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Butter Separation from Cream Using Ultrasonication: Optimization of Parameters Using RSM

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ABSTRACT: Butter is separated from cream using ultrasonication method. The efficiency of butter preparation is evaluated with respect to various parameters such as time, dilution, fermentation time using ultrasonication at room temperature and compares the yield with conventional stirring method. All the parameters were critically studied and it is observed maximum yield was obtained for sonication time at 30 min, 1:10 dilution and the fermentation time of 18h. Experimental parameters for butter separation have been optimized for maximum yield of butter using Response Surface Methodology. It has been observed from the present analysis that the predicted values are in good agreement with the experimental data with a correlation coefficient of 0.956. It is also observed that the butter prepared from ultrasonication method produce higher yield, lesser time to produce butter and homogenized butter grains obtained compared to conventional method. The viscosity and pH are same when compared to commercial butter and having the fat content of 83.14 %.

Keywords: *Butter Yield, Cream, Response Surface Methodology, Separation, Ultrasound*.

Introduction

Milk is an essential food for human health. It contains proteins, lipids, lactose, vitamins and minerals. There are various other dairy products can be obtained from processing milk. The milk is separated into cream and skim milk, and then standardized to achieve the various desired dairy products. Butter is a high energy concentrated natural dairy product consisting mainly of milk fat, water and non-fat solids. Butter is produced by a mechanical phase inversion of cream. (Bermúdez-Aguirre *et al*., 2008; Goudédranche *et al*., 2000; Juliano *et al*., 2013)

Generally in industrial scale butter is separated from cream using paddle type stirrer both in batch and continuous method. Usually cream is loaded to churner and is churned in different stages. In churning, air bubbles are formed and surrounded by a fat globule. Due to continuous shearing, the milk fat globules will collapse and larger aggregates of fat are formed known as butter grains (Leong *et al*., 2016; Rønholt *et al*., 2014). Natural creaming of fat is influenced by agglutination process. Agglutination is the process where immune globulins present in milk will promote the flocculation of globules as they come into contact with one another at low temperature, enhancing their effective size. But at high temperature, as the agglutinin detaches from the surface of globules and possibly even denatures (Leong *et al.*, 2014). Industries may operate at a low temperature to reduce free fat loss into the butter milk, hence higher separation efficiency is achieved by decreasing the temperature.

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Ultrasonication is widely used in food industry, for instance in processes such as emulsification (Shanmugam *et al.*, 2014), crystallization (Martini *et al*., 2008), creaming (Juliano *et al*., 2012), extraction (Karageorgou *et al.,*, 2014), cutting (Schneider *et al*. 2009), fermentation (Nguyen *et al*., 2012), homogenization (Wu *et al*., 2000), hydrolysis (Uluko *et al*., 2013), ultrafiltration (Muthukumaran *et al*., 2005), reducing the allergenicity of milk proteins (Tammineedi *et al*. 2013), and decreasing viscosity (Zisu *et al*., 2013).It has many advantages over conventional techniques, ultrasound at lower power higher frequencies causes vibrations in the molecules and has no effects on mechanical or chemical properties of the materials (Ashokkumar *et al*., 2008). The ultrasound method has potential benefits to dairy process industry such as higher product yields, shorter processing time, reduced operating and maintenance costs, improved taste, textures, flavor, color and reduction of pathogens at lower temperature (Chemat *et al.,* 2011).

