THE EFFECT OF MIXING TEMPERATURE AND MIXING TIME ON THE PHYSICAL PROPERTIES AND STABILITY OF GREEN TEA EXTRACT EMULGEL

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Abstract

The aims of the research are to determine the significant effect factor among mixing temperature, mixing time and their interaction on the physical properties and physical stability of green tea emulgel. In the beforehand research, the optimation study about composition between emulsifying agent (Tween 80 and Span 80) and gelling agent (Carbopol). This research tried to find the influence of mixing process, focussed on mixing temperature and mixing time using the factorial design with two factors (which are mixing temperature and mixing time) and two levels which are high level (70°C and 20 minutes) and low level level (60°C and 10 minutes). The physical properties dan physical stability parameter that used including viscosity, spreading ability and viscosity shift after storaged in special temperature and humidity (which are 40°C and 75% of relative humidity) for a month.. The results show that there are nothing factor that significant on determining viscosity and mixing time was significant factor on determining the spreadability. In other hand, mixing temperature and interaction between mixing temperature and mixing time were significant factors on determining the viscosity shift.

Keywords : *Green tea extract, mixing temperature, mixing time, emulgel, factorial design*

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INTRODUCTION

Tea is one of the most popular drinking that have been known for many years. People believes it can be used to keep healthy. Green tea is one of the kind of tea which had a different ways to produced. Based on the recent studies, one of the active compound in green tea was cathecin, which have an antioxidant activity (Ahmad et al., 1998). Because of it's function, green tea was formulating in many forms, like a oral supplement or cosmetic. One of them is green tea emulgel.

Emulgel are emulsions, either of the oil-in-water or water-in-oil type, which are gelled by mixing with a gelling agent. The mixing process will affect the characterictic physical properties and stability of the emulgel. Some factors that affect include mixing temperature and mixing time. Temperature of mixing can influence the ratio between internal and external phase, so it can affect the droplet formation in the emulsion. Time of mixing is important from the point of view of (a) ensuring adequate gross mixing, (b) ensuring equilibrium drop-size distribution is achieved and (c) avoidance of overprolonged mixing with its penalties of higher energy costs, capacity or throughput problems, and possible damage to the product (Peter, 1997).

Factorial design are used in experiments where the effects of different factors, or conditions, on experimental result are to be elucidated. Factorial design are the design of choice for simultaneous determination of the effects of several factore and their interactions (Bolton, 1997). The aim of this study was to develop green tea extract emulgel formulations focussed on mixing process, which are mixing temperature and mixing time. The influence of mixing temperature and mixing time was investigated using 2^2 factorial design. As a model, the green tea extract emulgel formulations was used in this study, based on research by Laverius (2011).

Table II. Quantitative composition of green tea extract emulgel formulation

Ingredient (mg)	Formulation			
	1	a	b	ab
Liquid paraffin	25			
Tween 80	5.60			
Span 80	3.75			
Carbopol 3% wt/v	135			
TEA*	2.85			
Green tea extract	0.031			
Propylene glycol	50			
Metyl paraben	0.75			
Propyl paraben	0.75			
Purified water to	500			
	Factors optimated in each formula			
	1	**a**	**b**	**ab**
Mixing temperature (°celcius)	60	70	60	70
Mixing time (minutes)	10	10	20	20

*TEA indicates triethanolamine

METHODS

Materials

The following materials were used in this research : green tea dry extract(*Camellia sinensis* L.) (PhytoLab), Carbopol (Ultrez), TEA (Bratachem), propylene glycol (Bratachem), Tween 80 (Bratachem), Span 80 (Bratachem), liquid paraffin (Bratachem), methyl parabens (Bratachem), propyl parabens (Bratachem), purified water.

Experimental Design

Four green tea extract emulgel formulations were prepared according to a 2^2 factorial design employing the factors and levels shown in Table I. The composition of green tea extract emulgel formulation is shown in Table II. The green tea extract emulgel formula was taken based on study by Laverius (2011).

