown in Table 3. The coefficients of some fatty acid constituents are negative, showing monotectic relationship between MSF and POMF. The coefficient of fatty acid constituents of all blends revealed a strong correlation among them and their properties, and R^2 varied from 0.9955 to 0.9999.

Figure 2 shows the correlation between fatty acids (referred to Table 1) and their properties (referred to Table 2) for all blends. The results of residuals (differences between calculated and experimental results) for all blends were too small and distributed randomly around the regression line. The residuals ranged from -0.04 to 0.06 for slip melting point followed by -0.02 to 0.03 for iodine value, -0.01 to 0.01 for acid value and from -0.003 to 0.002 for saponification value which was smaller and R^2 found to be 0.9993, 0.9955, 0.9999 and 0.9999. These indicate that the model explained 99.93%, 99.55%, 99.99% and 99.99% of the original variability.

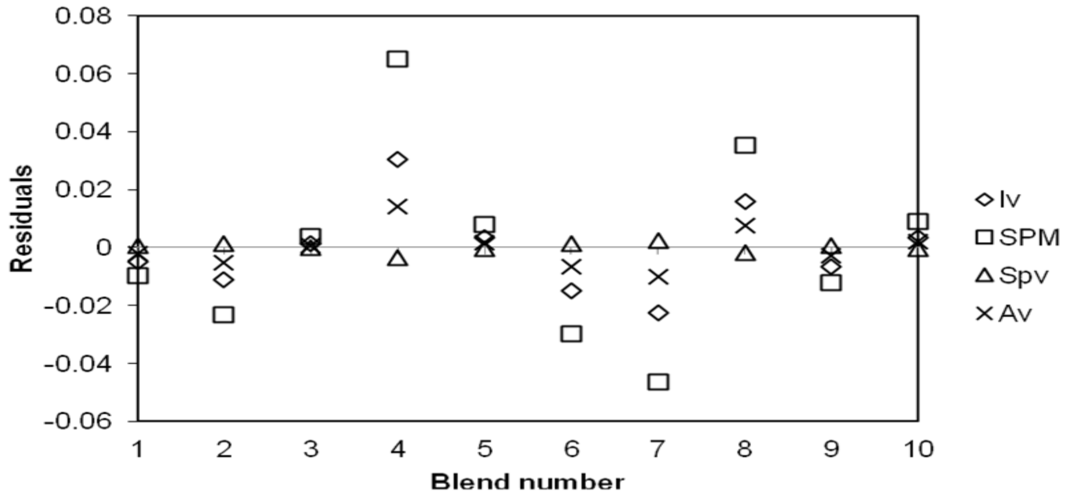

Figure 2. Residual of experimental and calculated properties of blends (referred to Table 2) versus fatty acid constituents of all blends (referred to Table 1).

Conclusion

MSF and POMF were used in order to prepare a new CBR blend which offers physicochemical properties within the limits of the commercial cocoa butter standards. The key physicochemical properties of the formulated CBRs were measured by the standard test methods. The fatty acid constituents in terms of triglycerides of blends 1 to 10 were correlated with their physicochemical properties and multiple regression equation was developed where strong correlations were found among fatty acid constituents and their properties. The iodine value (43.2 to 43.4 g I_2/100 g fat), saponification value (195.7 to 195.9 mg KOH/g fat), acid value (2.1 to 2.7%), and slip melting point (33.8 to 34.9°C) of certain blends were found similar to the commercial cocoa butter. The $scCO_2$ method gives efficient and natural method for producing chemical free new CBRs that have excellent properties similar to that of commercial cocoa butter. We propose new CBRs, with properties within the limits of the commercial cocoa butter, with the following composition: 70 to 85% MSF and 15 to 30% POMF.

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