High power Low frequency (20 kHz to 10 MHz); Low power high frequency (2–10 MHz) ultrasounds are used in many applications. Higher power at lower frequencies, which is referred to as “power ultrasound” has the ability to cause cavitations and low power ultrasound does not causes any physical or chemical alterations in the properties of the material is used in non-destructive applications; providing information about the physicochemical properties of foods, such as composition, structure etc. The high frequency ultrasound has capability to initiate separation of fat globules. During high frequency separation, the physical effects of unstable cavitations are negligible and therefore will not affect the integrity of milk fat globules. Application of ultrasound within 20 kHz to 1 MHz has no evidence of damage to the structural integrity (Chandrapala *et al.*, 2011). Leong et al. 2014 studied separation of fat from natural milk using sonication. Ultrasound fat separation rate is many times faster than natural creaming process. Ashokkumar *et al*. (2008) presented recent developments in high and low frequency ultrasonic processing of food and dairy systems. They reported that, fat separation from milk and viscosity modification in starch systems, nutritional food emulsions are favorable at higher frequency; viscosity modification and encapsulation of nutrients is favorable at low frequency ultrasound. Juliano *et al.* (2013) studied the effect of high frequency ultrasound (0.4–3 MHz) on milk fat separation from a recombined milk emulsion using industrial scale transducers. The authors reported that higher separation rate was obtained at sonicating frequency of 0.4 MHz in a vertical perpendicular double transducer setup. Liu *et al*. (2015) improved the renneting properties of milk by applying 20 kHz ultrasound at 30°C and pH 6.7. Improving renneting properties of milk is essential for cheese manufacturing. The author observed that ultrasonication of renneting property of milk is more efficient for manufacturing of cheese. Shanmugam *et al*. (2012) treated pasteurized homogenized skim milk using 20 kHz ultrasound under controlled temperature. The author reported whey protein in the milk were denatured and formed soluble whey-whey aggregates during the initial 30 min of prolonged sonication resulted in partial disruption of some whey proteins from these aggregates. Suh *et al*. (2012) optimized the manufacturing condition of spreadable modified butter by RSM. It is reported that the solid fat content value at 10 °C is more suitable indicator for the manufacturing spreadable modified butter. Ajala *et al*. (2016) optimized the operating variables for the better yield of shea butter extraction using RSM. The phyisco chemical properties for the sample also studied and reported.

From the literature it is evident that ultrasonication process widely used in the food processing application. The aims of the present work is to use ultrasonication technique to separate the butter from the cream and optimize the variables such as time, dilution and fermentation time to get better butter yield using Response Surface Methodology. Further the characteristics of the butter also been evaluated and reported.

- *Mechanism of butter separation using sonication*

Butter is manufactured by churning a cream at particular time, temperature and dilution. They include three stages:

1. Breaking of cream is observed.
2. Small globules adhere with each other to form size of pea.
3. A big mass of butter is formed.

The action is due to cavitation, which generates high shear force and microbubbles that enhances surface erosion, fragmentation and mass transfer resulting in high yield of extracted materials and fast rate of extraction.

Materials and Methods

- *Cream*

The cream is purchased from nearby supermarket. The composition of the 100g of cream consist of fat 40%, energy 381cal, carbohydrate 3.2g, protein 2g, calcium 40.6 mg, sodium 14.2 g. It is stored under refrigerator condition. For each experiment the cream is taken out from the refrigerator and the experiments are performed.

- *Experiment*

Bath type ultrasonicator used for the present experiment. Bath type sonicator having a stainless steel cylindrical jacket equipped with in-built piezoelectric transducer placed at the rectangular bottom and an external generator is used. The sonicator has constant frequency of 36 kHz and the ultrasonic time can be adjusted by the button provided in the sonicator. The sonicator is operated under the condition where the water is filled up to the mark point mentioned in the sonicator. Experimental are conducted in the magnetic stirrer, consists of hot plate with nob for controlling both the temperature and speed. The speed can be varied from 50rpm to 1000 rpm and the stirrer speed is fixed constant for a set of experiments.

- *Experimental procedure*

The fresh cream is purchased from nearby supermarket and immediately stored in a refrigerator. To conduct the experiment, cream is taken out from the refrigeration and kept outside to bring down to room temperature. A known amount of cream (5g) is taken in the glass beaker and the cream is diluted with distilled water of various dilutions in the ratio 1:5, 1:10, 1:15 and 1:20. After which, the beaker is placed in the constant frequency ultrasonic bath for different sonication time. Each experiment the solid- liquid phases are separated and the yield is calculated. The solid yellow lumps floating on the top is butter and the remaining whitish bottom portion is buttermilk. The butter formed on the top of the layer is scoped out and weighted. The same procedure is followed for stirrer method at the constant speed of 250rpm.