Table I. Factors and levels for 2^2 factorial design

Levels	Mixing temperature (°Celcius)	Mixing time (minutes)
Low	60	10
High	70	20

Preparation of Green tea Extract Emulgel Formulations

The oil phase of emulsion was prepared by dissolving Span 80 in liquid paraffin, whereas the aqueous phase was prepared by dissolving Tween 80 in a part of purified water. Then the oil phase was added to the aqueous phase (in temperature according to each level, 60°C or 70°C), with continuous stirring (400 rpm) according to each level (10 or 20 minutes). The green tea extract was dissolved in purified water, while methyl and propyl parabens were dissolved in propyleneglycol. The gel system in the formula was prepared by dispersing Carbopol in purified water, then the pH was adjusted to 6-8 using TEA. Then the emulsion was added to the gel with constant stirring, the green tea extract, methyl and propyl parabens were added. Those dispersion was mix with continuous stirring (400 rpm) for 20 minutes (Laverius, 2011).

Physical Properties Examination

The prepared green tea extract emulgel formulations were inspected visually for physical properties, which are viscosity and spreadability. The viscosity of the emulgel after compounded was determined using viscometer VT 04 (Rion™-Japan). The spreadability of the

Table III. Physical properties of green tea extract emulgel

Physical Properties	Formulation			
	1	a	b	ab
Viscosity (dPas)	193.33 ± 5.77	173.33 ± 20.82	196,67 ± 5.77	223.33 ± 46.19
Spreadability (cm)	3.90 ± 0.05	3.83 ± 0.08	3.72 ± 0.19	3.60 ± 0.00

Table IV. Main effect of factors

Respons	Main effect		
	Mixing temperature	Mixing time	Interaction
Viscosity	+6.67	+23.33	+26.67
Spreadability	-0.09	-0.21	-0,02

emulgel was measure using parallel-plate method (Garg et al., 2002).

Physical Stability Study

The samples of green tea extract emulgel formulations were stored in a humidity chamber for a month in condition 75% of RH and 40°C of temperature. Then the samples were tested for their viscosity to know the viscosity shift after stored.

RESULT AND DISCUSSION

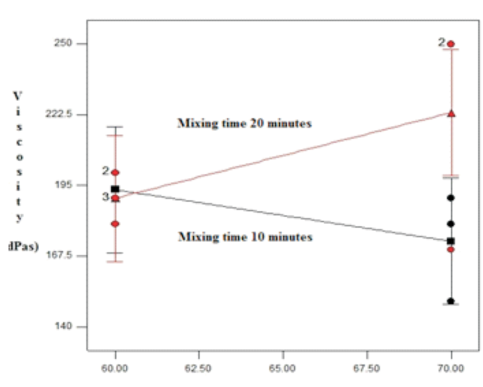
The recorded physical properties of green tea extract emulgel are collected in Table III, and the profile effect of mixing temperature and mixing time vs viscosity of green tea extract emulgel formulation are shown in Figure 1. Spreadability profile are shown in Figure 2.

Physical Properties Examination

The mixing process can influence the droplet formation of internal phase. Based on Peters (1997), there was the optimum process condition that can produce the optimum droplet size. In this optimum droplet size, good physical properties and physical stability can be achieved. Generally, the smaller droplet size will increasing the viscosity of the emulgel. The viscosity in all of the prepared green tea extract emulgel formulations was desired to have optimum value, so it can minimized droplet movement and the coalesence can be prevented. Based on the main effect in Table IV, all the factors will increasing the viscosity of emulgel, but the interaction between mixing temperature and mixing time has the biggest effect.

As seen in the Figure 1 (A), as mixing temperature get higher (60-70°C), will increasing viscosity respon in high level of mixing time (20 minutes), and will decreasing viscosity respon in low level of mixing time (10 minutes). The Figure 1 (B) shows the same profile, when mixing time get longer (10-20 minutes), will increasing viscosity respon in high level of mixing temperature (70°C), and will decreasing viscosity respon in low level of mixing temperature (60°C). The equation of factorial design for viscosity was Y = 636,667 -7,333 (X_A) – 32,333 (X_B) + 0,533 (X_A)(X_B). In this equation, X_A indicates mixing temperature and X_B indicates mixing time. Based on Anova, this equation was not significant, so it can not be used to predict viscosity respon. In this study there is no significant factor that affect the viscosity respon.