The butter yield is calculated from the following equation

$$Butteryield\ (\%) = \frac{Butterweight}{intialcreamweight} \times 100 \quad (1)$$

- *Response surface method (RSM)*

Response surface method (RSM) is used for modeling and optimization of process in which the experimental output response is influenced by experimental variables. In the present work the experiments are designed using Box-Behnken method of RSM for three factors such as sonication time, dilution ratio and fermentation time with three different levels. Each variable and their levels are sonication time (15, 30 and 45min); dilution (1:5; 1:10; 1:15) and fermentation time (8, 12, 16h) are selected based on the preliminary experiments. The design of experiments for this (Table 1), gave total of 15 runs of experiment out of which three experimental runs are at the centre points. The experiments are conducted at room temperature. The response is measured as percentage yield of butter and the results are analyzed using MINITAB 14 software tool.

Results and Discussion

- *Effect of Sonication Time, Dilution and Fermentation Time for Butter Yield*

The effect of sonication and stirring time (15 and 30 min) on different cream dilutions for butter separation is shown in the Figure 1(a) and 1(b). It is noticed from the figure, butter yield is higher for the ultrasonication process compared to stirring process. This is due to the shear forces in ultrasonication which enhances the rate of mass transfer compared to stirring process. The Cavitation creates the formation of micro jets rapidly and breaks the cell wall of cream which leads to the effective release of cell content thus there is higher butter formation in the process. It is also noticed from figure that the 30min of sonication time yield 40.33%. During the initial 30 min the better butter formation is observed and prolonged sonication resulted in partial disruption of some fat globules from these aggregates. The addition of water to the cream greatly reduces the concentration of cream, without affecting the fat globules stability. Further increasing the dilution of the cream, above 1:10 the butter formation reduces, due to more dilution makes difficult to form butter in the ultrasonication process. Increasing the ultrasonication time beyond 30min, the butter separation efficiency decreases. The effect of ultrasonication time on various fermentation times of 6h, 12h and 18 h on butter yield is shown in the Figure 2. After fermentation, the samples are taken in the sonicator for sonication. It is observed from the figure that the 18hr of fermentation time gives higher butter yield compared to 6 and 12 h. During fermentation the cream naturally sours as bacteria converts milk sugars into lactic acid. In ageing phase, cream is held at cool temperature to crystallize the butterfat globules, ensuring proper churning and texture of the butter. Higher fermentation time is not favoring the formation of butter due to the more bacterial action on the cream.

Table 1. The actual design of experiments and response for butter yield using ultrasonication

S. No	Time (min)	Dilution (v/v)	Fermentation (hr)	Experimental Yield (%)	Predicted Yield (%)
1	15	5	12	26.01	26.51
2	45	5	12	15.86	13.06
3	15	15	12	25.01	27.80
4	45	15	12	17.40	16.89
5	15	10	6	29.16	27.77
6	45	10	6	14.66	16.58
7	15	10	18	38.33	36.41
8	45	10	18	21.83	23.22
9	30	5	6	23.50	24.38
10	30	15	6	28.14	26.73
11	30	5	18	30.40	31.81
12	30	15	18	35.45	34.57
13	30	10	12	29.66	29.66
14	30	10	12	29.65	29.66
15	30	10	12	29.66	29.66

Figure 1(a)

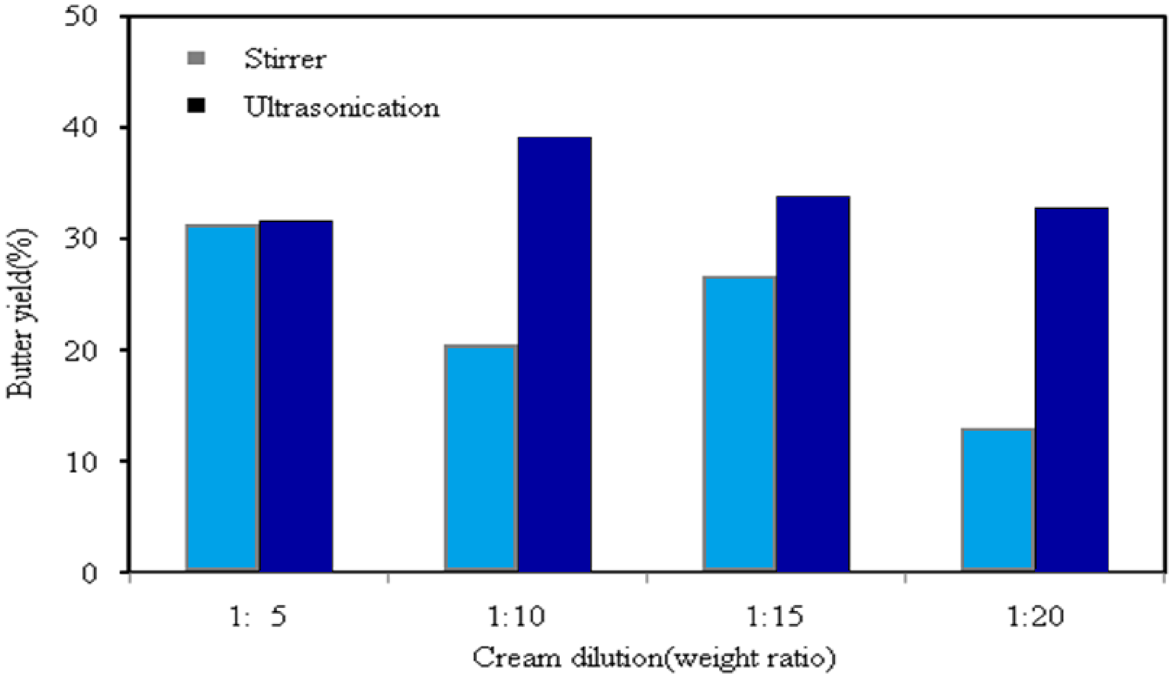

Figure 1(b)

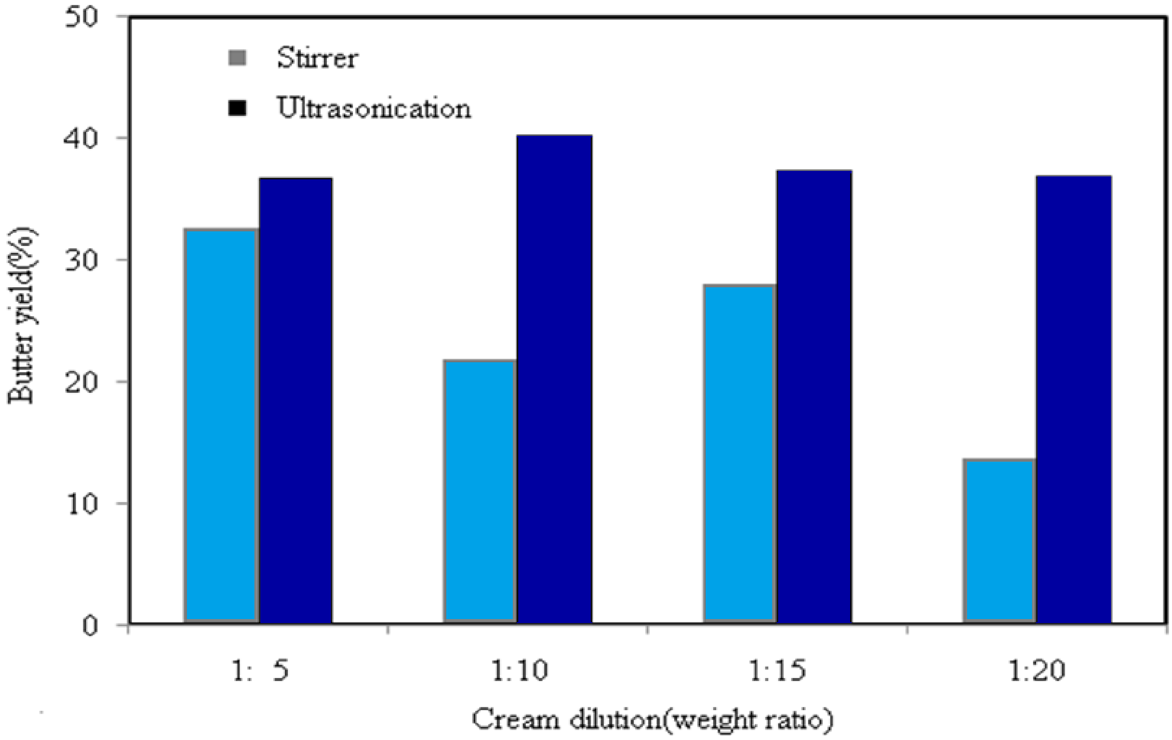

Fig. 1. Effect of Cream dilution on butter yield. (a) At constant stirring; ultrasonication time: 15 min. (b) At constant stirring; ultrasonication time: 30 min