In semisolid dosage forms, spreadability was inverted proportional with viscosity. The higher the viscosity, it will reduce the spreadability, and vice versa. Emulsified product may undergo a wide variety of shear stresses during either preparation or use. The flow properties of the product will be vital for the proper performance of the emulsion under the condition of use or preparation. Thus,



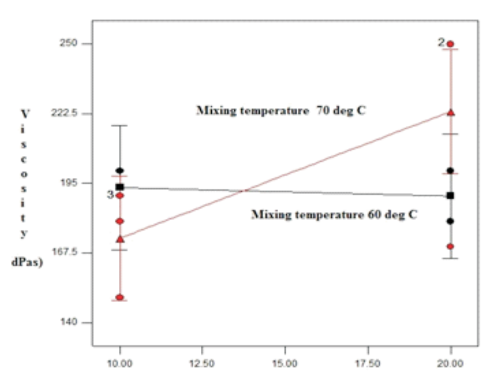


Figure 1. Profile effect of mixing temperature (A) and mixing time (B) vs viscosity of green tea extract emulgel formulation

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spreadability of dermatological and cosmetic product must be controlled to achive a satisfactory preparation (Sinko and Singh, 2012). The spreadability in all of the prepared green tea extract emulgel formulations was desired to have optimum value. Based on the main effect in Table 4, all the factors will decreasing the spreadability of emulgel, but the mixing time has the biggest effect.

equation was significant, so it can be used to predict spreadability respon. In this study mixing time was significant factor that affect the spreadability respon.

As mentioned above, time of mixing is important to ensuring equilibrium droplet size distribution is achieved. Based on Peters (1997), the longer time of mixing until optimum time, it can produce the droplet in smaller size. If the droplets are tend to smaller in distribution, the viscosity of the system will arise, and finally will decreasing the spreadability.

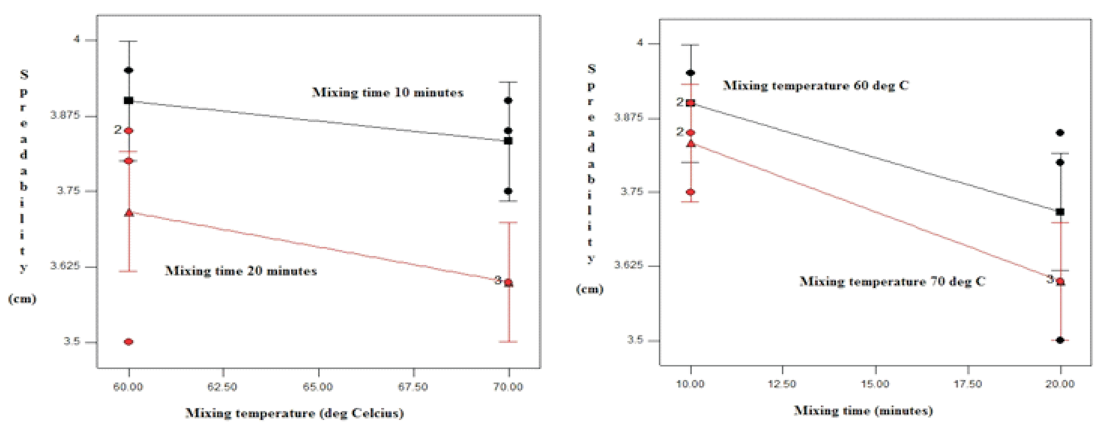

Figure 2. Profile effect of mixing temperature (A) and mixing time (B) vs spreadability of green tea extract emulgel formulation

As seen in the Figure 2 (A), the higher mixing temperature (60-70°C), will decreasing spreadability respon in both high and low levels of mixing time (10 and 20 minutes). The Figure 2 (B) shows the same profile, the higher mixing time (10-20 minutes), will decreasing spreadability respon in both high and low levels of mixing temperature (60 and 70°C). The equation of factorial design for spreadability respon was $Y = 4{,}183 - 1{,}667 \times 10^{-3} (X_A) + 0{,}0117 (X_B) - 5{,}00 \times 10^{-4}(X_A)(X_B)$. In this equation, X_A indicates mixing temperature and X_B indicates mixing time. Based on Anova, this