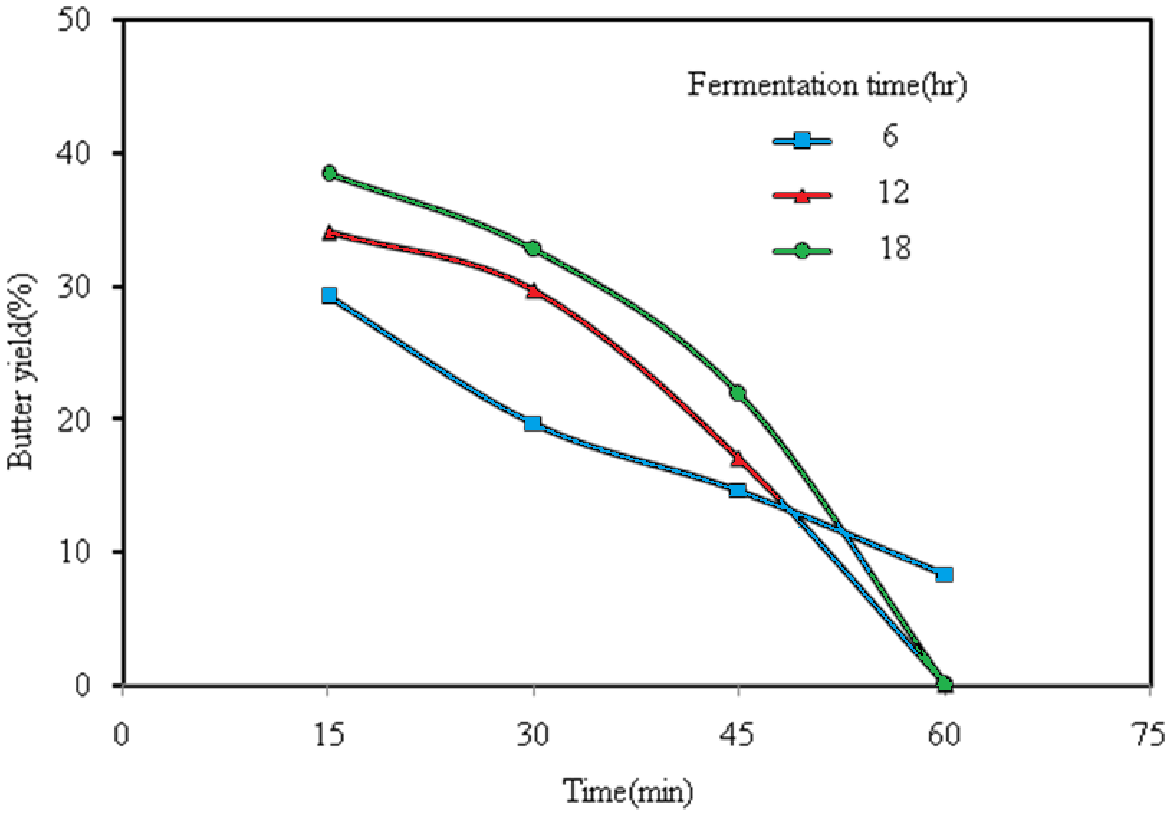

Fig. 2. Effect of fermentation time on ultrasonication separation of butter yield. Cream dilution: 1:10

- *Regression modeling*

The regression model equation for the percentage butter yield with experimental variables such as sonication time, (A); dilution, (B); and fermentation time (C) can be given as

$$\text{Yield (\%)} = 7.505 + 1.17217A + 2.04775B - 0.77854C$$
$$- -0.02661A^2 - 0.10435B^2 + 0.06448C^2 + 0.00847AB$$
$$- 0.00556AC + 0.00342BD \qquad (2)$$

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The prediction of percentage yield using the regression model equation(equ. 2) is also given in Table 1. The regression model predictions satisfactorily matches with the experimenal values with the correlation coefficent of 0.986. The parameters in the equation (2) has been optimized for the maximum percentage yield of butter and the optimized values for 35.8% Sonication time 30min; dilution 1:10 and fermentation time of 18h.

The interaction effect of experimental variables on percentage yield of butter is presented in three dimensional surface plots. The interaction effect of time and dilution; dilution and fermentation time of cream is shown in the figure 3. It is observed from the figure 3(a), at the 30 min of sonication time higher butter yield was observed. If long sonication resulted in disruption of butter occurs. The interaction plot of dilution and fermentation time is shown in the figure 3(b). It is noticed from the figure higher fermentation time shows the higher butter yield. During fermentation the cream naturally sours as bacteria converts milk sugars into lactic acid results increase the butter yield.

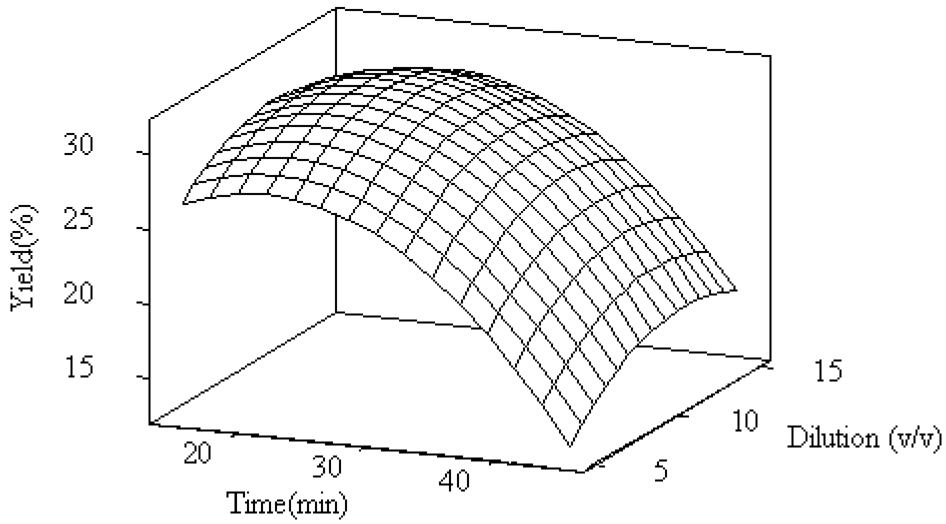

Figure 3(a)

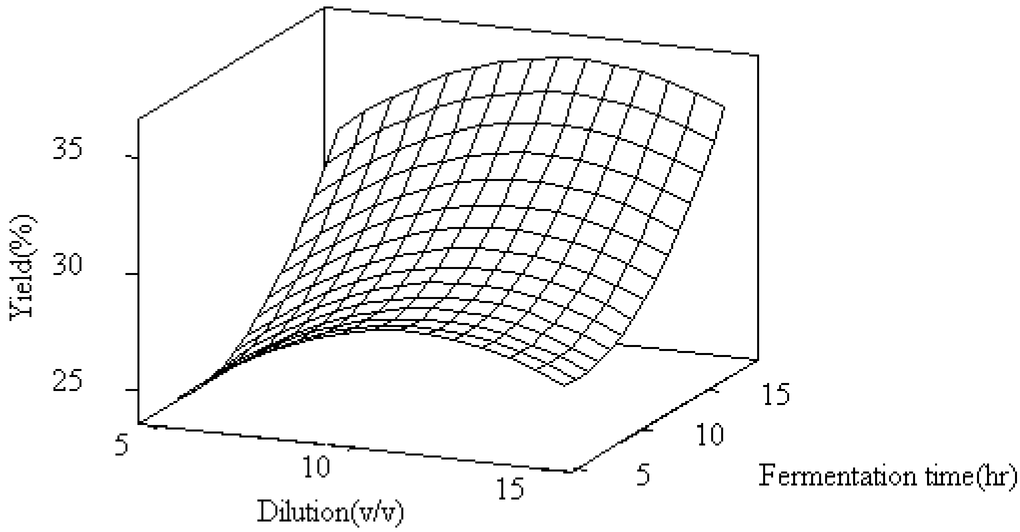

Figure 3(b)