Physical Stability Study

In this study, viscosity shift was used as a parameter to predict physical stability of the green tea extract emulgel formulations. As seen in the Table 5, at among the formulations, formula ab show the best physical stability. It proved that the viscosity value after stored for a month was not changed. Formula b show the worst physical stability because it show the

Table V. Physical stability of green tea extract emulgel

Physical Stability	Formulation			
	1	a	b	ab
Viscosity shift (%)	9.43 ± 8.33	9.34 ± 5.62	31.59 ± 2.29	0.00 ± 0.00

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Table VI. Main effect of factors

Respons	Main effect		
	Mixing temperature	Mixing time	Interaction
Viscosity shift	-15.84	+6.41	-15.75

biggest viscosity change after stored for a month. Based on the main effect in Table 6VI, the mixing time will increasing the viscosity shift, whereas the mixing temperature and the interaction between both factore will decreasing the viscosity respon.

study mixing temperature and the interaction between mixing temperature and mixing time were significant factors that affect the viscosity shift respon.

Viscosity shift affected by change of size distribution of droplets in the emulsion system.

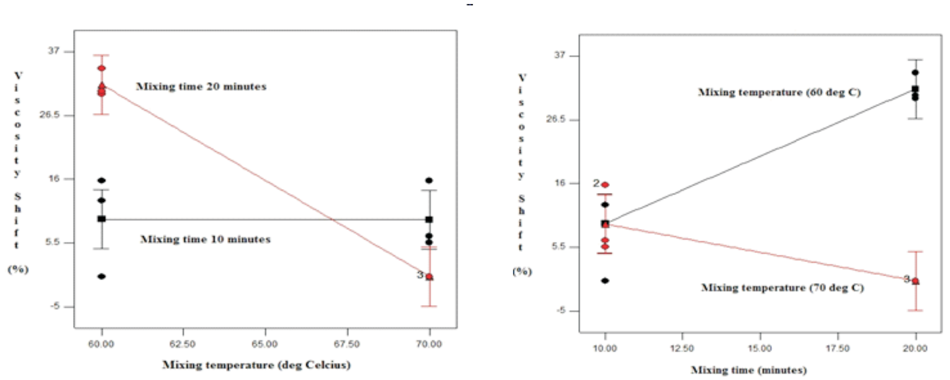

Figure 3. Profile effect of mixing temperature (A) and mixing time (B) vs viscosity shift of green tea extract emulgel formulation

As seen in the Figure 3 (A), the higher mixing temperature (60-70°C), will decreasing viscosity shift respon in high level of mixing time (20 minutes), and relative not change the viscosity shift respon in low level of mixing time (10 minutes). The Figure 3 (B) shows that more higher mixing time (10-20 minutes), will decreasing viscosity shift respon in high level of mixing temperature (70°C), and increasing viscosity shift respon in low level of mixing temperature (60°C). The equation of factorial design for viscosity shift respon was Y = -201,150 + 3,140 (X_A) + 21,113 (X_B) – 0,314 (X_A)(X_B). In this equation, X_A indicates mixing temperature and X_B indicates mixing time. Based on Anova, this equation was significant, so it can be used to predict viscosity shift respon. In this

In this study the change of size distribution of droplets are collected in Table VII.

Table VII. Distribution of droplet size (ìm, modus) of the emulgel

Formulation	Droplet size (?m, modus)	
	2 days	30 days
1	7,07 ± 0,06	4,00 ± 0,00
a	5,00 ± 0,00	3,33 ± 0,58
b	4,50 ± 0,00	4,00 ± 1,00
ab	2,60 ± 0,58	3,49 ± 1,53

As seen in table VI, at among the formulations, only formula ab show the change of size distribution of droplets in positive. It means that if the emulgel was stored for a longer time (30 days), so the droplets will change in bigger size. It will tends to be coalescence, and the emulsion system have a poor physical stability. In this study, based on Anova, only formula 1 that have a significant change. That means, although the distribution size of droplets seems to be change in formula a, b and ab, but statistically it says that there is no change in distribution size of droplets.

CONCLUSION

From the above result we can conclude that in these green tea extract emulgel formulations, mixing time was significant factor on determining the spreadability, and there are nothing factor that significant on determining viscosity. In other hand, mixing temperature and interaction between mixing temperature and mixing time were significant factors on determining the viscosity shift.

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