Fig. 3. (a) Surface plot of sonication time and dilution on percentage yield of butter, (b) Surface plot of dilution and fermentation time on percentage yield of butter

- *Statistical Analysis*

The significance of model were analysed using *p*- test and *t*-test. The p-values is used to check the significance and the effect of interaction among the variables. A larger magnitude of t-value and a smaller magnitiute of p-value is the significant term. It is noticed from the table 2, that the linear term (p=0.045) and qudratic term for ultrasonication time (p=0.007) is the significant in the model equation. The other terms are less signifcant (p>0.05) in the table 2. The analysis of variance (ANOVA) for percentage yield of butter using ultrasonication is shown in the Table 3. The significant effects of experimental variables on percentage yield on butter can be determined by ANOVA. The F value for the regression and square terms are higher. The large value of F indicates, most of the deviation in the response can be explained by statistical model. Generally, lesser P value (<0.05) considered to be significant in statistical model. The present model equations are further checked by regression coefficients (R^2 and R^2_{adj}). It can be seen from Table 3 that the values of R^2 and R^2_{adj} are 0.949 and 0.856 indicates that the model is highly significant.

- Characterization of butter

The property of butter such as pH, viscosity, fat content, water content and conductivity are characterized for butter separated from ultrasonication method. The pH value of butter is 6.2, which is nearly similar to pH value of butter obtained from conventional method (pH6-6.4) (handbook of dairy processing 2005). Viscosity is measured using Rheometer and the value is found to be 207 Pa.s and it closely matches with the commercial butter. The fat content was determined by DGHS manual 1, 2005 and it is observed that 83.14% of fat, it is higher compared to conventional butter fat content (80%) (Almanza-Rubio *et al*., 2016). The water content was calculated by heating the sample at 100°C for 2h followed by 30 min cooling at room temperature in an oven. The water content was 16.08% and it was equal to the butter prepared from conventional method. Conductivity was measured during the phase inversion of cream to butter formation. The conductivity value is 0.013µS/cm. In general the conductivity value will be in the range of 0.03 to 0.23µS/cm which confirms the butter formation (Rønholt *et al*., 2012).

Table 2. Estimated regression coefficient and corresponding' and 'p' values for percentage yield of butter

Term	Coefficient	Standard error	t	p
Constant	7.505	12.8045	0.586	0.583
A	1.17217	0.4353	2.693	0.043
B	2.04775	1.306	1.568	0.178
C	-0.77854	1.0883	-0.715	0.506
AA	-0.02661	0.0059	-4.477	0.007
BB	-0.10435	0.0535	-1.951	0.109
CC	0.06448	0.0371	1.736	0.143
AB	0.00847	0.0171	0.494	0.642
AC	-0.00556	0.0143	-0.389	0.713
BC	0.00342	0.0428	0.08	0.94

Table 3. ANOVA results for percentage yield of butter from cream using ultrasonication

Source	DF	Sum of the square	Mean square	F	P
Regression	9	610.968	67.8853	10.28	0.01
Linear	3	426.693	23.2513	3.52	0.104
Square	3	181.62	60.54	9.17	0.018
Interaction	3	2.655	0.885	0.13	0.936
Residual Error	5	33.011	6.6022		
Lack-of-Fit	3	33.011	11.0036		
Pure Error	2	0	0		
Total	14	643.979			

R^2 0.949; R^2_{adj} 0.856

Conclusion

Experiments were carried out to separate butter from cream. The effect of various parameters such as sonication time, cream dilution and fermentation time on butter yield was studied using Response Surface Methodology. It is observed from the result that 30 min of sonication, at 1:10 dilution and 18h of fermentation time gives better butter yield compared to conventional stirring method. Room temperature is more suitable for separation of butter since increase in temperature leads to dispersion of fat globules. The regression model prediction for the butter yield closely matches with the experimental observation with the correlation coefficient of 0.956. The butter properties were analyzed and reported. Thus this method will be helpful in large scale production of butter with more efficient.

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